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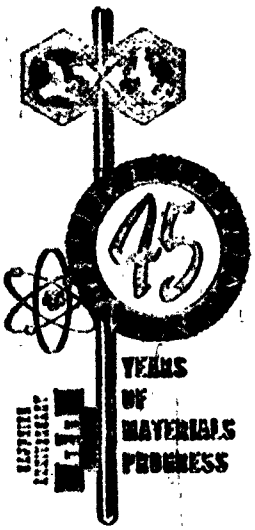
MATERIALS RESEARCH ABSTRACTS

A Review of the Air Force Materials Research and Development



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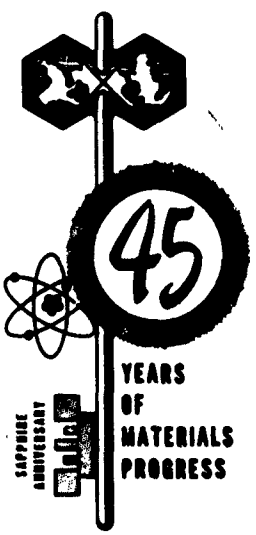
A Review of the Air Force Materials Research and Development

Edited by Charles D. Thibault
Science and Technology Division
Library of Congress



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Foreword

This book is one [illegible] prepared by the staff of the Science and Technology Divis[illegible] the Library of Congress, at the request and with the financial support of the Directorate of Materials and Processes (Materials Central), Wright-Patterson Air Force Base, Ohio. These three books have been compiled and published as part of the activities of the Materials Central in connection with the celebration of its forty-fifth year as the center of materials research and development for the United States Air Force. Three activities of Materials Central are highlighted in these publications. One is a compilation of award-winning technical papers prepared by Materials Central personnel, the second is a collection of abstracts of scientific and technical papers and reports prepared by Materials Central and its scientific and industrial contractors during the past decade and a half, and the third is a chronology of significant materials events beginning forty-five years ago and ending in the present. It is believed these three books will provide a valuable permanent set of references to anyone interested in the materials sciences.

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MATERIALS RESEARCH ABSTRACTS

MATERIALS RESEARCH ABSTRACTS

PART I

CHEMISTRY AND BIOCHEMISTRY

TR 5588
FUNGUS RESISTANCE OF RUBBER CORK COMPOSITION TAPE SPECIFICATION 12023. Francis Czarnecki, Pfc. May 1947.

Illinois State Natural History Survey.
TR 6518, Part 1
FUNGISTATIC CAPACITIES OF AROMATIC FLUORINE COMPOUNDS IN RELATION TO CLOTH-ROTTING FUNGI. PART 1 - FLUORINATED QUINONES AND PHENOLS. Leo R. Tehon and Sylvia Wolcyrz. AF 33(038)-10897. August 1952.

Examination of aromatic fluorine compounds -- six quinones and three phenol isomers -- by dilution plate and cloth square methods with respect to four cloth-deteriorating fungi showed general possession of fungistatic properties. All the quinones and two of the phenol isomers exhibited appreciable fungistatic capacities. The compound 2, 5-difluoro-1, 4-benzoquinone was most potent, completely inhibiting growth of Chaetomium globosum on unfavorable and favorable media at concentrations of 62 and 375 milligrams per liter, respectively, and completely protecting through 14 days squares of duck cloth saturated with an aqueous dispersion of the compound containing 500 milligrams per liter. Among the quinones fungistatic potency appeared to vary both with fluorine content -- the higher the content, the more potent the compound -- and, independently of fluorine content, with molecular structure. With phenol isomers, the position of the fluorine atom strongly influenced potency.

Illinois State Natural History Survey.
TR 6518, Part 2
FUNGISTATIC CAPACITIES OF AROMATIC FLUORINE COMPOUNDS IN RELATION TO CLOTH-ROTTING FUNGI. PART 2 - FLUORINATED PHENOLS, NITROBENZENES, AND ANILINES. Leo R. Tehon and Sylvia Wolcyrz. AF 33(038)-10897. August 1952

Of the twenty-five compounds tested since 1 March 1951, nine are phenols, nine are nitrobenzenes, four are anilines and three are other fluorinated preparations. Among the compounds and included for comparison are five that do not contain fluorine but are similar in structure to those that do.

To determine fungistatic potency, three test methods were employed: the agar dilution plate method, a treatment of squares of cotton fabric, and a treatment of cotton thread. Only those compounds that showed high toxicity by the first procedure were given the second and third tests. With the agar dilution plate method four fungi were used. With the other methods only the fungus Myrothecium verrucaria was used.

All of the compounds inhibited development of the fungi in some degree. Effective concentrations in agar were generally above 500 mg/liter for the phenols, above 200 mg/liter for both the anilines and the two nitrobenzenes, and above 5 mg/liter for the seven dinitrobenzenes.

Of the dinitrobenzenes, the most effective was 1-fluoro-3-bromo-4, 6-dinitrobenzene. It prevented the four test fungi from developing on agar that contained as little of it as 0.8 mg/liter and completely protected impregnated cotton thread that by weight contained only 0.15% of it.

Illinois State Geological Survey. TR 52-214
CHEMICAL INVESTIGATION OF FLUORINE COMPOUNDS AS FUNGICIDES. G. C. Finger, F. H. Reed, and J. E. Dunbar. AF 33(038)-26990. November 1952. PB 111 324. Order from OTS $1.00.

The chemical research program on organic fluorine compounds in reference to new fungicides was resolved into two phases: (1) the synthesis of compound types for rapid screenings and (2) a detailed study of the most promising types of compounds.

Approximately 35 fluorinated compounds belonging to the quinone, hydroquinone, phenol, aniline, and nitrobenzene types were synthesized and furnished the fungicidal testing program. The method of synthesis and the properties of each compound are described briefly. More than half of the compounds described are new and their chemical structures definitely proven. In several instances, 100-g. samples were prepared of the most promising compounds.

All of the compounds submitted for screening showed fungicidal properties with the dinitro and nitrophenols showing most promise. The greatest potency was discovered with 1-fluoro-3-bromo-4, 6-dinitrobenzene as it prevented fungus growth at 0.8 ppm. Complete protection of cotton thread and no loss of tensile strength were obtained by impregnation with the compound.

Reported for the first time is the synthesis of a chlorofluoroquinone by the 1, 4 addition of hydrogen chloride to a fluoroquinone followed by oxidation with hydrogen peroxide.

Illinois State Natural History Survey.
TR 6518, Part 3
FUNGISTATIC CAPACITIES OF AROMATIC FLUORINE COMPOUNDS IN RELATION TO CLOTH-ROTTING FUNGI. PART 3 - FLUORINATED ANISOLES, BENZYLS,

BENZOIC ACIDS, BIPHENYLS, PHENOLS, AND TOLUENES. Leo Roy Tehon. AF 33(038)-10897. November 1952.

Illinois State Natural History Survey.
TR 6518, Part 4
FUNGISTATIC CAPACITIES OF AROMATIC FLUORINE COMPOUNDS IN RELATION TO CLOTH-ROTTING FUNGI. PART 4 - FLUORINATED PHENOLS, BENZYL ALCOHOL, AND BIPHENYLS. Leo R. Tehon. AF 33(038)-10897. January 1954.

The fungistatic activities of eight aromatic fluorine compounds were investigated. The group was heterogeneous, comprising one chlorinated phenol, two nitrophenols, one benzyl alcohol, three biphenyls, and one biphenyl sulfide.

Potencies of each compound toward four fungi deleterious to cotton fabric were determined first by the agar dilution plate method. Of the more potent compounds, further tests were made to gauge their effectiveness in protecting cotton parachute webbing thread.

Least potent of the compounds was the benzyl alcohol. Most potent with a range of effectiveness of from 8.8 to 22.5 parts per million for the test fungi, was 4-fluoro-2-chlorophenol. The biphenyls, although only moderately potent, offered such advantages as lack of color, a solid state, and high melting point. Introduction of sulfur in the biphenyl linkage appreciably increased the potency of one compound.

In protection tests with parachute webbing thread compound content in treated thread of from 1.6 to 3.7 per cent, depending on the compound, was sufficient to prevent diminution of tensile strength in the presence of an actively growing fungus.

TR 54-30
THE INVESTIGATION OF FUNGICIDES FOR LEATHER. Martin A. Townsend, 1st Lt, USAF and Paul A. Albert, Capt, USAF, February 1954. ASTIA Document No. AD 33470. PB 133 165. Order from LC mi $2.70, ph $4.80.

Forty-two experimental formulations containing fungicidal chemicals have been evaluated as protective treatments against mildew on leather. Twenty-one were formulations containing orthophenylphenol, eleven formulations contained fluorinated compounds, three formulations contained trichlorophenyl acetate, three formulations contained paranitrophenol, two formulations contained parachlorometaxylenol, and the remaining two formulations contained di-lauryl dimenthyl ammonium bromide and 2,2 dihydroxy -5,5 dichlorodiphenyl methane, as the active fungicides. To determine the fungistatic effectiveness of each treatment, at least one of two methods was employed: the Petri plate method (mycelial mat), and the mixed spore suspension method. With the Petri plate method, only the fungus Aspergillus niger was used. With the mixed spore suspension method fourteen fungi were used.

All of the treatments inhibited the growth of fungi to some degree, with the exception of the formulations containing parachlorometaxylenol, 2,2 dihydroxy -5,5 dichlorodiphenyl methane, and di-lauryldimethyl ammonium bromide as the active fungicides.

In addition, most of the treatments which inhibited the growth of fungi were no more corrosive than the untreated leather, when in contact with cadmium plated steel, clad 24S-T3 aluminum alloy and brass.

Toxicity evaluations of orthophenylphenol and the fluorinated benzene derivatives show that these fungicides are nontoxic under the conditions studied.

University of Rhode Island.
WADC TR 54-421.
ANTIFUNGAL ANTIBIOTICS. Frank L. Howard. AF 33(616)-154. August 1954. PB 119 146. Order from LC mi $5.40, ph $15.30

One hundred forty-three candidate antibiotics furnished by twenty-one agencies were tested for their fungicidal activity. The molds specified and used were Aspergillus niger, A. terreus, Myrothecium verrucaria, and Chaetomium globosum. The difficult to inhibit spores of Alternaria solani, Helminthosporium carbonum and Curvularia lunata were used for germination tests. While most of the compounds were natural antibiotics, others were synthetic organic chemicals. Evaluation of antifungal action was obtained by four techniques: toxic agar in petri plates, spore germination inhibition on glass slides, impregnated cellulose pads on seeded agar, and retention of tensile strength impregnated thread after exposure to molds.

The five most antifungal antibiotics were found to be: comirin, benzyl mucochlorate, endomycin, netropsin sulfate, and rimocidin. The Squibb compound, 2-pyridinethiol 1-oxide and its twelve salts, compared favorably with standard copper 8-quinolinolate as a mildew inhibitor without necessarily staining the cloth. A furfural derivative exhibited evidence of high fungicidal action.

Illinois State Geological Survey. WADC TR 54-148.
CHEMICAL INVESTIGATIONS OF FLUORINE COMPOUNDS AS FUNGICIDES. G. C. Finger, F. H. Reed. AF 33(038)-26990. August 1954. PB 111 592. Order from OTS $1.00.

Fifteen aromatic fluorine compounds were prepared for fungicidal screening tests since 1 September 1952. The syntheses and properties of these compounds and intermediates are described in detail except in cases of known or borrowed derivatives. Ten of the test samples are new to the scientific literature. Classes represented are fluorinated benzoic acids, phenols, anisoles, toluenes, biphenols, biphenyl sulfide, and benzyl derivatives.

A search was made for colorless and thermally stable fungicides in the biphenyl and biphenyl sulfide classes. Three solid fluorinated hydroxybiphenyls, also called biphenols, were synthesized with 2,2-dihydroxy-5,5-difluorobiphenyl showing the most promise. When a sulfur linkage was introduced into the latter to give a biphenyl sulfide, fungicidal potency was increased so that 24-95 parts per million prevented growth of four test fungi.

Cotton thread impregnated with the sulfur compound showed no discoloration or loss in tensile strength, and when treated with 1000 ppm solution was completely protected. Likewise, 2-fluoro-6-nitrophenol, a potent fungicide, and the difluorobiphenols did not appear to change the tensile strength of the thread.

Flexfirm Products. WADC TR 53-474.
DEVELOPMENT OF AN IMPROVED FUNGICIDAL VINYL COATING FOR COTTON FABRIC. Richard R. Heitkamp, William J. Dewar, Dudley D. Eichorn. AF 33(616)-81. August 1954. PB 121 020. Order from OTS $1.25.

Secondary plasticisers and extenders were evaluated for their ability to impart fungistatic properties when incorporated into vinyl coatings and applied to cotton fabric. The evaluations were carried out by determining the tensile strength of coated fabric samples before and after two weeks soil burial as described in Federal Specification CCC-T-191a Supplement Section IV, part 5, pages 54 and 55.

The most satisfactory candidate plasticizers and extenders were then formulated into coatings that would meet the requirements of Military Specification MIL-F-4143.

Of all the secondary plasticizers and extenders, only one, dehydroabietylammonium pentachlorophenoxide, satisfied all requirements in providing the protection from microbial attack and at the same time giving the vinyl formulator the utmost leeway in producing an unlimited color range in waterproof coatings.

Some secondary plasticizers were found to have limited effectiveness where heavy coatings can be tolerated.

WADC TR 55-72
A COMPILATION OF DATA FROM EVALUATIONS OF THE FUNGUS RESISTANCE PROPERTIES OF AIR FORCE MATERIALS. E. L. Hamilton. April 1955. ASTIA Document No. AD 75796. PB 130 408. Order from LC mi $3.60, ph $9.30.

The main object of this work is to provide the designer with a guide for selection of fungus resistant materials in the design and maintenance of Air Force materiel which will require some degree of protection against microbiological degradation.

The materials discussed fall into three general classes: (1) those employing a fungicidal treatment, (2) those without treatment, but which show a natural resistance to fungi because of their chemical composition which does not readily provide fungi with a source of nutrient, and (3) the chemicals or formulations that are toxic to micro-organisms.

Fungicidal treatments which have proven unsatisfactory in the particular formulation tested are also listed. However, those found unsatisfactory may well prove satisfactory when used or tested under other conditions.

Many materials, if selected properly on the basis of future use in combination with other materials in the finished item, may provide a satisfactory fungis resistant material without the necessity of a chemical add-on treatment.

WADC TR 55-508.
INVESTIGATION OF SELECTED CHEMICALLY ALTERED COTTON MATERIALS. Earlana L. Hamilton. February 1956. PB 121 215. Order from OTS $0.75.

Existing natural cotton materials require the addition of an add-on fungicidal treatment when resistance to fungi is required. Cotton which has been altered chemically under controlled conditions shows inherent resistance to fungi. A material with the properties of cotton but showing high fungus resistance is needed for use where an add-on chemical fungicidal treatment is not acceptable. For this reason, acetylated and cyanoethylated cotton have been evaluated. Some of the advantages of chemically altered cotton fabric compared to cotton treated with an add-on fungicidal treatment are as follows: permanence and stability of physical characteristics after alteration, increased fungus resistance, non-toxic to humans, and ability to remain unaffected by leaching action of water.

Preliminary tests showed that acetylated cotton provided different degrees of fungus resistance as the amount of alteration varied. Work was initiated to find the level of alteration by acetylation which gave adequate resistance to fungi, yet retained the desired physical properties. The cotton acetylated at 12.6±0.4% level in an 8 ounce or lower weight fabric was found to be the best degree of alteration of those evaluated since it provided satisfactory fungus resistance with the least impairment of the desired physical strength properties. The 10 and 12 ounce fabrics altered at a 12.6 ± 0.4% level did not have adequate fungus resistance.

In addition to the chemical alteration of cotton by acetylation, there is the reaction of acrylonitrile with cellulosic fibers to produce a partially cyanoethylated cotton material. The degree of chemical alteration by cyanoethylation is measured by the percent nitrogen content of the cellulosic fibers after modification. Preliminary testing of cyanoethylated cotton in a 3.6 ounce weight fabric with a 3.6% nitrogen content showed that satisfactory fungus resistance could be obtained by cyanoethilation.

More extensive evaluations are needed before comparison of the two types of chemical alteration under study can be made.

WADC TR 56-208.
THE EFFECTS OF FUNGUS GROWTH AND MOISTURE UPON THE STRENGTH PROPERTIES OF REINFORCED PLASTICS. R. C. Tomashot, E. L. Hamilton. August 1956. ASTIA Document No. AD 97183. PB 121 513.

The effects of fungus growth and moisture upon the strength properties of several reinforced plastic laminates were investigated. Material variations included the type of reinforcement, the chemical type of resin, and the finish on the glass fabric reinforcement. Of the material variations, the type of reinforcement and finish on the glass fabric were significant in regard to both the amount of fungus growth and the reduction of the strength properties. The effect of fungus growth upon the strength properties was not considered significant as compared to the effect caused by the presence of moisture.

WADC TR 56-431
AN ELEMENTARY STATISTICAL APPROACH TO MICROBIOLOGICAL RESEARCH. James L. Mahan. September 1956. ASTIA Document No. AD 97279. PB 121 713.

The statistical approach is illustrated by making an analysis of data obtained from an experiment which was designed to evaluate a number of commercial fungicidal treatments on cotton webbing. The treatment and analysis of the data consists of the following:

1. A statistical summary of the data consisting of the Mean, Standard Deviation, and the Standard Error of the Mean.
2. An Analysis of Variance of the data. In addition, the Newman-Keuls test is used to arrange the significant treatment combinations according to their effectiveness.
3. The results of the analysis of variance and the Newman-Keuls test are considered in relation to information concerning the phenomona associated with the use of fungicides.
4. The information obtained from the analysis of variance and the statistical summary of the data are used to estimate the minimum sample size required for tests of fungicides according to specification requirements.
5. The importance of random sampling is discussed briefly and illustrated.
6. The importance and value of the statistically designed experiment are discussed. References to selected books concerning experimental design are given.

Applied Science Laboratories, Inc.
WADC TR 56-480 Sup 1.
EVALUATION OF FUNGUS RESISTANCE OF COTTON THREAD TREATED WITH SELECTED FUNGICIDAL

FORMULATIONS Supplement 1 Statistical Analysis of Data. Arthur Rose, Thomas B. Hoover. AF 33(600)-26749 S/A 3. October 1956. ASTIA Document No. AD 110441. PB 121 804.

Under Supplemental Agreement No. 3 to Contract AF 33(600)-26749 Applied Science Laboratories, Inc., has made a statistical analysis of the data obtained in an evaluation of the fungus resistance of cotton thread treated with selected fungicides. The data and a description of the test program were reported in WADC TR 56-480. The statistical treatment includes analysis of variance of breaking strengths of threads exposed to the fungus tests and also of the corresponding unexposed control threads. The direct effects of each of the main factors (color, finish, fungicidal agent, level of concentration of fungicide, and test method) upon the loss in breaking strength are evaluated and confidence limits are estimated.

WADC TR 56-252.
WEATHERING RESISTANCE OF FUNGICIDAL VINYL COATED COTTON FABRICS. John C. Saylor, Jr. 1/Lt. January 1957. ASTIA Document No. AD 110711. PB 121 913.

The fungicide, dehydroabietylammonium pentachlorophenoxide, formulated in experimental yellow and olive drab vinyl coatings on cotton fabric has been evaluated for resistance to weathering. A comparison has been made with copper 8-hydroxyquinoline formulated in an experimental olive drab vinyl coating and with an olive drab USAF stock vinyl coated fabric containing the same fungicide. The evaluations were based on breaking and tearing strength after outdoor weathering at the Alaska, Florida, New Mexico, and Wright-Patterson AFB exposure sites and on color change after weathering. The amount of breakdown in strength of the materials evaluated in this study has been correlated with the amount of light energy received from the sun.

The dehydroabietylammonium pentachlorophenoxide treated coated fabrics were not acceptable as compared to the coated fabrics with copper 8-hydroxyquinoline which maintained satisfactory strength and color after outdoor exposure. It was observed that shrinkage of the coated fabrics occurred during outdoor weathering.

WADC TR 56-384 Pt 1.
AN EVALUATION OF FUNGICIDAL TREATMENTS IN COTTON CARGO PARACHUTE WEBBINGS STORED AT WRIGHT AIR DEVELOPMENT CENTER. Emma F. Little. May 1957. ASTIA Document No. AD 118322. PB 131 158.

Five fungicidal formulations, with or without water repellents, were applied to four types of cotton cargo parachute webbings. Three of the formulations contained copper 8-quinolinolate, one contained dihydroxy-dichloro-diphenyl methane, and one contained dehydroabietyl ammonium acetate.

In order to determine the efficiency of these formulations in the webbings, five evaluations were made: (1) soil burial, (2) simulated tropical storage, (3) shelf storage under laboratory conditions, (4) agar plate exposure, and (5) agar plate exposure following laboratory shelf storage. Cellulolytic fungi used as test organisms were Aspergillus terreus, Myrothecium verrucaria, and Chaetomium globosum. Conclusions were based on general appearance and breaking strength data derived from tests of webbings and webbing threads, chemical analyses and visual observations. All formulations of copper 8-quinolinolate evaluated in this research were equally effective in webbing, providing the minimum metallic copper content as copper 8-quinolinolate was 0.14% by weight of the treated material. Further, webbings containing a minimum of 0.9% dihydroxy-dichloro-diphenyl methane by weight of the treated material, were equally fungus resistant. None of the fungicidal treatments caused significant tenderizing of the webbings after one year in simulated tropical or in laboratory shelf storage conditions. Webbings containing dehydroabietyl ammonium acetate were not resistant to fungi in soil and this fungicide caused primary skin irritation to human beings. Therefore, this fungicide was not considered satisfactory for USAF use.

WADC TR 58-32.
BACTERIAL ACTIVITY IN JP-4 FUEL. Sam Bakanauskas. March 1958. ASTIA Document No. AD 151034. PB 131813.

Studies were made with sludge samples obtained from tanks used to store JP-4 fuel, a kerosene-type fuel used in USAF jet aircraft. The tanks were located at Lincoln AFB, Schilling AFB, and Davis-Monthan AFB. Results of these studies indicated the following:

1. Sludge was caused by bacteria, and by products resulting from bacterial metabolic activity.
2. JP-4 fuel, and additives (corrosion and gum inhibitors) approved for USAF use in JP-4 fuel, are not bacteriostatic agents. JP-4 fuel, and some additives were found to be nutritive for bacteria.
3. A 1.5-2.0% concentration of sodium tetraborate, by weight, or a 2.0% concentration of potassium tetraborate, by weight, in water bottoms of JP-4 storage tanks can produce bacteriostatic conditions within the tank.

Georgia Institute of Technology.
WADC TR 57-711.
EVALUATION OF FUNGICIDAL TREATMENTS FOR COTTON FABRICS. Joseph J. Moder, Charles W. Stuckey. AF 33(616)-3867. April 1958. ASTIA Document No. AD 151127. PB 151142. Order from OTS $2.50.

Separate rolls of cotton sateen fabric were treated with seven different fungicidal formulations, each formulation being applied at three concentration levels, both with or without water repellents. Six of the fungicide formulations contained copper 8-quinolinolate and one contained dehydroabietyl ammonium pentachlorophenate.

In order to determine the effectiveness of these treatments, and also to obtain information for use in designing future evaluation and acceptance tests, cloth breaking strength measurements and visual observations were made on specimens of fabric treated as follows: (1) original fabrics, (2) fabrics stored indoors on shelves or hung in a laboratory, and (3) fabrics exposed outdoors. The original fabrics were tested before and after soil burial and agar plate exposure, while only the latter was used on the other test specimens cited in (2) and (3).

For the original fabrics, there was about a 10% strength difference between the strongest and weakest fungicide treated fabric. All treatments prevented fungus growth on agar plate exposure; however, considerable strength loss resulted from soil burial, after which the strongest fabric was almost three times as strong as the weakest fabric.

Twelve months of indoor shelf storage produced a negligible strength loss both before and after agar plate exposure for all fungicides. For open fabric panels hung in the laboratory at W-PAFB for two years, the copper 8-quinolinolate fungicides retained more than 94% of their original strength after agar plate exposure; however, the dehydroabietyl ammonium pentachlorophenate fungicide retained only 75% of its original strength.

The pattern of strength loss resulting from outdoor exposure varied among the test sites because of difference in the physical environment. A single linear equation, applicable to all test sites, was derived relating the strength retention to langleys of actinic exposure and relative humidity.

Fabrics treated with dehydroabietyl ammonium pentachlorophenate consistently had the lowest breaking strength after outdoor exposure at all sites. It is difficult to differentiate between the six copper 8-quinolinolate fungicide formulations because of the inconsistency in their behavior for the different test conditions and specimen types studied.

Lowell Technological Institute Research Foundation. WADC TR 57-366.
EVALUATION OF FUNGICIDAL VINYL COATED COTTON DUCK. Jacob K. Frederick, Jr., Robert E. Otto, 1/Lt., USAF, David H. Pfister. AF 33(616)-3929. June 1958. ASTIA Document No. AD 155686. PB 151464.

This investigation was concerned with determining the effect of shelf storage or outdoor exposure for periods of four, six, ten, and twelve months followed by soil burial for two weeks upon the breaking and tearing strengths, low temperature flexibility, gasoline resistance, hydrostatic pressure resistance, weathering resistance, high temperature blocking, and color of 8.0 ounce vinyl coated cotton duck containing in the fabric or in the coating various fungicides. In addition to the fungicides, some of the fabrics contained a stabilizer designed to prevent light deterioration. The exposure sites were in Alaska, Florida, New Mexico, and Ohio. The fungicides utilized in these studies were copper hydroxynaphthenate ($CuOHN_a ph$), copper 8-quinolinolate (Cu-8-Q), dehydroabietylammonium-pentachlorophenoxide (DAAP), dodecyldimethy-benzyl-ammonium-cyclopentane-carboxylate (DDBACC), and dihydroxydichloro-diphenyl-methane (3DM). The factors of breaking and tearing strength were considered to be of primary importance, while the others were considered as of secondary importance.

The primary factors were subjected to an analysis of variance and an analysis of covariance. The analyses considered main effects of fabric direction, exposure time, sites, and fabrics, and the interactions of fabric direction and time, fabric direction and sites, fabric directions and fabrics, time and sites, time and fabrics, and sites and time.

In addition, differences between fabrics were ranked by using the Duncan Test.

The secondary factors were evaluated by ranking and by examination techniques rather than by any formal statistical tests.

The major conclusions derived were as follows:

1. Fungicides studied had no effect in preventing deterioration prior to soil burial. In soil burial, Cu-8-Q, $CuOHN_a ph$, and DDBACC supplied significantly better protection against strength loss than did DAAP and 3DM.
2. The duration of exposure had a significant effect on the loss in strength of fabrics subjected to outdoor exposure, but had no effect on fabrics subjected to shelf storage only.
3. The presence of a stabilizer had no significant effect on the prevention of strength deterioration as compared to similar fabrics not containing such a stabilizer.
4. Light can have a significant effect on strength loss, with a direct and linear relationship existing between increasing light energy and increasing strength loss.
5. The fabrics treated with the fungicides providing best protection against soil burial strength loss also showed the least harm in the low temperature flexibility test.
6. Hydrostatic pressure resistance was not adversely affected by sites, time of exposure, nor types of exposure.
7. Color had a bearing on gasoline resistance as did fungicides. Yellow fabrics and all fabrics treated with 3DM showed more deterioration than the other fabrics.
8. The chemical analyses for residual fungicide content, showed considerably less than was stated to be present for all the fungicides except $CuOHN_a ph$.

WADC TR 56-384 Pt II.
AN EVALUATION OF FUNGICIDAL TREATMENTS IN COTTON CARGO PARACHUTE WEBBINGS STORED AT COLLEGE, ALASKA. Emma F. Little. August 1958. ASTIA Document No. AD 155857. PB 151252. Order from OTS $1.50.

Fungicidal formulations with water repellents and without water repellents were applied to four types of cotton cargo parachute webbings made to Specification MIL-W-5665A. Three of the formulations contained copper 8-quinolinolate and the fourth contained dihydroxy-dichlorodiphenyl methane.

Treated and untreated webbings as received from the manufacturer, and warp threads from these original webbings, were tested for initial breaking strength at Wright Air Development Center prior to storage. These breaking strengths were used as the control data for the materials stored for periods of 4, 8, and 12 months at College, Alaska. Threads from stored webbings were broken to detect any losses in breaking strength due to tendering by the fungicidal treatments. After periodic shelf storage, the webbings were evaluated for fungus resistance. The fungi used as test organisms were Aspergillus terreus, Myrothecium verrucaria and Chaetomium globosum. Visual examinations were made after fungus resistance evaluations, and breaking strength data were derived from threads from webbings after storage and after fungus resistance investigations. A statistical analysis was made of these data. Any losses in fungicide efficiency were determined from the data showing the differences in breaking strength retention of the exposed threads when compared to the original controls.

All formulations of copper 8-quinolinolate, except Formulation B, evaluated in this research were satisfactory fungicidal treatments in cotton cargo parachute webbings, Specification MIL-W-5665C, providing the minimum metallic copper content as copper 8-quinolinolate was 0.14% by dry weight of the treated material. On this basis, the minimum amount of copper 8-quinolinolate required to give adequate fungus resistance was 0.83% by dry weight of the treated material. The formulation of dihydroxy-dichlorodiphenyl methane was equally acceptable for USAF use providing the minimum fungicide content was 0.9% by dry weight of the treated material. Water repellents added to the basic fungicidal formulations, slightly improved the overall fungus resistance of the treated webbings. The phthalocyanin dye used for Type XV webbing was not compatible with any of the fungicidal formulations. Untreaded webbing containing this dye was badly discolored and tacky. Mineral dye was compatible with all materials.

The Scientific Oil Compounding Co. WADC TR 58-303.
DEVELOPMENT OF EFFECTIVE NON-TOXIC FLUORINATED FUNGICIDAL FORMULATIONS FOR COTTON MATERIALS. Charles C. Yeager, Jay C. Chapin.

AF 33(616)-5322. October 1958. ASTIA Document No. AD 203525.

Methods of formulating 2, 2-dihydroxy 5, 5-difluorodiphenyl sulfide (DDFDS) were investigated with the purpose of developing effective solvent soluble and water miscible formulations capable of producing fungicidal and fungicidal-water repellent finishes for cotton materials.

Initial investigations included studies on the solubility of the fungicide in a wide range of solvents and storage stability of the resulting concentrated solutions. It was necessary to find solvents, or mixtures of these materials, which would dissolve the greatest amount of the DDFDS and retain it in solution for prolonged periods of time under variable room temperature conditions. A combination of hexylene glycol with a mixture of hexyl and amyl alcohols was found to be the best for the solvent formulations. Di-isobutylketone was found to be the best for water emulsion formulations. Samples of a standard cotton sateen fabric were treated with many experimental formulations to determine their effectiveness against degradation caused by fungi and ultraviolet light. The DDFDS, when properly formulated, was found to be a very effective fungicide. However, the compound did decompose during extended, accelerated, artificial weathering. Various non-pigmented anti-oxidants and screening agents were evaluated to counteract this breakdown but none were found to be wholly effective.

Preliminary work indicated that stabilization of the DDFDS can be accomplished with selected amine compounds. Formulations utilizing the stabilized fungicide proved promising when used in a fabric material especially those applied from an aqueous emulsion system.

WADC TR 58-201
THE EFFECT OF SOLAR RADIATION ON THE BREAKING STRENGTH OF OUTDOOR EXPOSED WEBBINGS. Robert A. Wilkinson. November 1958. ASTIA Document No. AD 206893. PB 151 590. Order from OTS $1.50.

This program was to obtain data as to what degree solar radiation affected the breaking strength of webbings exposed to natural weather. Four groups of webbings were evaluated for breaking strength after being exposed to natural weather for specific time intervals encompassing one year. The exposure sites were Wright-Patterson Air Force Base, Ohio; Las Cruces, New Mexico; and College, Alaska.

Data obtained indicate that the service life of the present type of runway barriers can be increased to 180 days in areas of extremely strong sunlight and to 360 days in areas of moderate or small amounts of sunlight by either one of the following two methods:

1. By using 260 denier 17 filament, Type 300, OD color nylon yarn in the manufacture of the webbing.
2. By using Type 330 nylon yarn in the manufacture of the webbing.

South Florida Test Service, Inc.
WADC TR 58-502
NATURAL WEATHERING AND INDOOR OPEN SHELF STORAGE EXPOSURE TESTING OF AIR FORCE FABRIC MATERIALS. Warren W. Smith. AF 33(600)-34695. March 1959. ASTIA Document No. AD 210226.

Webbing and cloth specimens submitted by Wright Air Development Center were exposed to natural direct weathering and indoor open shelf storage environment by the South Florida Test Service, Inc. at Miami, Florida for a period of twelve (12) months. The purpose of this project was to determine degree of degradation of the various samples during the exposure period.

Various degrees of degradation were noted on the specimens exposed to natural direct weathering, however none was noted on the specimens subjected to indoor open shelf storage.

WADC TN 59-142
RESISTANCE OF MICROORGANISMS TO HIGH VACUUMS. Sam Bakanauskas. April 1959.

A study was made to determine the effect of prolonged dynamic vacuums on the viability of selected spore-forming fungi and bacteria. The microorganisms were exposed to pressures ranging from 1 x 10-5 to 5 x 10-7 mm Hg for periods of 2, 4, 8, 16 and 32 days.

Microorganisms used were: Aspergillus niger WADC 8, Aspergillus flavus WADC 26, Bacillus globigii WADC B5, Bacillus mycoides WADC B8, and Bacillus cereus WADC B6. With the exception of B. cereus WADC B6, spores of all microorganisms remained viable after 32 days exposure.

University of Cincinnati. WADC TN 57-232. EVALUATION OF FOUR AROMATIC FLUORINE COMPOUNDS AS FUNGICIDES FOR VEGETABLE AND CHROME TANNED LEATHERS. Jerome J. Jansing, William T. Roddy. AF 33(616)-2466. August 1957.

Fungus resistance evaluations were made on vegetable-tanned and chrome-tanned leather materials treated with basic formulations of selected aromatic fluorine chemicals.

Results indicate that 3, 3-difluoro-4, 4-dihydroxybiphenyl; 5, 5-difluoro-2, 2-dihydroxybiphenyl; and Bis (-2-hydroxy-5-fluorophenyl) sulfide each have a good potential of being developed into non-toxic and effective fungicides for use in leather materials.

PART II

MATERIALS

Acoustic Materials and Studies

Soundrive Engine Co., Los Angeles, Calif. Report No. 79. AFOSR-TN-54-276.

STUDIES OF HIGH AMPLITUDE SOUND. A. MEASUREMENTS OF THE ACOUSTIC IMPEDANCE OF A RESONATOR AT LARGE AMPLITUDES. B. MEASUREMENTS OF THE ATTENUATION OF REPEATED SHOCK WAVES. C. A. LOGARITHMIC AMPLIFIER. O. B. Wilson, Jr. and D. A. Bies. AF 18(600)495. August 1954. ASTIA Document No. AD 52274.

The experimental investigation of the acoustic impedance of a Helmholtz-type resonator at very high amplitudes of excitation is presented. Sound levels up to 180 db and particle velocity amplitudes in the neck of the resonator up to 1.5×10^4 cm/sec were used. The results at lower levels are compared with those of previous investigators whose work has been confined to levels below 140 db and particle velocity amplitudes in the resonator neck below 4×10^3 cm/sec. Experimental work on the rate of attenuation of repeated shock waves in a 5-in. tube is reported. Qualitative agreement with theory is found, but there is at present no adequate explanation of quantitative discrepancies. The design and construction of a logarithmic amplifier is discussed.

California Institute of Technology. Norman Bridge Laboratory of Physics, Pasadena. Technical Note No. 3. AFOSR-TN-55-112.

THEORY OF THE PLANE WAVE ACOUSTIC FILTER WITH PERIODIC STRUCTURE. W. G. Cady. AF 18 (600)593. 1955.

After a brief discussion of the general properties of filters of different types (e.g., optical, electrical, mechanical), attention is given to an acoustic filter consisting of an arbitrary number of identical sections placed between any 2 media. Each section consists of 2 plane-parallel layers, which may be of any materials and thicknesses. It is assumed that there are no losses due to absorption in these materials, and that plane waves of arbitrary frequency are incident normally on the filter. The theory of filters of this type is developed, involving an expression for the specific acoustic impedance at any point, and also a convenient iteration factor which facilitates the calculation of reflection and transmission. The formulas are then specialized for the case in which the thickness of each layer is the same fraction of a wavelength, including a discussion of quarter-wave layers. A proof is given of the reversibility of the filter. Numerical calculations have been carried out for filters with assumed constants. The results for one case are presented in the form of curves relating the coefficient of reflection to the frequency.

California Institute of Technology. Norman Bridge Laboratory of Physics, Pasadena. Final report. AFOSR-TN-55-79.

RESEARCH IN HIGH-FREQUENCY ULTRASONICS. W. G. Cady. AF 18(600)593. March, 1955. ASTIA Document No. AD 61828.

This final report summarizes the work done during the 2-yr. contract. After a survey of proposed undertakings, abstracts are given of completed work on composite piezoelectric resonators, theory of acoustic filters having a periodic structure, and theory of compressional waves in crystals. A report is given on the attempts to generate acoustic vibrations at a frequency of 3000 mc/sec. Descriptions are given of the transducer and cavity resonator used in the unsuccessful experimental tests. As a result of this research study, it was suggested that improvements should be made by: (1) obtaining a microwave generator with very high frequency stability, and at the same time having the frequency adjustable over a small range, at least; (2) constructing and mounting a transducer in such a manner as to ensure exposure to a strong electric vector without undue heating; (3) providing greater uniformity in the thickness of quartz or other transducer material; and (4) searching for other substances having piezoelectric properties in high-frequency fields which can be prepared as uniform thin films.

Soundrive Engine Co., Los Angeles, Calif. Report No. 82. AFOSR-TR-55-10.

STUDIES OF VERY HIGH AMPLITUDE SOUND. O. B. Wilson, Jr. and D. A. Bies. AF 18(600)495. April, 1955. ASTIA Document No. AD 72433. PB 137707.

A standing wave technique was used to investigate the acoustic impedance of a Helmholtz-type resonator over a wide range of sound levels. At large sound pressure levels above 150 db, the sound waves propagating down the impedance tube became essentially repeated shock waves so that it was necessary to use a filter in the monitoring system, and to consider only the behavior of the fundamental component in the repeated shock wave. In this way, a range of sound pressure levels in the resonator from 100 to 170 db was investigated. Two different methods of mounting the resonator were considered. In one case the resonator was mounted on the side of the impedance tube next to a solid end plate terminating the tube, and in the other case the resonator was mounted on the solid end plate coaxially with the impedance tube. At the higher levels of excitation a rise in resonant frequency and in acoustic resistance was noted in both cases, but the details of the effects in each case were quite different. With the resonator mounted on the side the rise in the acoustic resistance followed Sivian's formula (Jour. Acoust. Soc. Amer., v. 7:94, 1935) while the mass end correction decreased only at very high sound pressure levels. With the resonator mounted on the end, the rise in acoustic resistance was at first more rapid, then less rapid than

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Sivian's formula would predict. The decrease in mass end correction with increasing sound pressure level began at a lower level and was more pronounced than in the former case.

Massachusetts Institute of Technology. Research Laboratory of Electronics, Cambridge.
A THEOREM CONCERNING NOISE FIGURES. A. G. Bose and S. D. Pezaris. DA 36-039-sc-64637. 1956. Published in I.R.E. Trans. of Professional Group on Circuit Theory, v. CT-3: 190-196, September, 1956.

A theorem is formulated and proved which determines the greatest lower bound of the single-frequency noise figure of a general system consisting of n amplifying devices and passive coupling elements. According to the theorem, this lower bound is equal to the noise figure of an optimum system using a selected one of these amplifying devices.

University of Minnesota. WADC TR 59-70.
BIBLIOGRAPHICAL REVIEW OF PANEL FLUTTER AND EFFECTS OF AERODYNAMIC NOISE. J.V. Rattayya, L. E. Goodman. AF 33(616)-5426. June, 1959. ASTIA Document No. AD 215448. PB 151890. Order from OTS $1.25.

The literature in the field of panel flutter and aerodynamic noise has been surveyed and a bibliography assembled. This work has been a first step in a comprehensive research investigation of acoustic fatigue of aircraft panels undertaken by the staff of the Aeronautical Engineering Department, University of Minnesota. It is felt that the material is of sufficient interest to workers in the field to justify reporting at this stage.

A critical review of the more important of the two hundred and fifteen bibliographical references is included. This summary does not represent original research, although of course, the opinions expressed in the description of published work are those of the authors of this report. It is thought that duplication of effort may be avoided and progress advanced by the availability of a comprehensive summary of what has thus far been accomplished toward the understanding and amelioration of a serious difficulty in airframe design.

Armour Research Foundation. WADC TR 58-460, Part I.
DEVELOPMENT OF NEW SOUND ABSORBING MATERIALS FOR NOISE SUPPRESSORS PART I. Development of Equipment for Evaluating Acoustical and Durability Properties of Sound Absorbing Materials at Elevated Temperatures. William E. Lawrie. AF 33(616)-5060. June 1959. ASTIA Document No. AD 215445. P B 151907. Order from OTS $2.00.

This report describes the development of equipment and techniques for the measurement of high temperature acoustical and mechanical properties of absorbing materials for use in aircraft engine test cells. An acoustical impedance tube has been constructed to determine the high temperature acoustical properties and the necessary techniques developed to obtain accurate measurements in the presence of the temperature distribution found in the tube. In addition, a test cell has been constructed that simulates the environment to which the acoustical materials will be exposed. This cell is to be used to determine the appropriate mechanical properties of acoustical materials to ascertain that the materials will withstand the environment of the aircraft engine test cells.

Armour Research Foundation. WADC TR 58-460, Part II.
DEVELOPMENT OF NEW SOUND ABSORBING MATERIALS FOR NOISE SUPPRESSORS PART II. Evaluation of Commercially Available Materials. William A. Lawrie. AF 33(616)-5060. June 1959. ASTIA Document No. 215445. PB 151907.

The acoustical and mechanical properties of six acoustical materials at high temperatures are described. The materials Basalt Wool, Thermoflex, J. T. Sound Insulation, Fiberglas, Firebrick and Metal Fiber, were selected on the basis of their room temperature acoustical properties and/or their probably mechanical properties at high temperatures.

The acoustical properties of the materials were determined by means of an acoustical impedance tube described in Part I of the reports on this project. Essentially, the standard room-temperature methods of measuring normal absorption coefficient and acoustic impedance are used, modified to correct for the effects of temperature gradients.

The mechanical properties of the materials were measured by means of equipment and techniques developed during Phase I of the program. The properties were determined in a manner that would provide information regarding the ability of the materials to withstand the environment of aircraft engine test cells.

Midwest Research Institute. WADC TR 59-231.
DEVELOPMENT OF A FATIGUE TESTING APPARATUS FOR TESTING HONEYCOMB SANDWICH STRUCTURAL PANELS IN SERVICE-SIMULATED SONIC FATIGUE. Edmond F. E. Zeijdel, William H. Ashely. AF 33(616)-5172. September 1959. PB 161317. Order from OTS $1.00.

Theoretical and experimental investigations for the development of a fatigue testing apparatus for testing honeycomb sandwich panels in service-simulated sonic fatigue are described in this report. These investigations indicate the need of a 200,000-v. AC generator to simulate electrostatic fields of approximately 400,000-v/cm. In addition, it is indicated that the medium surrounding the test specimen must be changed to prevent corona discharge. For this purpose sulfur hexafluoride at 9 to 10 atmospheres may be utilized.

The design of a 200,000-v. AC generator is complicated and expensive. An alternative procedure to accomplish the same objectives is therefore recommended.

WADC TR 59-304.
ROLE OF STRUCTURAL DAMPING IN ACOUSTICAL FATIGUE. W. J. Trapp, B. J. Lazan. January 1960. PB 161742. Order from OTS $1.00.

Techniques proposed to reduce or eliminate materials fatigue damage due to high level noise fields of propulsion system and aerodynamically induced pressure fluctuations are discussed. The mechanisms, the significant parameters involved and the response of materials and structures are analyzed. The activities undertaken and notions on the solution of the problem by employing certain damping concepts are presented.

University of Southampton, United Kingdom. WADD TR 61-70.
EXPERIMENTAL STUDY OF THE RANDOM VIBRATIONS OF AN AIRCRAFT STRUCTURE EXCITED BY JET NOISE. B. L. Clarkson, R. D. Ford. AF 61(052)-332. March 1961. ASTIA Document No. AD 266374. PB 171912. Order from OTS $7.00.

Recordings have been made of the strains induced in a full scale rear fuselage test structure of the Caravelle airliner when one jet engine is running at maximum take-off thrust. The structure is a conventional sheet-stringer combination attached to pressed out frames.

The analysis has been concentrated on the strains in the centres of panels. Correlation measurements have indicated that the lower frequencies (up to 500 c.p.s.) are associated with overall vibration modes and have low strain amplitude. The larger panel strains occur at higher frequencies with the frames acting as boundaries. In these measurements the main resonance peak in each panel occurs at about 600-700 c.p.s. and has been identified with the fundamental twisting mode (i.e., adjacent panels 180 out of phase). There are generally two smaller peaks in the 800-1000 c.p.s. range but the modes of vibration have not been completely identified due to lack of information.

An attempt has been made to calculate the panel resonant frequencies theoretically, assuming that the frames act as boundaries. Although this work appears promising it has not yet progressed far enough for any definite conclusions to be drawn from it.

WADC TR 59-676.
WADC - UNIVERSITY OF MINNESOTA CONFERENCE ON ACOUSTICAL FATIGUE. W.J. Trapp, D. M. Forney, Jr. March 1961. ASTIA Document No. AD 266374. PB 171912. Order from OTS $7.00.

This report is composed largely of invited papers and some selected seminar material presented at the Conference on Acoustical Fatigue held at Beecher's Resort, Annandale, Minnesota, from 29 September 1959 through 2 October 1959. The conference was organized jointly by the Materials Laboratory of Wright Air Development Center and the University of Minnesota. It was designed to establish communication between individuals from the several disciplines involved in acoustical fatigue problems, namely, acoustics, applied mechanics, and service failure analysis. It was hoped thereby that an improvement in the over-all understanding of the acoustical fatigue problem would ensue. Another important objective was to define clearly the critical problem areas and aspects which would require emphasis in the future. Over 100 participants from the United States and abroad were invited to attend and take part in the discussions. A list of attendees is included in this report.

Ceramics and Cermets

Battelle Memorial Institute. TR 6218, Part 1.
REFRACTORIES FOR MELTING TITANIUM. L. W. Eastwood and C. M. Craighead. W33-038-ac-21229. June 1950.

American Electro Metal Corporation. TR 6601, Part 1.
AN INVESTIGATION OF THE ALLOYS OF ALUMINUM AND MOLYBDENUM. R. L. Wachtell. AF 33(038)-10716. March 1951.

Battelle Memorial Institute. TR 6512.
MECHANICAL PROPERTY TESTS ON CERAMIC BODIES. W. H. Duckworth, A. D. Schwope, and J. K. Johnston. AF 33(038)-8682. April 1951.

Ohio State University Research Foundation. TR 6515.
IMPREGNATION OF CARBIDE SKELETAL BODIES WITH SELECTED METALS OR ALLOYS. Earle T. Montgomery, Thomas S. Shelvin, and Clinton C. McBride. AF 33(038)-6841. May 1951.

Battelle Memorial Institute. TR 52-67.
MECHANICAL PROPERTY TESTS ON CERAMIC BODIES. W. H. Duckworth, A. D. Schwope, and O. K. Salmassy. AF 33(038)-8682. March 1952. ASTIA Document No. AD 3245. PB 111416. Order from OTS $2.00.

A critical survey was made of the significant theories of strength, for guidance in developing relationships among the strength properties of ceramics. The mechanistic theories appeared to offer the greater possibilities, but no theory treated all controlling variables, and all theories lacked adequate experimental support. The need remains apparent for a unified theory and supporting experimental data.

The principal laboratory effort was on the size dependence of strength. Both plaster and a nickel-titanium carbide body decreased in apparent strength with increases in gage-section size in bend tests. In an extensive program of bend tests on plaster to record details, strength decreased with increases in either gage-section length or gage-section breadth in about the same manner. However, the apparent strength increased with increases in gage-section depth. The possibility of the true size effect's being masked by size-dependent testing variables was indicated. There was no trend apparent in the standard deviation of strength values with variations of gage-section length, breadth, or depth.

Further information was obtained on the effect of the type of test on mechanical properties. Of particular interest is the fact that, with sufficient refinement, the bend and torsion tests appear to yield practically the same strength values. The development and refinement of tests was continued in an effort to obtain the precise mechanical-property data needed in this program.

Sintercast Corporation of America. TR 52-92.
INVESTIGATION OF INFILTRATED AND SINTERED TITANIUM CARBIDE. C. G. Goetzel, J. B. Adamec, J. L. Ellis, and D. Trauberman. AF 33(038)-16103. April 1952. PB 109766. Order from LC mi $2.75, ph $7.50.

An investigation was carried out for the purpose of evaluating composite bodies consisting of titanium carbide and nickel-base alloys and intermetallic compounds, produced by the infiltration and conventional powder metallurgical processes for use as structural materials in high-temperature components of aircraft engines.

The nickel alloys and the one compound tested were Nichrome V, Hastelloy "C", Inconel, and NiAl, respectively. Production techniques were developed for nine in. long stress rupture test bars, containing Inconel as infiltrant. These were produced by the infiltration process and submitted to the Materials Laboratory, Research Division, Wright Air Development Center for stress rupture tests together with specimens of similar composition made by sintering.

Tests carried out on the material were:

1. oxidation testing in air at 1600°, 1800°, and 2000°F;
2. X-ray diffraction analysis of the oxide products;
3. thermal shock testing at 2500' and 2300°F;
4. modulus of transverse rupture at room temperature;
5. modulus of transverse rupture at 1800°F;
6. ductility at 1800°F.

It was found that, among the materials tested, titanium carbide-Inconel, infiltrated by the capillary infiltration method, had the most favorable combination of high-temperature strength, ductility, and oxidation resistance at elevated temperatures. Its facility of production, however, was inferior to that of titanium carbide infiltrated with other nickel alloys. While it was possible to produce from Inconel-infiltrated titanium carbide 3 x 1/2 x 1/2 in. specimens of great physical and structural uniformity, nine in. long bars required for stress rupture specimens still possessed some regions of less homogeneity as evidenced by microporosity.

In the modulus of transverse rupture tests, the Inconel-infiltrated type of material proved to be far superior in strength at room temperature, strength at 1800°F, and bending capacity at 1800°F, over a cemented titanium carbide of similar composition made by cold-pressing and sintering.

American Electro Metal Corporation.
TR 52-291.
AN INVESTIGATION OF VARIOUS PROPERTIES OF NiAl. Richard L. Wachtell. AF 33(038)-10716. September 1962. PB 121021. Order from OTS $1.25.

Production of the alloy NiAl and a modified composition NiAl + 5% Ni has proved feasible as well as its subsequent fabrication by powder metallurgical technique. Properly hot-pressed bars of the NiAl + 5% Ni composition show strengths in modulus of rupture as high as 144,000 psi at room temperature and 68,000 at 980°C.

The air oxidation resistance of the modified NiAl + 5% Ni composition is excellent up to 1095°C., with weight gains of the order of 1.25 MG./CM^2 being exhibited after 300 hours of exposure.

Heat shock properties are likewise excellent, as judged by NACA tests, and by performance in the Air Force Heat Shock apparatus.

American Electro Metal Corporation.
TR 6601, Part 2.
AN INVESTIGATION OF THE ALLOYS OF ALUMINUM AND MOLYBDENUM. Richard L. Wachtell. AF 33(038)-10716. October 1952.

In addition to the conclusions cited in the previous report (Air Force Technical Report 6601, Part 1), the following significant facts have been established from the work which is reported herein:

1. No firm explanation has been established regarding the mechanism of rapid oxidation of certain of the Molybdenum Aluminum alloys, but its existence under conditions of good ventilation suggest a mechanism different from that of the usual "catastrophic oxidation" of Mo containing alloys.
2. Oxidation resistance data have been obtained for several alloys, and it is shown that a properly chosen Mo/Al alloy, specifically, the No. 2 alloy + 10% Al, or No. 2 + 15% Al, as described in the text, will develop excellent oxidation resistance in all temperature ranges tested (870°C to 1150°C).
3. Hot pressing of shapes of these alloys is extremely difficult due to the sticking tendencies of the material, but three representative parts have been made; a rocket nozzle, a turbosupercharger bucket, and stress to rupture bar.
4. Control equipment has been developed for the hot pressing operation which makes possible a fair reproducibility of strength in small bars of 40,000-50,000 PSI (modulus of rupture).
5. A pre-alloyed powder of the same overall chemical analysis as the No. 2 alloy + 10% Al has about the same oxidation resistance, but is more difficult to hot press.
6. Methods have been developed for the relatively large scale production (1 kg batches) of Mo/Al powder.
7. Metallographic studies indicate that the physical properties of the alloys under investigation may be influenced by heat treatment.

Pennsylvania State College. TR 53-9.
REFRACTORY MATERIALS FOR USE IN HIGH-TEMPERATURE AREAS OF AIRCRAFT. Norman R. Thielke. W33-038-ac-16374. January 1953. ASTIA Document No. AD 9729. PB 130819. Order from LC Mi $3.60, Ph $9.30.

Theoretical approaches to the mechanism of thermal expansion were reviewed and applications thereof to the alkali halides were examined for clues to the thermal behavior of refractory materials. Inadequate theory and incomplete data precluded any valid generalizations. Crystal structure appeared as an important determiner of expansion behavior.

Aluminum titanate bodies matured only above 1260°C; five hours' heating at 1400°C developed moderate strength and low expansivity. Expansion of such bodies to 1000°C approached zero; a rapid rise accompanying decomposition ensued between 1000° and 1400°C. Thermal hysteresis was indicated as a time-consuming effect related to reaction equilibrium. The isostructural nature of aluminum titanate and pseudobrookite was confirmed and the expansion anisotropy of the former was investigated. Substitution of equivalent or isomorphous oxides in the aluminum titanate formula yielded no marked improvement in overall properties. Addition of numerous high-silica glasses also failed to strengthen the bond of aluminum titanate bodies without sacrifice of low expansivity.

Slip-cast, clay-bonded alumina and beryl turbine nozzle blades failed after 1-4 cycles of thermal shock; similar clay-bonded aluminum titanate blades easily withstood 75 cycles of shock from 885° to 25°C under simulated service conditions.

A literature review relative to crystallographic inversions indicated the merit of solid soluble constituents or a restraining glassy envelope as means of control of damaging volume changes during thermal cycles.

Pennsylvania State College. TR 53-165.
ALUMINUM TITANATE AND RELATED COMPOUNDS. Norman R. Thielke. AF 33(616)-139. June 1953. PB 111440. Order from OTS $1.50.

The preparation of aluminum titanate and its structural, thermal and mechanical properties were investigated. A melting temperature of 1865°C and a region of instability between 1260°C and about 860°C, plus extreme thermal expansion anisotropy in the crystal rise to unusual properties in the matured crystalline aggregate. These include negative expansion coefficients, marked resistance to thermal shock, thermal expansion hysteresis and low transverse strength. The expansion behavior and strength of matured aggregates are variously altered by heat treatment, by kiln atmosphere and by incorporation of minor amounts of oxides or gasses.

Aluminum titanate is recommended as a thermal shock resistant refractory material in applications involving low tensile loads and oxidising conditions at temperatures within its stability ranges. Fabricated shapes may serve satisfactorily as nozzle diaphragm blades, flame tube liners, refractory coatings or other ceramic elements of combustion systems.

Armour Research Foundation Illinois Institute of Technology. TR 53-91.
INVESTIGATION OF REFRACTORIES SUITABLE FOR MELTING TITANIUM AND ITS ALLOYS. Marvin Eisenberg and James Stavrolakis. AF 33(038)-23280. July 1953. PB 114343. Order from LC Mi $2.50, Ph $5.25.

Theoretical analysis of the published data pertaining to simple and complex fluorides suggested that it would be possible to produce certain fluoride and oxyfluoride complexes with melting points high enough to render them infusible at the temperature of molten titanium. Because of their potentially high chemical stability and the noncontaminating nature of the products of reaction between fluorides and titanium, fluorides seem particularly desirable as possible container materials for molten titanium.

Attempts were made to prepare several synthetic oxygluoride minerals, as well as a number of new oxyfluoride compounds. Though it was possible to produce highly refractory fluoride ceramics, success was not attained in producing the exact compound desired. Nor was the product completely stable within the required temperature range. An analysis of the results suggests that the optimum fabrication technique was not achieved.

Under the handicap of pronounced instability, a number of the complex fluoride refractories produced were nevertheless superior to zirconia in resistance to molten titanium. However, a product suitable for industrial application has not yet been produced.

American Electro Metal Corporation. TR 54-13.
INVESTIGATION OF THE EFFECT OF RAW MATERIAL PRODUCTION VARIABLES ON THE PHYSICAL AND CHEMICAL PROPERTIES OF CARBIDES, NITRIDES, AND BORIDES. Herman Blumenthal. AF33(616)-89. February 1954. PB 121110. Order from OTS $1.75.

The purpose of this project is to investigate the effect of raw material production variables on the physical and chemical properties of carbides, nitrides, and borides. In the investigations carried out so far, this aim has been narrowed down to the study of titanium carbide.

Ferrous titanium carbides, produced commercially by as many different procedures as possible, have been purchased and are being used for this investigation. The various materials have been analysed spectrographically, chemically and by x-ray diffraction to determine the amounts and kinds of impurities peculiar to the production technique employed. Changes of chemical composition during ball milling, hot pressing and other processing steps leading to the production of solid pieces have been followed by the same analytical techniques as well as metallographic studies.

The question of the effect of the presence of impurities has been approached from two sides, namely (1) by removing and (2) by adding impurities. Some impurities, such as iron, graphite and oxide films, have been partly removed by purification treatments like leaching with acids and flotation. Other impurities, such as TiO, TiN and graphite, have been added to pure TiC.

The effect of various ball milling media on the chemical composition of the milled product has been studied.

In the production of unbonded TiC bars, hot pressing has been used exclusively, while Ni-bonded bars have been produced (1) by hot pressing and (2) by cold pressing followed by sintering either under a protective atmosphere or in vacuum.

Infiltration experiments have been conducted as an attempt to develop a test procedure for the evaluation of quality of titanium carbide powders.

Hot pressed unbonded bars have been tested for the following properties: maximum density obtainable, ability to infiltrate, and electrical resistivity.

It has been found that the chemical composition of TiC produced by different procedures varies as to combined and free carbon, oxygen and nitrogen content as well as metallic impurities.

The amount and kind of impurities present influence maximum density obtainable by hot pressing unbonded bars, and their ability to infiltrate.

The amount of iron and oxygen picked up during ball milling in a steel mill varies greatly with the ball milling medium.

Ni-bonded bars of high density could be produced so far only by hot pressing. Cold pressing followed by sintering in hydrogen or in vacuum resulted in bars of low density.

The ability of a porous bar to infiltrate depends on its density, impurity content, and particle size of the original powder.

University of Illinois. TR 54-27.
THE EFFECTS OF PRE-COMPRESSION ON THE THERMAL SHOCK RESISTANCE OF PURE OXIDE CERAMICS. Darwin Marshall and Dwight G. Bennett. AF 33(616)-87. May 1954. ASTIA Document No. AD 38146. PB 135072. Order from LC Mi $2.70, Ph $4.80.

A combination thermal cycling and compressive loading apparatus was constructed for investigating the effects of pre-compression on the thermal shock resistance of a pure oxide ceramic body.

It was found that selective compressive loads applied in the direction of the long axis of a rectangular bar composed of alpha alumina effectively increased its thermal shock resistance. This was indicated by a progressive increase in the average modulus of rupture of each of several groups of bars; each group being uniformly thermal shocked at a compressive load of 1000, 3500 and 7000 psi, respectively. It was also found that about one half of the total improvement was obtained with the initial load of 1000 psi.

The sonic technique was used as a method of detecting flaws in thermal shocked but unloaded specimens. When specimens were thermal shocked in compression, however, the sonic method, as used in this investigation, was unable to indicate the presence of flaws.

National Bureau of Standards. TR 54-1.
PHYSICAL CHARACTERISTICS IN TITANIUM-CARBIDE-BASE CERMETS AT ELEVATED TEMPERATURES. Friedrich P. Knudsen, R. Eugene Moreland and Roman F. Geller. AF 33(616)-52-2. May 1954.

The project's principal purpose was to determine, at temperatures of 1200°F to 2000°F, the creep behavior and strength of cermets which may find application in the high temperature areas of aircraft.

Initially a series of bending tests were made on variations of Kennametal's composition K151A. The variations contained from 5% to 30% nickel bonding. The 5%, 15% and 20% compositions were recommended, on the basis

of their creep resistance and high strength, for further long-term tensile creep tests.

A limited number of tensile creep tests were made on Kennametal's compositions K151A-20% nickel, K151A-25% nickel, K152A, K162B and on Firth Sterling's composition, FS-27. The cermet K162B was deemed the most promising of the tested compositions, with respect to the properties of creep resistance and high strength. Metallographic examinations were made of all the tensile tested specimens and, in general, the test strains had been too small to cause any noticeable deformation or changes.

Thermal expansion determinations were made on Kennametal's compositions K138, K138A, K151 and K151A. The coefficients of expansion between room temperature and 1205°C ranged from 8.1 $X10^{-6}$ to 8.6 $X10^{-6}$

American Electro Metal Corp.
WADC TR 52-291 (Part 2).
AN INVESTIGATION OF VARIOUS PROPERTIES OF NIAL. W. H. Hess. AF 33(038)-10716. August 1954. PB 121022. Order from OTS $1.50.

Work with (NiAl + 5% Ni) has been continued. Stress-to-rupture specimens 9-1/2 in. long have been prepared, and various methods of preparation are discussed.

The addition of "stiffeners" to increase strength at high temperatures has been tried. These additions usually act to decrease the oxidation resistance considerably.

A new aluminide, TiAl, is investigated.

WADC TR 53-37.
CERAMIC COATING CONFERENCE 27 and 28 MAY 1952. Robert J. Brinkman. September 1954.

Ceramic coatings are presently being used on various aircraft components such as inner liners, tailcones, turbosuperchargers, etc., for the protection of the metal from oxidation at elevated temperatures.

Various types of laboratory testing procedures for the evaluation of these coatings are presented. Tests for adherence, thermal shock resistance, oxidation and corrosion resistance are a necessity for proper evaluation of the coatings.

Progressive versus single-step testing should be considered in each phase of the evaluation and the variables in each test should be recognized. Quality control measures presently being taken in the production of ceramic coated exhaust components are described. Metallographic examination of the ceramic coating-metal interface for effects of carbon absorption, surface decarburization and intergranular corrosion is another method of evaluating the protective value of the coating.

An outline of the Tentative Testing Procedure decided upon at the conference is included in the Appendix.

Pennsylvania State University.
WADC TR 53-9 (Part 2).
REFRACTORY MATERIALS FOR USE IN HIGH-TEMPERATURE AREAS OF AIRCRAFT. Norman R. Thielke. AF 33(038)16375 (17284). September 1954. ASTIA Document No. AD 48907. PB 130809. Order from LC Mi $3.60, Ph $9.30.

During this research period the emphasis on oxide refractory development continued from the previous period; the study of selected carbide systems and examination of thermal shock testing procedures was initiated.

The work with oxide-base materials included investigations of (1) the effects of various metal oxide additions on the crystallographic inversions in SiO_2, $AlPO_4$, and ZrO_2; (2) the temperature dependence of the modulus of elasticity determined by the sonic method; (3) the improvement of the mechanical strength and thermal stability of aluminum titanate bodies; and (4) the thermal conductivity, thermal shock resistance, and thermal expansion behavior of aluminum titanate.

Among RO_2-type oxide additions to SiO_2, only TiO_2 was effective in suppressing the low-to-high inversion of cristobalite, the temperature being reduced from 225°C to approximately 120°C. Mixtures of $AlPO_4$ and SiO_4 had lower inversion temperatures than either end member by itself. SiO_2 had little effect on the inversion temperature of ZrO_2, but the abrupt shrinkage during inversion was substantially eliminated by 0.10 and 0.20 mol substitutions. Additions of TiO_2 and MnO_2 to ZrO_2 lowered the inversion temperature from 1050°C to approximately 850°C.

Curves for the temperature variation of dynamic modulus of elasticity determined by a sonic method were presented for the following materials: 75-S aluminum alloy, SAE 1010 steel, fused silica, Armco iron, soft glass, Linde sapphire, Magnesia, titanium dioxide, magnesia spinel, steatite, and a vitrified and semi-vitrified whiteware body.

The effects of the following variables on the strength and thermal expansion of aluminum titanate bodies were investigated: (1) excess alumina, (2) addition of manganese compounds, (3) milling media, (4) addition of soluble ammonium compounds, (5) oxidizing and reducing firing conditions, (6) source of Al_2O_3 and TiO_2, and (7) reheating. Regardless of firing treatment or any other variable, it was found that the lower the thermal expansion of aluminum titanate the lower the transverse strength and vice versa.

It was shown that aluminum titanate may be stabilized by isomorphous substitutions of iron titanate and magnesium dititanate. Test bodies stabilized by incorporation of 25 weight percent of iron or magnesium isomorphs had low thermal expansion, but also low strength and excessive crystal growth.

The thermal conductivity of aluminum titanate rings was measured, the mean value being approximately 1.11 kcal/m°C.hr. Using the ring test apparatus, the rings were not broken at a Δ T of 428°C, which was the maximum temperature difference obtainable in the apparatus. The static thermal stress resistance of aluminum titanate was shown to be superior to that of other typical ceramic bodies.

Measurement of directional thermal expansion of aluminum titanate by high temperature x-ray powder methods gave coefficients of + 118, + 194, and 26 x 10^{-7} for the a, b_1 and c coordinate directions in the interval 25° to 970°C. The extreme expansion anisotropy was believed to be responsible for the low strength, low net expansivity, and high thermal shock resistance of aluminum titanate.

The work concerning carbides mainly involved construction of a Gartland-type carbon tube resistor furnace for investigation of compositions in the system TaC-NbC-TiC. It was planned to obtain intercarbide bodies by reactions between carbides, between equivalent metal mixtures and carbon, and between equivalent oxide mixtures and carbon.

It was shown that two factors must be used to characterize the thermal shock resistance of materials, the thermal conductivity, k, and the thermal stress resistivity, $\frac{E\alpha}{S}$. A method for the determination of these factors was described.

The Wright Field simulated service test for turbine blade materials was evaluated in terms of blade failures of seven types.

The predominance of transverse ruptures at a considerable distance from the ends of the blades suggested the possibility of a theoretical treatment of the failures. Thermal stresses in turbine airfoil shapes were approximated by use of a substitute body comprising a plate with rounded edges.

National Bureau of Standards.
WADC TR 54-457.
DEVELOPMENT OF MATERIALS FOR TRANSMITTING INFRARED ENERGY. F. W. Glaze, Webster Capps, Douglas Blackburn. AF 33(616)53-16. March 1955. ASTIA Document No. AD 75594. PB 135542. Order from LC Mi $4.80, Ph $13.80.

Ohio State University Research Foundation.
WADC TR 53-287.
STUDY OF THE SYSTEMS TiC-SiC-B_4C AND TiC-VC-ZrC. O. E. Accountius, R. F. Stoops, H. E. Konrad, H. M. Greenhouse, C. C. McBride. AF 33(038)16911. March 1955. ASTIA Document No. 75506. PB 133061. Order from LC Mi $9.30, Ph $31.80.

American Electro Metal Corp.
WADC TR 52-291 (Part 3).
AN INVESTIGATION OF VARIOUS PROPERTIES OF NICKEL ALUMINIDE. PART 3. INVESTIGATION OF THE INTERMETALLIC COMPOUNDS OF ALUMINUM. W. H. Hers. AF 33(038)10716. April 1955. PB 121023. Order from OTS $2.00.

Modification of NiAl by the addition of sirconium or titanium has produced products with improved high temperature properties without decreasing the oxidation resistance.

NiAl + 4% Zr is so far the best, having an excellent oxidation resistance - a weight gain of 2.9 mg/cm^2 after 260 hours in air at 1000°C - a transverse rupture strength of 130,000 psi at room temperature and 160,000 psi at 1100°C, and a stress rupture life of 100 hours at 1000°C and 12,000 psi.

Impact strength of this material varies considerably, needing much development work to achieve consistency. The best results in the NACA drop impact tests are over 15 inch-pounds.

These materials can be handled by hot pressing, cold pressing and sintering, slip casting, hydrostatic pressing, with machining in the pre-sintered state making complicated shapes possible.

A method of homogenising and purifying gamma TiAl was developed, and the first physical tests were made with this improved material.

Ohio State University Research Foundation.
WADC TR 54-33.
PRELIMINARY MICROSCOPIC STUDIES OF CERMETS AT HIGH TEMPERATURES. E. T. Montgomery, T. S. Shevlin, H. M. Greenhouse, H. W. Newkirk. AF 33(038) 16911. April 1955. PB 131470. Order from OTS $1.25.

New York State College of Ceramics.
WADC TR 54-414.
THE PRESSURE-CARBONIZATION OF CARBON BONDED SILICON CARBIDE-GRAPHITE FOR USE IN UNCOOLED ROCKET NOZZLES. B. R. Goss, T. L. Charland, J. R. Tinklepaugh. AF 33(616)2007. April 1955.

The resistance to flame erosion of AF-ed 410, a carbon bonded SiC-Graphite composition, was improved by the substitution of larger grain sized SiC for the "Settling tank fines" previously used. A new method of forming and firing carbon bonded SiC-Graphite was developed in which the material was carbonized under pressure in stainless steel dies. The process resulted in a product with 15% higher density and 40% lower porosity than that previously obtained. This improvement in properties also resulted in greater resistance to flame erosion.

Battelle Memorial Institute.
WADC TR 53-50 (Part 1).
BEHAVIOR OF BRITTLE-STATE MATERIALS. O. K. Salmassy, W. H. Duckworth, A. D. Schwope. AF 33(038)8682 June 1955. PB 111987. Order from OTS $4.00.

The factors influencing the fracture of brittle ceramic materials were studied: the effects of size and stress state were given primary consideration. In addition, initial consideration was given to the effects of strain rate and temperature.

The strength of plaster of Paris was found to decrease with an increase of size in the simple stress states of tension, compression, bending, and torsion. Initial analyses indicated that Weibull's statistical theory of strength could be used to predict the observed effects of size and stress state on the strength of plaster.

The effects of combined stresses on the fracture strength were studied by means of tests conducted on cylinders of plaster subjected to internal pressure and axial loading. Initial analyses of data from these combined-stress tests indicated that fracture data could be analyzed using the elastic theory of thick-walled cylinders.

The effect of superposed bending stresses on tension-test data was analyzed using Weibull's theory. This analysis indicated that superposed bending stresses should increase the observed tensile strength of a brittle material. Tension data on plaster agreed qualitatively with this prediction.

Analysis of the standard compression test indicated that fracture data from this type of test were unreliable and that the standard compression test could not be used in a research program where precise quantitative fracture data were required.

Exploratory studies were made of the effect of varying the strain rate or the stress rate on the fracture of plaster of Paris. These studies indicated a decrease of fracture stress with increased rates of loading, an effect opposite to that reported in the literature for other brittle materials. The relation between the effects of rate of loading and stress duration (static fatigue) was considered.

Battelle Memorial Institute.
WADC TR 53-50 (Part 2).
THE BEHAVIOR OF BRITTLE-STATE MATERIALS. Omar K. Salmassy, Edward G. Bodine, Winston H. Duckworth, George K. Manning. AF 33(038) 8682. June 1955. PB 121002. Order from OTS $4.25.

The fracture of brittle-state materials, primarily brittle ceramics, was studied. The principles for the selection, evaluation, and design of brittle materials from a statistical or probability viewpoint were set forth.

The distribution curve of fracture stresses was found to give an adequate description of the strength of a brittle body under a given set of conditions.

The mean fracture stress of a brittle body was found to be inadequate for the design of a brittle structure.

The factors influencing the fracture of brittle ceramic materials were studied, including the effects of size,

stress state, strain rate and delayed fracture, temperature, and flaws.

The entire distribution curve of fracture stresses was indicated to be a function of the size and stress state of a brittle body. All the materials investigated, glass, porcelain, nickel-bonded titanium carbide, plaster of Paris, and embrittled steel showed the same qualitative effect of size and stress state.

Weibull's statistical theory of strength predicted the effect of size, and the effects observed in the simple stress states of tension, bending, and torsion. Weibull's theory was not adequate, however, for predicting the effects of combined stresses.

The research indicated that static-fatigue data will require statistical analysis before they are safe for use in the design of certain ceramics under sustained loads. The materials studied exhibited an increase in strength with increasing strain rate.

The Pennsylvania State University.
WADC TR 54-467
REFRACTORY MATERIALS FOR USE IN HIGH TEMPERATURE AREAS OF AIRCRAFT. Norman R. Thielke. AF 33(616)-199. July 1955. PB 121046. Order from OTS $2.50.

The fracture patterns of TiC-Cermet stator blades which had been exposed to a simulated service test and the microstructures of these materials were investigated. An attempt to correlate fracture patterns and microstructures was unsuccessful.

The oxidation behavior of cermets was studied theoretically and experimentally. Theoretically, the tendency to oxidize was derived from an oxidation free energy plot for the reactions representing the complete oxidation of Ti-, Nb-, Ta-, Si-, Fe-, Co-, Ni- and Cr-carbides. Experimentally the oxidation behavior of carbide and metal powders was investigated using the differential thermal analysis technique. The oxidation resistance of carbide powders increased approximately in the following order: VC, TiC, NbC, B_4C, WC, ZrC, TaC, Cr_xC_y, SiC. A similar classification has been found for metal powders with Cr and Si ranking best.

The ternary system TiC-TaC-NbC has been investigated, using sintering temperatures up to 2530°C. The reactions were almost complete, indicating a complete solid solution throughout the system. Of all the sintered compositions in this system, TiC exhibited the greatest oxidation resistance.

The sintering and shrinking behavior of oxide mixtures simulating the oxides which are being formed during the oxidation of carbide Cermets, has been studied in search for compositions which form a dense and adherent oxide film.

Aluminum titanate bodies with additions of alumina, manganese carbonate and cesium Pyrex glass showed a modest increase in strength which was offset by an increase in thermal expansion.

An analysis of the applicability of thermal shock tests has been made. It follows that such tests are indispensable for the empirical determination of the maximum stresses as well as for a check on the theory of thermal shock. However, they are not suited as material tests.

A static method to measure the two thermal stress resistance factors R and R' is described and measurements on porous and dense titania, steatite, cordierite and β spodumene bodies are presented.

Stanford Research Institute.
WADC TR 54-491, Part 2.
INVESTIGATION OF CARBON (GRAPHITE) BASE MATERIALS SUITABLE FOR ROCKET AND RAM-JET APPLICATIONS. William D. Smiley. AF 33(616)-2023. November 1955.

The details of the laboratory preparation and laboratory performance of carbon base materials developed for rocket nozzle applications are reported. Rocket nozzles were fabricated from these materials for evaluation at the WADC and Bell Aircraft Corporation rocket test stands.

The most promising nozzle material developed in this program is zirconium-impregnated graphite. The formation of an adherent refractory skin of zirconium dioxide on the surface during operating service is responsible for the dimensional stability of this type of nozzle.

Firth Sterling, Inc. WADC TR 55-327.
AN INVESTIGATION OF THE FEASIBILITY OF PRODUCING METAL BONDED CARBIDE BODIES BY THE EXTRUSION PROCESS. William W. Wellborn. AF 33(616)-229. December 1955. ASTIA Document No. AD 93170. PB 136766. Order from LC Mi $3.00, Ph $6.30.

The main purpose of this investigation was to determine if useful metal-bonded carbide objects could be produced by the extrusion process. A further aim of this investigation was the comparison of the properties of such objects as might be produced by extrusion, with the properties of objects of the same composition produced by pressing, shaping, and sintering. The principal equipment used was a 1050-ton extrusion press, which had been built to extrude carbide charges.

The investigation revealed that it was possible to extrude these bodies into some shapes. However, shapes with irregular cross sections, or thin, sharp edges, were extremely difficult to extrude and it was impossible to hold size. The physical properties of extruded materials were found to be measurably lower than materials compacted by conventional means. The incidence of flaws in extruded material was found to be greater.

The Ohio State University Research Foundation.
WADC TR 54-33, Part 2.
PRELIMINARY MICROSCOPIC STUDIES OF CERMETS AT HIGH TEMPERATURES. Thomas S. Shevlin, Herbert W. Newkirk, E. George Stevens, Harold M. Greenhouse. AF 33(038)-16911. February 1956. PB 136184. Order from LC Mi $7.20, Ph $44.80.

SECTION 1 - CHANGE IN THE MICROSTRUCTURE OF SOME TiC BASE CERMETS, WITH TIME, AT HIGH TEMPERATURES

The effect of heat treatment in a vacuum or inert-gas atmosphere at 2000°F on the microstructure of Kentanium samples K-151-A and K-152-B is discussed. Equipment and techniques employed in carrying out the research are discussed in detail. The effects of a solution-reprecipitation mechanism are discussed and a tentative theory is advanced relating the observed changes in microstructure to the steep slope of the stress-rupture curves for these cermets.

SECTION 2 - STUDY OF SOME TiC BASE CERMETS BY MEANS OF HIGH TEMPERATURE X-RAY DIFFRACTION TECHNIQUES

High temperature x-ray diffraction studies on phases stable at 2000°F in samples K-151-A and K-152-B and evidence for the solid solubility of TiC in Ni at this temperature are presented. In addition, the thermal expansion

data of Ni from room temperature of 2000°F has been determined by high temperature x-ray techniques. The equipment used and the techniques employed in this research are discussed in detail. Evidence is presented which indicates that a solution-precipitation mechanism is responsible for the microstructure changes observed and reported in Section 1.

SECTION 3 - ELECTRON MICROSCOPY OF SOME TiC BASE CERMETS

A new electron microscopy replication technique is presented and its application in resolving the microstructure of sample K-152-B is discussed. The colloidal dispersion present in the Ni phase of this cermet had been resolved and found to be small particles of TiC. The origin of this precipitate and its effects on the high temperature strength of TiC-Ni cermets are discussed.

SECTION 4 - COEFFICIENT OF EXPANSION STUDIES ON HIGH TEMPERATURE ALLOYS

The relationship between thermal shock resistance and the linear thermal expansion of the phases composing a cermet is discussed. Techniques and apparatus for fabricating and testing alloys have thermal expansion characteristics similar to titanium carbide are presented. Thermal expansion data for the systems Fe-Co-Ni, Fe-Co-Ni-Cr, Cr-Mo-Ni, TiC/Fe-Co-Ni and TiC/Fe-Co-Ni-Cr are listed.

SECTION 5 - PREPARATION OF HIGH Ni-LOW TiC CERMETS FOR IMPACT AND THERMAL SHOCK STUDIES

Techniques and apparatus for the fabrication of TiC-Ni cermets suitable for impact and thermal shock studies and containing 73.1% Ni, 80.9% Ni, 87.9% Ni and 94.2% Ni by weight are given.

SECTION 6 - INVESTIGATION OF TiB_2 and $MoSi_2$ AS THE CERAMIC COMPONENT OF A CERMET

The possibility of using a mixture of TiB_2 and $MoSi_2$ as components of a new high temperature cermet is discussed.

SECTION 7 - STUDY OF THE EFFECT OF HEAT TREATMENT ON IMPACT STRENGTH OF K-151-A AND K-151-B

The effect of heat treatment of the impact strength of Kentanium samples K-151-A and K-151-B is discussed. Apparatus and techniques employed in this research are discussed in detail.

Stanford Research Institute.
WADC TR 54-491.

INVESTIGATION OF CARBON (GRAPHITE) BASE MATERIALS SUITABLE FOR ROCKET AND RAM-JET APPLICATIONS. William D. Smiley. AF 33(616)-2023. August 1956.

The details of the preparation, application, and testing of high temperature, oxidation and erosion resistant materials for nozzles are reported. A composition whose starting components were 4 mol percent molybdenum - 52 mol percent silicon - 44 mol percent carbon and which was prepared from a mixture of 75 weight percent silicon carbide and 25 weight percent molybdenum disilicide, has shown promising high temperature properties, including good oxidation resistance and refractoriness. Solid nozzles have been prepared from this material, together with liners for graphite nozzle, for testing of the WADC rocket test station.

The Beryllium Corporation.
WADC TR 57-86.

HOT PRESSING OF BERYLLIA ROCKET NOZZLES. J. K. Evans, G. D. Kriebel. AF 33(616)-3699. January 1957. ASTIA Document No. AD 132567. PB 140363. Order from LC Mi $2.40, Ph $3.30.

The effects of time, temperature, pressure, and particle size on the density of hot pressed beryllium oxide were investigated. Experimental conditions and densities obtained are tabulated for two types of beryllium oxide and their mixtures. A description of the equipment, procedures, and starting materials is included. The fabrication of rocket nozzles according to the best procedures is described. Photomicrographs of hot pressed beryllia of maximum density are shown. A limited study of the machining of hot pressed beryllia is included.

Sylvania Electric Products, Inc.
WADC TR 56-329.

IMPROVEMENT OF THE IMPACT RESISTANCE OF CERMETS. A. C. Pezzi, H. P. Kling. AF 33(616)-2911. April 1957. ASTIA Document No. AD 118195. PB 131093.

This program investigated the possibility of improving the impact resistance of cermets by the application of a well bonded ductile metallic coating to the surface. Commercially available bars of K161B were used as a base cermet. Coating methods investigated included diffusion bonding of electro deposited nickel, infiltration of porous sintered coatings, direct application of high temperature brazes, diffusion bonding to shaped tubing and vacuum casting. The application of 0.018" of electrodeposited nickel, bonded by a suitable vacuum heat treatment, raised the impact strength from values of 2.56 and 3.36 in-lbs at room temperature and 1800°F respectively to values of 21.48 and 18.96 in-lbs. at the same temperatures. Limited test results indicated that diffusion bonding of shaped tubing also raised the impact strength appreciably. The infiltrated coatings showed promising structures when examined metallographically, but the impact strength was essentially unchanged with the compositions used. It is possible that other compositions might be more beneficial. The direct application of brazing alloys was deemed unsatisfactory. The vacuum cast coatings had interesting microstructures but were not evaluated.

Vitro Laboratories. WADC TR 56-521.

PREPARATION OF PROTECTIVE COATINGS BY ELECTROPHORETIC METHODS. Allen C. Werner, James J. Shyne, Harold N. Barr, Bernard Triffleman. AF 33(616)-3118. April 1957. ASTIA Document No. AD 118224. PB 131062.

An investigation was conducted of the preparation of oxidation-resistant coatings for molybdenum by electrophoretic methods. Multilayer coatings of 80% nickel-20% chromium and nickel-bonded chromium carbide provided good static air-oxidation resistance. Inclusion of a 50% dense nickel-chromium layer resulted in satisfactory ballistic impact resistance. Erosion was fair. All the test panels failed under severe thermal shock.

Electrokinetic impregnation of graphite to provide increased oxidation resistance was unsuccessful, a maximum impregnation of 2.1% by weight being obtained. The effect of varying suspension concentration, impregnation voltage, periodic reversal of the current, activators for the graphite and the suspensions and the use of ultrasonic energy were studied. Impregnation up to 2.6% by weight was obtained by pumping a slurry through graphite without application of any EMF.

The New York State College of Ceramics
WADC TR 57-39.

CERAMIC REINFORCED ALLOYS AND PLATED CERMETS. M. T. Curran, R. P. Riegert, R. K. Francis, R. S. Truesdale, J. R. Tinklepaugh. Af 33(616)-2414. May 1957. ASTIA Document No. AD 130754. PB 131188.

A precision casting technique was developed for the reinforcement of super alloys with ceramic rods. Procedures were found for the electroplating of cermets with nickel and chromium to improve their impact resistance and for the chemical plating of nickel on titanium carbide grains. The effect of molybdenum as an alloying agent in the cermet binder was observed.

The Brush Beryllium Company. WADC TR 57-32.

A STUDY OF HOT PRESSING OF BERYLLIA ROCKET NOZZLES. Chester A. Bielawski, Theodore M. Harkulich, Russell E. Long. AF 33(616)-56-23. May 1957. ASTIA Document No. AD 118278. PB 131202.

A preliminary investigation was conducted on the variables of particle size, temperature, pressure, and time as related to the fabrication of beryllia rocket-nozzle throat inserts by hot pressing. Results of this work were applied toward the fabrication of twenty, 100-pound-thrust throat-insert specimens which were submitted for evaluating the feasibility of using this material in uncooled rocket motors. The twenty specimens were composed of four groups representing variations in starting materials with regard to particle size and purity and variations in fabrication procedures, including time, temperature, and heat treatments, which affected the final structure.

Bausch & Lomb Optical Co. WADC TR 55-500 Pt I.

AN INVESTIGATION OF INFRARED TRANSMITTING MATERIALS. N. J. Kreidl, H. C. Hafner, J. R. Hensler, R. A. Weidel, E. C. Letter. AF 33(616)-2769. July 1957. ASTIA Document No. AD 130904.

The development of calcium aluminate glasses permitting useful transmission out to about 6 microns and having satisfactory working properties to permit normal castforming procedures in 10 pound melts is described. These glasses have high softening temperatures, thermal expansion coefficients equivalent to soda-lime-silica glasses, and can be polished by standard techniques. Investigations of other glass systems are discussed.

A fundamental study to determine if pressed and sintered compacts of small crystals can be made into suitable infrared transmitters is described. Work has been limited to a few materials which transmit infrared energy in their single crystalline forms. Relationships of material preparations, particle sizes, sintering techniques, and transmissions obtained are discussed.

Sintercast Corporation of America. WADC TR 57-135.

A STUDY OF GRADED CERMET COMPONENTS FOR HIGH TEMPERATURE TURBINE APPLICATIONS. Henry W. Lawendel, Claus G. Goetzel. AF 33(616)-3670. August 1957. ASTIA Document No. AD 131031. PB 131434.

The purpose of this investigation is a preliminary study of graded cermet turbine components designed to improve the ductility and toughness of the root and airfoil tip sections. Ballistic impact tests at low and high temperature, before and after heat shock cycling, were performed on a wedge-like simulated turbine bucket shape divided into three general areas representing the root, airfoil tip, and airfoil body sections of a turbine bucket. The wedges were produced of materials believed to have a satisfactory strength to withstand the temperatures and stresses prevailing in high temperature gas turbine operation: For the cermet core of the bodies, a cermet was selected on the basis of preliminary tests which consisted of titanium carbide infiltrated with molten Inconel-X; for the metal-rich layers applied to the root and airfoil tip sections, an alloy was selected consisting of Inconel-X enriched with titanium carbide. The core material had a carbide concentration corresponding to 61-63% of theoretical carbide density as measured on skeleton bodies prior to infiltration. The stress to withstand 100 hours life at 1800°F of this core material was found to be in the order of 12,000 asi. The metal-rich alloy layers analyzed approximately 10% by weight of titanium carbide; the stress of this material to withstand a 100-hour life at 1600°F was found to be in the order of 20,000 psi.

The graded layers were applied during the infiltration of the wedge by filling with an excess of infiltrant a gap formed between the skeleton and an investment mold used in the infiltration process. Thus a structure was obtained in which the graded surface layer constitutes a continuation and extension of the nickel alloy matrix of the cermet core containing dispersed titanium carbide inclusions. The test results indicate a substantial improvement to be gained in ballistic impact strength by providing metal-enriched areas at the edges and tip of the airfoil. An oxidation-resistant ductile alloy layer deposited as a cladding on the entire surface of the simulated turbine component resulted in an equally important improvement of the thermal shock resistance of the titanium carbide cermet bodies.

Marquardt Aircraft Co. WADC TR 57-577 Pt I.

DEVELOPMENT AND EVALUATION OF INSULATING TYPE CERAMIC COATINGS PART I. Development and Small Scale Testing. S. Sklarew, C. A. Hauck, A. V. Levy. AF 33(616)-2957. Feb., 1958. ASTIA Document No. AD 150957.

Eight systems of metal reinforced refractory coatings were investigated. These systems were based on sodium silicate, frit, aluminum phosphate, and L-389, binders with refractory grain fillers and they were designed to insulate aircraft structural members operating in the thermal range of 2000° to 3000°F.

Thermal drops of as high as 10°F per thousandth inch of coating thickness at equilibrium were observed with lag times up to 60 seconds to attain equilibrium under laboratory test conditions.

P. R. Mallory & Co., Inc. WADC TR 57-665.

DEVELOPMENT AND EVALUATION SERVICES ON CERAMIC MATERIALS AND WALL COMPOSITES FOR HIGH-TEMPERATURE RADOME SHAPES. John J. Dorsey. AF 33(616)-3397. February 1958. ASTIA Document No. AD 150965. PB 131987.

Since the speeds of aircraft and guided missiles are increasing, the present organic base radomes are becoming obsolete. Various flame spraying methods which might be used to fabricate a radome from inorganic or ceramic materials were investigated.

Numerous compositions were mixed and flame sprayed. Silica, alone or with additions, was the only material which could be fabricated into a hard, dense deposit. Alumina and other miscellaneous compositions were also flame sprayed but in all cases, the deposits were either full of laminations and voids or cracking occurred on cooling.

Means of obtaining more uniform deposits were investigated. These included obtaining a more uniform powder flow and increasing flame temperature both by additions to the powder and by external means. Linde "flame plated" alumina deposits were also investigated.

Rain erosion specimens and flat plates were flame sprayed and machined.

Bars were cut from large flame sprayed deposits in order to observe the uniformity obtained with the flame spray technique.

Flame spraying onto silica cloth is also discussed. Flame polishing in conjunction with this method produces a much smoother surface than when the silica is sprayed directly on a carbon block. Several radomes were also flame sprayed using a silica cloth base.

Apparently, the most valuable application of flame spraying would be to patch, adjust electrical thickness or to repair rain erosion damage.

A brief, but comprehensive, summary of all experimental results is given at the end of the report.

University of Illinois. WADC TR 58-12 Pt I.
HIGH-TEMPERATURE ELECTRICAL INSULATING INORGANIC COATINGS ON WIRE. Clifton G. Bergeron, Arthur L. Friedberg, Paul F. Schwarzlose, Robert J. Beals, William M. Faber. AF 33(616)-3943. March 1958. ASTIA Document No. AD 151079. PB 131811.

An investigation was undertaken to develop and evaluate high-temperature electrical insulating inorganic coatings for copper wire, and to develop methods for applying these materials to wire.

Test procedures were developed for determining the dielectric strength, dielectric constant, and dissipation factor of experimental coatings on both sheet copper and copper wire.

Certain coating compositions formulated in the alkali-B_2O_3-SiO_2 system were found to have the requisite dielectric properties at room temperature.

Equipment was designed and constructed for continuously applying and firing vitreous coatings onto copper wire. The considerations relating to flexibility and dielectric properties of the coated wire are discussed.

Georgia Institute of Technology. WADC TR 58-13 Pt I.
HIGH-TEMPERATURE INSULATION FOR WIRE. J. D. Walton and J. N. Harris. AF 33(616)-3944. March 1958. ASTIA Document No. AD 151062. PB 131812.

A literature survey was undertaken to review possible methods for use in developing an electrical insulating coating for the high temperature protection of wire.

Ceramic-organic coatings were developed which cover the range from room temperature to 1300°F when applied directly to metal. These coating withstand 1500° -- but a wetting problem was encountered with the enamel on metals. However, since the final ceramic organic coating is applied over a base coating of aluminum oxide the wetting problem is not expected to cause difficulty. The effect of solution of aluminum oxide in the enamel is being investigated.

Curing studies have shown that methods of curing resin-frit coatings is an improtant parameter in obtaining proper burn-out temperature of resins.

Aluminum phosphate and silica were tested briefly as possible base coatings for the final ceramic-organic coating but adherence and corrosion problems caused this work to be discontinued.

Several anodizing baths have been tried, among them boric acid, oxalic acid, and sulfuric acid. Among these, sulfuric acid seems to give the best anodised coating.

Application of aluminum to copper wire has been the area where the most difficulty has been encountered. Although coatings with very good adherence have been obtained, first indications were that the problem in plating seemed to be in pre-cleaning of the copper. For this reason, gold plate was applied as an initail coating in some tests prior to aluminum plating the copper.

WDAC TR 58-184.
A SURVEY OF THE THEORIES CONCERNING CERAMIC TO METAL ADHERENCE. A. E. Eagles, A/2c. August 1958. ASTIA Document No. AD 155774.

Various theories have been offered as to the mechanism of ceramic coating to metal bonding. To date, however, no one theory has proved to explain the phenomenon adequately. This report reviews the literature pertinent to the subject.

Bausch & Lomb Optical Co. WADC TR 58-275 Pt I.
FABRICATION OF INFRARED TRANSMITTING MATERIALS BY HOT PRESSING TECHNIQUES. Norbert J. Kreidl, Harold C. Hafner, Joseph R. Hensler, Eugene C. Letter. AF 33(616)-5123. October 1958. ASTIA Document No. AD 202844. PB 150149. Order from LC Mi $8.10, Ph $27.30.

The fabrication of infrared transmitting materials by hot pressing techniques has been studied. BaF_2 was used as a model material. The temperatures studied ranged from 600 to 1225°C and the pressures from 1500 to 8000 psi. The equipment used to obtain these conditions is described. The effects of various hot pressing atmospheres including nitrogen, helium, argon and carbon dioxide, were investigated. A study was carried out to identify the impurities present and to study their effects.

Bausch & Lomb Optical Co. WADC TR 55-500 Pt II.
AN INVESTIGATION OF INFRARED TRANSMITTING MATERIALS. N. J. Kreidl, H. C. Hafner, J. R. Hensler, R. A. Waidel, E. C. Letter AF 33(616)-2769. October 1958. ASTIA Document No. Ad 203786.

A limited compositional study of calcium aluminate glasses resulted in several prototype compositions which have superior handling and melting properties than those reported previously.

One of these compositions was melted successfully in a 30 lb. melt. Germanate glasses as well as several silicate glasses were studied on a limited basis.

Some mechanical and chemical property measurements of calcium aluminate glasses have been completed and are compared with conventional glasses.

An investigation of methods to inhibit surface deterioration of calcium aluminate glasses by water or water vapor has been started. Methods for eliminating the water absorption band at 2.7-3.0μ were also studied.

Various methods for compacting polycrystalline materials were studied including cold pressing, hot pressing, controlled freezing and sintering. Compacts that transmit considerable infrared can be produced by these methods. Hot pressing seems the most practical method for continued study. Infrared transmitting compacts of NaCl, BaF_2 and MgO have been produced. A mathematical relationship demonstrated the high radiation losses in a compact due to scattering induced by voids.

Bausch & Lomb Optical Co. WADC TR 55-500 Pt III.

INVESTIGATION OF INFRARED TRANSMITTING MATERIALS. Norbert J. Kreidl, Harold C. Hafner, Joseph R. Hensler, Robert A. Weidel. AF 33(616)-2769. October 1958. ASTIA Document No. AD 202842.

The water absorption band at 2.7-3.0/μ in calcium aluminate glasses is undesirable so methods to eliminate it were studied. Techniques successful in silicate glasses were not applicable to the calcium aluminates. For example, D_2O added to a silicate glass batch shifted, as expected, the water absorption band to approximately 3.7/μ. A similar addition to a calcium aluminate batch produced no change in the position of the water absorption band. Techniques were developed for protecting the surfaces of calcium aluminate glasses from deterioration by water or water vapor. These glasses were "hardened" by conventional techniques. "Hardening" did not inhibit moisture attack.

The infrared transmission of typical calcium aluminate glasses changed slightly after exposure to 1×10^7 rep gamma radiation from a Co^{60} source.

State University of New York, College of Ceramics at Alfred University. WADC TR 58-452.

METAL FIBER REINFORCED CERAMICS. R. S. Truesdale, J. J. Swica, J. R. Tinklepaugh. AF 33(616)-5298. December 1958. ASTIA Document No. AD 207079. PB 151610.

Techniques were developed for the sintering and hot pressing of alumina and alumina containing 5 wt. %, 10 wt. % and 20 wt. % molybdenum fibers. The physical and mechanical properties of alumina containing these percentages of 1/8 in. long by 0.002 in. dia. fibers were determined and compared to those of the alumina. The alumina was superior in strength and impact resistance but there was some indication that the aluminas containing 10 and 20 wt. % additions of fiber were superior in thermal shock resistance. All alumina samples containing 10 and 20% additions developed microcracks while only some 5% samples developed these cracks.

University of Utah. WADC TR 59-381. CERAMIC STUDIES SUMMARY TECHNICAL REPORT. P. Gibbs, G. S. Baker, E. K. Beauchamp, M. H. Miles. AF 33(616)-5876. August 1959. PB 161146. Order from OTS $0.50.

A. Progress is reviewed in the preparation of high purity and doped sintered alumina samples. A high-precision fast-recording, bending creep furnace with resolution of at least 10^{-6} strain in the outer fiber is described. Preliminary tests suggest a stick-slip behavior with jumps of about 5.10^{-5} strain in the outer fiber.

B. Progress in an electrical study of corundum-metal interface is reviewed. Migration of silver ions from a coating is found above 200°C on application of several volts/cm. The observed conduction current rises to an asymptotic value of nearly an amp. in seconds. The rise time for this process depends upon the temperature as well as the length of time during which the specimen "rested" without any field. The value of the asymptotic current was greatest at about 350°C.

Douglas Aircraft Co., Inc. WADC TR 59-261. INFLUENCE OF SURFACE ON CERAMIC MECHANICAL PROPERTIES. George R. Pulliam. AF 33(616)-5875. September 1959. PB 161309. Order from OTS $1.00.

This study was devoted to establishing the importance of gaseous and liquid environments, electromagnetic irradiation, and non-stoichiometry on the deformation and fracture processes in ceramic materials. The surface condition of the material and its chemical environment have been found to have a large effect on these processes. The small ductility of magnesium oxide is reduced by air and basic solutions. Acid solutions produce ductility in previously brittle magnesium oxide. Exclusion of air by a protective coating or a protective atmosphere preserves the ductility. Photochemical reactions essentially stop the creep of silver chloride. A more quantitative evaluation of these effects should be conducted to permit the establishment of the mechanisms operating.

Armour Research Foundation. WADC TR 59-316. MECHANISM OF WEAR OF NONMETALLIC MATERIALS. Edwin J. Latos. AF 33(616)-5962. October 1959. ASTIA Document No. AD 232467. PB 171474. Order from OTS $0.50.

The principal objective of this project is a basic study to evaluate the mechanism of wear of nonmetallic materials at evaluated temperatures. Materials selected for this program included single crystals of sapphire and quartz. A friction apparatus was designed for use at high temperatures and has an operating range of 0 to 50 grams load and a speed range of 10^{-3} to 10^{-1} cm/sec. The friction specimens are placed into suitable holders and heated with an induction generator.

Initial tests established the reliability of the design of the friction apparatus to operate effectively from room temperature to 1600°F. Design of suitable specimen holders should permit friction studies at temperatures to 4000°F. Sliding of sapphire on sapphire indicated an increase in the coefficient of friction with temperature to approximately 900°F and a decrease with further increases in temperatures to 1600°F. The coefficient of friction also increased with load in the temperature region reported.

The future activities of the project will include the correlation of the high temperature friction data with the Bowden and Tabor and Archard and Hirst mechanisms of wear.

Armour Research Foundation. WADC TR 59-300. RESEARCH AND DEVELOPMENT SERVICES LEADING TO THE CONTROL OF ELECTRICAL PROPERTIES OF MATERIALS FOR HIGH TEMPERATURE RADOMES. Leon M. Atlas. Af 33(616)-5929. October 1959. PB 161423. Order from OTS $1.00.

The rise of dielectric constant and loss tangent in alumina ceramics as they are heated creates serious problems in radome design. This report describes work performed on the first phase of a long range effort to reduce and control this variation in dielectric properties. During the first ten months of the program, attention was concentrated on preparing alumina ceramics of higher purity than is commercially available, and ceramics with low concentrations of added impurities. Alumina batches containing less than 100 ppm of foreign cations were prepared by the acid solution of 99.999% aluminum metal, and this material was fired to ceramic discs of similar purity. Specimens were also prepared with introduced Ca and Si at two different levels below 0.1%.

High purity specimens were subjected to annealing treatments in various gases including oxygen, steam, moist hydrogen, and dry hydrogen. Attempts were made to follow effects of these annealing treatments on the defect condition of the samples by measurements of optical reflectance and thermoluminescence after irradiation with gamma rays.

After grinding and acid leaching, alumina discs were forwarded to the Laboratory for Insulation Research at Massachusetts Institute of Technology for electric evaluation. Preliminary results from this laboratory show an appreciably smaller temperature variation of dielectric constant and loss tangent than for the purest commercial aluminas tested.

Brush Beryllium Co. WADC TR 59-29, Part I. AN INVESTIGATION OF INTERMETALLIC COMPOUNDS FOR VERY HIGH TEMPERATURE APPLICATIONS. R. M. Paine, A. James Stonehouse, W. W. Beaver. AF 33(616)-56-12. January 1960. PB 161683. Order from OTS $3.50.

Intermetallic compounds from thirty-five binary metallic systems were prepared, fabricated into oxidation-test specimens, and tested for oxidation resistance in dry air at 2300°F. Only high-melting (above 2550°F) compounds were studied. These included aluminides, beryllides, silicides, germanides, and sirconides, as well as numerous miscellaneous compounds.

The intermetallic compounds or compositions which are shown to have sufficient oxidation resistance at 2300°F to be potentially useful at this temperature include: $NbAl_3$, $TaAl_3$, $CrBe_2$, $MoBe_{12}$, Nb_2Be_{17}, $NbBe_{12}$, $TaBe_2$, Ta_2Be_{17}, $TaBe_{12}$, $TiBe_2$, $TiBe_{12}$ "WBe_5," $ZrBe_{13}$, Zr_2Be_{17}, Cr_3Si, Ti_5Si_3, "TiSi," $TiSi_2$, and $TaCr_2$.

A literature survey of intermetallic compounds is included.

Firth Sterling, Inc. WADC TR 58-432, Part II. AN INVESTIGATION OF THE MECHANICAL PROPERTIES OF CERMETS AS RELATED TO THE MICROSTRUCTURES. Ira Binder. AF 33(616)-5084. January 1960. ASTIA Document No. AD 208148. PB 151722. Order from OTS $2.25.

TiC/Ni compositions with 10, 20, and 40 percent nickel were tested for transverse rupture strengths up to 1600°C. Transverse bend, cantilever beam, hot deformation, and hot extrusion experiments were performed in order to study the onset of plastic deformation. These materials were plastically deformed, and their microstructures were studied to learn about the mode of deformation. Plastic flow of the nickel binder and alignment of the carbide grains were discovered.

Other refractory hard-metal compositions, both single-phase and cemented, were tested in transverse rupture up to 1600°C and were also subjected to various forms of hot deformation. Transverse strength peaks, versus temperature, were found for each material in the range 800-1300°C. Reasons for this behavior are developed. Microstructures of these materials were examined in connection with the strength tests and deformation studies.

Stanford Research Institute. WADC TR 59-448. MECHANICAL PROPERTY SURVEY OF REFRACTORY NONMETALLIC CRYSTALLINE MATERIALS AND INTERMETALLIC COMPOUNDS. William D. Smiley, Leon E. Sobon, Frank M. Hrus, Elden P. Farley, J. Emery Chilton, Eugene F. Poncelet, John J. Kelly. AF 33(616)-5907. January 1960. ASTIA Document No. AD 234775. PB 154846. Order from LC Mi $11.10, Ph. $51.60.

In this program, the literature on the mechanical properties of nonmetallic polycrystalline compounds and intermetallic compounds with melting points above 1500°C was surveyed. The theoretical concepts of flow and fracture were reviewed. It was determined that the mechanical properties of many potential high temperature materials have not been evaluated. Data for those which have been partially evaluated were in most cases either incomplete, or meaningless because of failure to associate the data with the structure, purity, and thermal history of the material. It was concluded that the intermetallic compounds which are brittle at room temperature and slightly ductile at high temperature (where some of them are also stronger) are more worthy of future study than the much worked-over oxides and interstitial compounds. Another conclusion of this study was that conventional parameters, such as tensile strength, etc., may not be of value for designing high temperature structural components, and that data from tests closely simulating actual use conditions are necessary before the designer can confidently use brittle materials.

Additional research is warranted in certain areas, where there is either a lack of pertinent data on compound classes, or a need for more functional information on prevention and cure of imperfections.

College of Ceramics, Alfred Univ. WADC TR 58-452, Part II. METAL FIBER REINFORCED CERAMICS. J. J. Swica, W. R. Hoskyns, B. R. Goss, J. H. Conner, J. R. Tinklepaugh. AF 33(616)-5298. January 1960. PB 161481. Order from OTS $1.25.

The principal geometric variables involved in ceramic-refractory metal fiber composites were evaluated using thermal shock resistance as the most important criteria. Several different ceramic-metal fiber composites were investigated. Using the alumina-molybdenum and alumina-mullite-molybdenum fiber systems, the comparative properties of the two basic types of composites were demonstrated. Composites were developed which had flexural strengths exceeding 30,000 psi following four severe thermal shock cycles.

Manuscript released for publication February 1961 as a WADD Technical Report.

Bell Aircraft Corp. WADC TR 59-432. REFRACTORY INORGANIC MATERIALS FOR STRUCTURAL APPLICATIONS. H. A. Pearl, J. M. Nowak, Joseph C. Conti, R. J. Urode. AF 33(616)-5930. February 1960. PB 161737. Order from OTS $2.75.

Thirty-five refractory inorganic material systems were evaluated in exploring possible techniques to prepare refractory inorganic materials into high temperature aircraft structural elements. A system as defined in this program is a specific combination of materials and structure. Twenty-one of these systems were designed and fabricated at Bell Aircraft Corporation. The remaining 14 were obtained from various other organizations and agencies. The fabrication techniques were evaluated by the results of room temperature modulus of rupture, impact and thermal shock tests. The best over-all Bell Aircraft system was based on .003 alumina-silica paper impregnated with alumina-silica cement. This system is tentatively selected for additional investigation in Phase II of this program to begin June 15, 1959. Specimens of this system were tested at elevated temperature (2000°F) for modulus of rupture, compressive strength and compressive shear.

A state-of-the-art survey on foamed ceramics was conducted in this study and is included as an appendix of this report.

Stanford Research Institute. WADC TR 59-454. SYNTHESIS OF NEW HIGH TEMPERATURE MATERIALS. J. L. Engelke, F. A. Halden, E. P. Farley. AF 33(616)-

5888. February 1960. PB 161720. Order from OTS $1.25.

A number of mixed transition metal carbides, borides, and nitrides were prepared and examined in terms of melting point, structure, and oxidation resistance. None of the measured melting points were higher than the value reported for HfC·4TaC (3940°C). The melting point for a series of solid solutions between HfC and NbC increased regularly with hafnium content.

A material with the composition Mo_2BC, when heated in an air stream, was found to lose weight rapidly without loss of metallic character, suggesting its possible use as an ablation material. A cubic modification of hafnia was obtained in material sprayed from a plasma arc. A low coefficient of thermal expansion and high melting temperature makes this a material of interest.

An implosive-shock technique for compound synthesis, which subjects materials to extremely high temperature-pressure conditions for short periods of time, was developed. The method was used successfully to synthesize boron phosphide.

Carborundum Company. WADD TR 60-124. DEVELOPMENT OF NON-OXIDIC REFRACTORY FOAMS. Ian M. Logan, Douglas C. Wise, John J. McGahan, Carl VonDoenhoff. AF 33(616)-6294. April 1960. PB 171178. Order from OTS $1.25.

Fundamental technology has been developed for the preparation of non-oxidic refractory foams of controlled pore diameter, spacing, and continuity.

Foaming procedures used in making commercially available foam plastics were employed to produce the resin-type foams which formed the basis for the final refractory foams.

It was shown that foam structures can be varied as to strength, density and pore size by varying the proportions of resins and solvents used to generate the foams. Lesser controls were also possible by varying the temperature and pressure during the foaming process. Good control and predictable results were obtained.

Foams were prepared of the following materials: carbides of titanium tungsten, molybdenum, tantalum, and columbium; borides of titanium and of zirconium; and silicides of tungsten and of molybdenum.

Battelle Memorial Institute. WADC TR 60-108. INVESTIGATION OF SINTERABLE POWDERS AND PROPERTIES OF BERYLLIA CERAMICS. James E. Johnson, A. K. Smalley, Winston H. Duckworth. AF 33(616)-6238. April 1960. PB 161891. Order from OTS $0.75.

Information was developed on the effects of processing variables and microstructure on the fracture strength of ceramics made from sinterable oxide powders. The characteristics of high strength being associated with low porosities and small average crystal sizes was observed in ceramics of both MgO and BeO, but no quantitative correlation was found. Highest strengths were obtained when the time and temperature of sintering were sufficient for densification above about 96 percent of theoretical, provided that the sintering time and temperature was insufficient to give crystal sizes larger than the smallest observed, 5-6 microns for BeO ceramics and about 10 microns for MgO ceramics.

In the case of BeO powder prepared by pyrolysis of high-purity sulfate, calcining to an intermediate temperature (1700-1800°F) gave powder that densified most readily when compacted and sintered, but one that did not have the greatest tendency toward crystal growth. Thus, this most sinterable powder produced the strongest BeO ceramic.

The lack of quantitative correlations between bulk density, average crystal size, and strength, together with appreciable scatter in strength values, indicated that strength was affected by some factor or factors other than these two. Localized areas of crystal growth found in BeO specimens may have had an influence.

Linde Co. WADC TR 60-52. RESEARCH INVESTIGATION TO DETERMINE THE OPTIMUM CONDITIONS FOR GROWING SINGLE CRYSTALS OF SELECTED BORIDES, SILICIDES AND CARBIDES. A. D. Kiffer. AF 33(616)-6326. April 1960. ASTIA Document No. AD 238061. PB 161792. Order from OTS $1.00.

This work was undertaken to produce selected crystals in the refractory hard metals class for mechanical and other property determinations. A Verneuil-type process using an arc heat source and argon shield gas was employed. Single crystal boules of titanium diboride and tungsten disilicide, 1/4-inch diameter and up to 4-1/2 inches long, were made. Most of them cracked upon cooling. The largest single crystal pieces recovered were 1/4 inch diameter and over 1/2 inch long. Dimolybdenum carbide boules had large sections of a "single crystal" Mo_2C matrix containing about 10% by volume of another phase distributed uniformly through it. No Mo_2C single crystal pieces free from this phase were made. In very limited work with ditungsten pentaboride only polycrystalline boules were produced. A major problem was encountered in getting powders suitable for Verneuil-type crystal growth. Best results were obtained from compounds prepared by fusing together commercially available pure elements and crushing the lumps into a suitable particle size fraction. Process improvements and purer powders are required to produce better quality TiB_2 and WSi_2 crystals. More experimental information is required on the molybdenum-carbon and the tungsten-boron systems.

Battelle Memorial Institute. WADD TR 60-54. CHARACTERISTICS GOVERNING THE FRICTION AND WEAR BEHAVIOR OF REFRACTORY MATERIALS FOR HIGH TEMPERATURE SEALS AND BEARINGS. Lewis B. Sibley, Arthur E. Mace, Daniel R. Grieser, C. Malcolm Allen. AF 33(616)-3995. May 1960. PB 171010. Order from OTS $1.50.

An investigation of the basic factors involved in the wear and friction of ceramics, cermets, and high-temperature alloys sliding at speeds of 100 to 200 fps and temperatures from 500 to 1000°C (1000°F to 1800°F) has been made. Bearing pressures covered a range of 5 to 50 psi. Statistical correlation of measured wear rates with friction and material properties indicates that, at these temperatures, the wear rate of ceramics and cermets may be approximately described by the following relationship:

$$\text{Wear rate} \propto \frac{\mu}{R^{1.25} D^{0.75}},$$

where μ is the coefficient of friction, R is the thermal-stress-resistance factor, and D is the thermal diffusivity of the mated material on which wear predominates, which is usually the material with the lowest thermal-stress resistance.

A mechanism of wear has been evolved based on the above correlation and on the experimental study of friction and wear surface-temperature fluctuations using special transducers and color motion-picture photography. The predominant wear mechanism in the high-speed sliding of ceramic and cermet materials appears to involve

formation of hot spots at asperity contacts and subsequent fracture of the material near these hot spots as a result of thermal stresses. In this situation the wear rate is influenced both by the configuration of the rubbing parts and by the thermal-stress-resistance properties of the materials. When one of the mating surfaces is interrupted, the total wear is greater. Wear tends to predominate on parts with interrupted wear surfaces, such as slider bearings, in comparison with their continuous mating surfaces or ring surfaces, such as in face seals, and on ceramic and cermet materials with low-thermal-stress-resistance factor and low thermal diffusivity.

Promising commercial materials for high-temperature dry sliding bearings, gas bearings, and seals include Al_2O_3-Cr-Mo cermets, SiC ceramics, and TiC-Ni-Mo cermets. One experiment with a SiC-Si_3N_4 ring stator mated against a TiC-Ni-Mo-NbC rotor, as in a face seal, was operated for a total of 20 hours up to 1000°C (about 1800°F), 200-fps sliding speed, and 20-psi unit load with only slight surface damage and a total wear of 6.7 mils, most of which occurred on the rotor.

University of Illinois. WADC TR 58-12, Part III.
HIGH-TEMPERATURE ELECTRICAL INSULATING INORGANIC COATINGS ON WIRE. David L. Wilcox, Clifton G. Bergeron, Paul F. Schwarzlose, Arthur L. Friedberg. AF 33(616)-3943. May 1960. PB 161954. Order from OTS $2.00.

The program on high-temperature electrical insulation on wire, in its third and final year, covered the work on flexibility of coated wire, coating trials on clad copper wires, firing procedure for two coats of 58C on copper wire, vitreous silica and boron nitride mill additions to glass coating 58C, and vitreous coatings sheather with organics. Also, as a part of the last year's effort, test data were obtained on high-temperature insulated wire supplied by various manufacturers.

Flexibility studies indicated the ratio of coating thickness to wire diameter and the cooling cycle were important factors affecting flexibility of the coating. It was demonstrated that a heat treatment of the coating may increase coating flexibility. Contrary to what was expected, coatings on inconel-clad copper wire did not show an increase in flexibility over coatings on plain copper wire.

The results of coating trials on inconel-clad, stainless steel-clad (oxalloy), nickel-clad (Kulgrid), silver-clad, and aluminum-clad copper wires are given. A firing study of coating 58C resulted in the optimum firing procedure for two coats of 58C on copper wire.

It was demonstrated that additions of vitreous silica and boron nitride to the mill formula of coatings 58 and 58C enhance the electrical properties of the resulting coating.

Sheathing of vitreous coatings with organics showed promise as a method of retaining the flexed vitreous coating on wire. It was thought that in service the organic coating may burn off at a temperature where the vitreous coating could be capable of sealing itself.

Electrical tests of flexed coated specimens, indicated that room temperature dielectric constant and dissipation factor were not sensitive to the first fine cracks which appeared.

Armour Research Foundation. WADC TR 59-316, Part II.
MECHANISM OF WEAR OF NONMETALLIC MATERIALS. C. H. Riess. AF 33(616)-5962. May 1960. PB 161955. Order from OTS $0.75.

A fundamental study was made of the wear mechanism of nonmetallic materials at high temperatures. Single-crystal sapphire was examined in a friction apparatus made for studies up to 2200°C. A sapphire ball loaded at 22, 50, and 127 g was passed over a sapphire plate at 0.0053 cm/sec at 29-921°C. Both slider and plate were oriented such that the C$_0$-axis was normal to the direction of traverse.

With cleaned, as-received materials coefficient of friction values were scattered. Prior heat treatment at 900-925°C reduced scatter and gave peak values of 0.25-0.30 near 600°C. The results suggest that the surfaces of the sapphire were imperfect. Observations by other investigators on variation of modulus of rupture appear to have a common basis.

The wear areas on both ball and plate were 6 microns wide, and subsurface changes extended to 30 microns. Electron microscopy inspection suggests that extremely high surface temperatures occurred coincidently with the friction process, but direct evidence of an adhesion mechanism is inconclusive.

Atlantic Research Corporation. WADD TR 60-109, Part I.
DEVELOPMENT AND EVALUATION OF SOLID PROPELLANT ROCKET MOTOR CASE INSULATING MATERIALS SYSTEMS. James D. Batchelor, N. Vasileff, Stewart W. McCormick, Eugene L. Olcott. AF 33(616)-6316. June 1960.

This report summarizes the results of the first year of a program designed to improve the capabilities of rocket motors which use end-burning grains through: (1) development of laboratory test procedures to be used as criteria for the evaluation of insulating materials, and (2) development of materials for application as motor case insulation. A literature survey and stress analysis of the motor insulation system were conducted. Procedures have been established to measure the elastic properties of tube specimens and the thermal conductivity of composite materials. The pyrolysis of resins and resin-bonded ceramic materials has been studied using a laboratory image furnace, and the gaseous and liquid decomposition products have been collected and analyzed.

The materials development work has included the comparison of insulating behaviors, erosion resistance, and charring rates of several resins in a torch test. It also includes a study of the carbon yield from resins and composite materials. With respect to insulation ability, resins which yield a high percentage of volatile products usually appear best in the torch test, but no simple correlation is evident. The yield of carbon necessary to produce coherent char structure can be investigated by measuring the physical and thermal properties of typical charred residues.

An extension of this program during the coming year, under Contract AF 33(616)-6831, will include specialized motor firings to test materials under service conditions; the laboratory work will be continued to correlate laboratory and motor data.

Bell Aircraft Corporation. WADC TR 59-744 Volume III.
INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOLUME III. Screening Test Results and Selection of Materials. July 1960. ASTIA Document No. AD 247110.

The purpose of this contract was to investigate the feasibility of utilizing available heat resistant materials in the fabrication of leading edges for hypersonic boost-glide vehicles. This particular volume presents the results of

the preliminary portion of the material evaluation portion of the program. Material requirements were established for this specific application. In order to assess the suitability of a given material, knowledge of many material characteristics is required. Of primary importance are oxidation resistance, high thermal emissivity, high thermal conductivity, low thermal expansion, reproducible strength characteristics, and fabricability. Other characteristics which must be considered are specific heat, modulus of elasticity, erosion characteristics and density. Over 100 technical reports were reviewed, and approximately 50 suppliers and fabricators were contacted to obtain data on materials which might be suitable for leading edge applications at 2500°F to 3000°F. A total of 106 materials were considered including 22 refractory metals, 30 protective coatings and 54 refractory non-metals.

From the review of available materials it was apparent that much of the information required for assessing suitability was lacking. A screening test program was initiated, therefore, to fill the gaps in available data and to provide consistent sets of data upon which to base intelligent selections. The methods used in choosing the materials to be screened considered availability and the complex interrelation among design and material parameters. From the 106 candidate materials three metals, five coating systems and seven non-metals were subjected to screening tests. Determination of oxidation resistance, thermal emissivity, thermal conductivity, thermal expansion, mechanical strength and modulus of elasticity, were made from room temperature to 2700°F.

Using the data obtained experimentally and the methods developed for assessing suitability, the .5% titanium alloy of molybdenum, Chromalloy W-2 coating, Durak MG coating and siliconized ATJ graphite were found to be the most promising materials for the specific application. Consequently these materials were selected for further evaluation.

Bell Aircraft Corporation. WADC TR 59-432, Part II.
REFRACTORY INORGANIC MATERIALS FOR STRUCTURAL APPLICATIONS. Harry A. Pearl, John M. Nowak, Joseph C. Conti. AF 33(616)-5930. July 1960. PB 171148. Order from OTS $2.25.

A process was developed for making both small and large, simple and complex curvature geometric shapes from honeycomb ceramic sandwich constructions. Final fired sections had apparent densities as low as 26 lb/ft^3.

A simple mechanical method was developed for continuous fabrication of ceramic corrugated ribbon or sheet for making honeycomb cores. The core is flexible and easily machinable in its prefired condition. The skins are as easy to handle as paper.

Alumina-silica paper impregnated with an alumina-silica slurry was used as a refractory model system for developing the fabrication techniques. The paper originally acts as a carrier but finally becomes an integral part of the fired refractory. The fabrication and processing methods are adaptable to other refractory materials possessing higher strength and temperature stability.

Other types of ceramic and metal-ceramic composites were investigated and evaluated.

WADD TR 60-143.
RESEARCH STUDY TO DETERMINE THE PHASE EQUILIBRIUM RELATIONS OF SELECTED METAL CARBIDES AT HIGH TEMPERATURES. R. T. Dolloff. July 1960. ASTIa Document No. AD 244270. PB 171365. Order from OTS $0.75.

The work here reported is the result of an investigation of phase equilibria in the ternary system, silicon-boron-carbon. Techniques for extending the high temperature range of measurements have been developed. Accurate phase diagrams for the silicon-carbon and the boron-carbon binaries have been obtained and details of the ternary system have been determined in the high carbon content region. The data were obtained by high temperature differential thermal analysis and by photoelectric thermal analysis, both supplemented by metallographic, X-ray and chemical techniques.

Results for the boron-carbon binary system indicate that boron melts at 2130 ± 10°C and that B_4C has a congruent melting point of 2470 ± 20°C. Boron and B_4C form a eutectic at 2080 ± 20°C, whereas carbon and B_4C form a eutectic at 2390 ± 20°C.

Results for the silicon-carbon binary indicate a eutectic between silicon and SiC at 1402 ± 5°C. SiC melts incongruently at 2540 ± 40°C. The peritectic point is located at 27 percent carbon.

Results for the ternary system indicate a eutectic between B_4C, SiC and carbon at 2250 ± 20°C. No compound formation has been observed in the high carbon region of the ternary system.

General Electric Research Laboratory.
WADD TR 60-184.
EFFECT OF BASIC PHYSICAL PARAMETERS ON ENGINEERING PROPERTIES OF INTERMETALLIC COMPOUNDS. D. L. Wood, J. H. Westbrook. AF 33(616)-6144. August 1960. ASTIA Document No. AD 246540. PB 171393. Order from OTS $1.00.

To facilitate subsequent studies of the nature of the brittleness in intermetallic compounds, a method for producing sound, uniform, and reproducible test specimens has been devised and an investigation made of the effects of basic physical parameters on the mechanical properties.

Tensile test specimens of Bi_2Tl and AgMg, produced directly by extrusion, have provided information applicable to future studies of the properties of NiAl, a material whose melting point and oxidation resistance render it not impractical as an alloy base should a solution to the ductility problem be found.

A pronounced yield point is found in AgMg; high strain rate sensitivity of the yield stress has been observed and the effects of grain size, composition, and test temperature have been documented. Specimens will withstand loading only at very slow strain rates; after yielding occurs, however, rapid strain rates may be employed. With total elongations of more than 50 percent, the material is ductile under previously brittle conditions. Measurement of the yield stress as a function of both strain rate and temperature enables a calculation of the activation energy for the yielding process.

University of Illinois. WADC TR 59-526, Part II.
PROTECTIVE COATINGS FOR REFRACTORY METALS. Clifton G. Bergeron, Victor J. Tennery, Arthur L. Friedberg, Doris M. Maraney, Robert D. Shannon. AF 33(616)-5734. August 1960. PB 171193. Order from OTS $1.50.

Various ceramic coating systems for tungsten metal were developed and evaluated. These included silicide coatings, ceramic crystals in a glass matrix, zirconia-clad coatings, vapor-deposited SiO, and recrystallizing glasses. The system considered protective for tungsten at temperatures of 3000°F or higher, from a practical viewpoint, was a coating consisting of 35% glass and 65% zircon.

Tungsten wire specimes coated with this glass-zircon coating were protected for ten hours at 3000°F. A study of the nature of the degradation of this coating tested at 3000°F as a function of time was carried out based on microstructural observations and X-ray diffraction analysis of the reaction products. The protective lifetime of the coating was related to the degree of attack of the coated tungsten metal as indicated by interfacial roughness and tungsten oxide formation, and to the progressive decrease in the thickness of the coating under test.

University of Utah. WADD TR 60-473.
SURFACE AND ENVIROMENTAL EFFECTS ON CERAMIC MATERIALS. P. Gibbs, G. S. Baker, B. Gale Dick, Jr., E. K. Beauchamp, M. H. Miles, W. G. Rogers. AF 33(616)-5876. August 1960. PB 171074. Order from OTS $0.75.

Creep and recovery phenomena in sintered polycrystalline alumina have been studied. Effects of temperature and impurity content on creep behavior and the results of observation of polished and etched surfaces are discussed. The results are analyzed in terms of a tentative conventional mechanical analog.

An electrical study of the behavior of silver ions on a corundum surface is described. Current flow between evaporated silver films on Al_2O_3 crystals is investigated as a function of geometry, temperature, atmosphere, history, and applied voltage. Migration of silver in some cases leaves visible deposits on the uncoated crystal. In general, the phenomena are complex and atmosphere-dependent.

The initial stages of creep in single crystals of αAl_2O_3 (synthetic sapphire) form of 0.1 inch diameter cylinders three inches long were loaded in three point loading to give slip on the (0001) $[11\bar{2}0]$ slip system. Creep rates were measured in the temperature range 1000°C to 1200°C and in the stress interval of 500 to 1000 Kg per cm.^{2} resolved shear stress on the outermost fiber of the specimen. The stresses covered the region from too little stress to initiate creep up to the fracture stress. An approximate sixth power dependence of creep rate on stress was observed. The dependence of creep rate on temperature was measured for total creep less than 1% strain. A plot of log creep rate versus reciprocal temperature gives an activation energy of 85 Kcal/mole. Specimens oriented so that (1120) $[1\bar{1}00]$ slip system was operative but the (001) $[11\bar{2}0]$ slip system had zero resolved shear stress gave no significant creep at these temperatures.

Arthur D. Little, Inc. WADD TR 60-377.
STABILITY OF CERAMIC MATERIALS AT TEMPERATURES TO 2000°C. Joan Berkowits. AF 33(616)-6154. September 1960. PB 171405. Order from OTS $1.75.

A study was made of the oxidation resistance of a number of commercial samples of $MoSi_2$ and WSi_2 in the temperature range 1600-1700°C. A thermal conductivity technique was developed to measure oxygen pick-up by the samples continuously. From the thermal conductivity measurement and the net weight change of the samples, an estimate was made of the rates of formation of volatile and non-volatile oxides. On the basis of X-ray analysis, oxidation products are believed to be volatile XO_3 (vapor) and non-volatile glass of the form $SiO_2 \cdot nXO_m$ where X = Mo or W; n and m are not known. Tests were conducted at a total pressure of one atmosphere in a helium stream containing about 10 mm of oxygen and flowing at 119 cc/min. The oxidation of WSi_2 was also studied in a helium stream containing 5 mm of water vapor. The principal solid product in this case was metallic tungsten.

The Marquardt Corporation. WADC TR 59-102, Part II.
DEVELOPMENT AND EVALUATION OF INSULATING TYPE CERAMIC COATINGS. H. Leggett, R. L. Johnson, E. W. Blocker, E. D. Weisert. AF 33(616)-5441. October 1960.

A metal reinforced, insulating ceramic coating system has been developed which will successfully withstand temperatures greater than 4000°F. The composite is of the gross type and it consists of a chemically bonded zirconia or zirconia chromia phase bonded at low temperatures, reinforced with and anchored to a coated refractory metal. The composite system exhibits low thermal conductivity and excellent thermal shock resistance.

The developed macro-composite utilizes the desirable properties of refractory metallic structures by providing both insulation and oxidation protection. The metallic reinforcement, protected in the same manner, strengthens the ceramic layer and serves to anchor it to the basic metal structure.

Douglas Aircraft Company, Inc. WADD TR 60-338.
INFLUENCE OF ENVIRONMENT ON CERAMIC PROPERTIES. George R. Pulliam, Bruce G. Leonard. AF 33(616)-5875. October 1960. PB 171398. Order from OTS $0.75.

This study was concerned with environmental and non-stoichio-metric effects on three different materials. The materials used were ZrO_2, AgCl, and MgO single crystals. Definite evidences of low temperature plastic strain were found for O-deficient ZrO_2. These evidences were non-linear stress-deflection curves at room temperature, appearance of slip lines on large grains after stress, and creep of ZrO_2 at 350°C. The creep of polycrystalline AgCl was found to be highly sensitive to electromagnetic radiation. When an AgCl specimen in creep was illuminated with 440mu light the creep rate was reduced. Light of 700mu caused the previously reduced creep rate to be increased. It is felt that precipitates of photolytic silver hardened the AgCl in much the same manner as the precipitates in the precipitation hardenable stainless steels. The 700mu light had the effect of bleaching the precipitates out, and thus, permit the creep to continue. Creep specimens illuminated with 700mu light with no previous 440mu treatment were unaffected by the irradiation. MgO single crystals colored brown by u.v. irradiation were found to have an absorption peak at 4.3ev associated with oxygen. Ultra-violet irradiation increased the height of this peak so that it tailed off in the visible region. It is this tailing off that imparted the brown color to the crystals. Vacuum treatment of such specimens to 1300°C removed the peak and prevented further coloration by ultra-violet illumination.

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The vapors issuing from mullite and Graphite Knudsen cells containing pure metals and alloys of the triad Cu-Ag-Au have been analysed mass spectrometrically. From the experimental ratios of diatomic to monoatomic species and the vapor pressures of the elements, the following dissociation energies are obtained:

$D_0^0(Cu_2) = 43.2 \pm 2.2$ kcal $D_0^0(AgAu) = 47.6 \pm 2.2$

$D_0^0(Ag_2) = 35.7 \pm 2.2$ kcal $D_0^0(AgCu) = 40.7 \pm 2.2$

$D_0^0(Au_2) = 51.9 \pm 2.2$ kcal $D_0^0(AuCu) = 54.3 \pm 2.2$

These are based on H_{298}^{vap} 81.1, 68.4 and 87.5 cal/mole for Cu, Ag and Au where D_0^0 of AgAu depends on the value for Cu, and D_0^0 of AgAu and AuCu on Au. The uncertainties quoted do not include the uncertainty in ΔH^{vap}

These results are interpreted in terms of chemical bonding theories. The relation between bonding in the gas and in the pure condensed phases is again observed. Furthermore, a previously unobserved qualitative relation between the dissociation energy of the asymmetrical molecule and the heat of formation of the corresponding alloys is indicated.

University of Brussels. WADD TR 60-782, Part II.
VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURE PART II. Mass Spectrometric Studies of the Vaporization of Sulphides and the Dissociation Energy of S_2. R. Colin, P. Goldfinger, M. Jeunehomme. AF 61(052)-225. November 1960. ASTIA Document No. AD 258381. Order from OTS $1.60.

The equilibria $S_2 \rightleftharpoons 2S$ and $CaS \rightleftharpoons Ca + S$ have been observed in the vapor phase above CaS. $D_0^0(S_2) = 4.4 \pm 0.2$ e.v., $D_0^0(SO) = 5.4 \pm 0.2$ e.v., $D_0^0(CaS) = 3.7 \pm 0.2$ e.v., and $H_{298}^{vap}(CaS) = 5.9 \pm 0.2$ e.v. have been determined mass spectrometrically.

The Carborundum Company. WADD TR 60-244, Part II.
CONTINUOUS FILAMENT CERAMIC FIBERS. Edward H. Girard. AF 33(616)-6246. February 1961. PB 171061. Order from OTS $2.25.

A number of refractory materials were examined to determine their possible usefulness as materials for crucibles for melting, fining, and drawing high-silica glass compositions. Niobium di-boride gave the most promising results of the materials examined.

Time and temperature relationships as well as variations in batch composition were investigated in an effort to improve premelt characteristics. The addition of vanadium pentoxide was particularly effective. It had been conceived that a well-fined premelt should be a better starting material for fiber drawing than an unreacted batch composition. Fiber drawing studies, however, showed no improvement in either the drawing characteristics or in the physical characteristics of fiber when well-fined premelt was used.

The Carborundum Company. WADD TR 60-749, Part I.
FACTORS AFFECTING THERMAL SHOCK RESISTANCE OF POLYPHASE CERAMIC BODIES. P. T. B. Shaffer, D. P. H. Hasselman, A. Z. Chaberski. AF 33(616)-6806. February 1961. ASTIA Document No. AD 258537. Order from OTS $1.75.

An investigation of the factors which affect the thermal shock resistance of polyphase ceramic systems has been conducted using the model system zirconium carbide-graphite. The research has been divided into two areas:

1. Theoretical calculations of the individual material properties, which show that the included graphite particles may be regarded as spherical pores.

2. Experimental data, which substantiate the postulate that increased thermal shock resistance is a result of an increase in the ratio of strength over Young's modulus, brought about by the addition of the low Young's modulus phase.

New York University. WADD TR 60-825.
REFRACTORY COATINGS FOR TUNGSTEN. C. G. Goetzel, P. Landler. AF 33(616)-6868. March 1961. ASTIA Document No. AD 258574. Order from OTS $1.50.

A preliminary study of the feasibility of protecting tungsten wires against oxidation at 3300°F (1815°C) was undertaken. A tungsten disilicide coat was found to be the most promising coating for this operating temperature.

Several siliconizing methods were given a cursory treatment, including vapor plating and dipping into molten copper-silicon bath, and the simplest, cementation, selected for closer investigation. This operation was performed under flowing hydrogen atmosphere and such variables as temperature, time, and composition of the cementation pack were studied as a main part of our exploratory work. A single brittle tungsten-disilicide (WSi_2) layer is formed by this method. The inherent brittleness of tungsten appears further aggravated by the development of micro-cracks in the layer as well as in the substrate during siliconizing.

Oxidation tests using direct electrical resistance heating were performed. It was found that pack-siliconized wires can withstand oxidation at 3300°F in still air for prolonged periods up to and exceeding 10 hours. This is accomplished without any destruction of the coating in the high temperature zone owing to the formation of a viscous glassfilm on the surface. A pronounced tendency toward self-healing is a useful property of this type of coat.

A serious drawback of the disilicide coat, however, is its low temperature oxidation ("Disilicide Pest") which occurs in parts exposed to temperatures between 1200° and 2280°F (650°-1250°C) for prolonged periods. It causes a destruction of the disilicide layer over a period of 5 to 15 hours, depending on coating thickness. It was the actual life determining factor in those tests where other failures caused by improper technique had been eliminated.

Preliminary attempts to deposit ZrO_2 on top of siliconized wires by means of poor adhesion. Where bonding was obtained, no improvement in life under high temperature oxidising conditions could be ascertained.

Elastomers

Connecticut Hard Rubber Company. WADC TR 57-651, Part II.
DEVELOPMENT OF RUBBERLIKE MATERIALS FOR APPLICATIONS INVOLVING CONTACT WITH LIQUID ROCKET PROPELLANTS. John H. Baldrige. AF 33(616)-5572. June 1959.

Elastomeric materials with satisfactory resistance during an immersion period of seven days at 160°F have been developed for the following propellants: JP-X fuel mixture, diisopropenyl acetylene and hydrazine. Compounds based on an experimental polymer have proved to be very compatible with n-propyl nitrate and unsymmetrical dimethyl hydrazine, but additional work has been directed toward development of other materials, based on commercial polymers, which will be resistant to these liquids. Compounds have been tested which were resistant to inhibited red fuming nitric acid at room temperature.

No elastomers tested have been found resistant at room temperature to fluorine gas or nitrogen tetroxide.

Results are presented for screening tests of plastics and fabrics in several propellants and for tests of elastomers in propellants at 350°F and 400°F.

Products Research Company. WADC TR 58-89, Part II.
DEVELOPMENT OF HIGH TEMPERATURE SEALANTS. Irvin P. Seegman, Paul A. Mallard, William Cheorvas, Ron E. Thompson, Francis H. Ingham, R. Loren Varner, Sarkis H. Kalfayan. AF 33(616)-3976. September 1959. ASTIA Document No. AD 229864.

This report describes work accomplished during the second year of research on the development of fuel and pressurization sealants resistant to temperatures up to 700°F.

Service temperature limits were established for sealants developed during the first year, as well as for new sealants. These materials were investigated for resistance to HEF-3 and to gamma radiation. Studies were made on the use of anti-rads to improve radiation resistance.

Viton A based fill and drain, faying surface, and filleting sealants were developed which had satisfactory resistance to JP-5 fuel either at 450°F for 168 hours or at 600°F for 10 hours, and to oven aging either at 450°F for 168 hours, or at 650°F for one hour.

A Viton A based sealant system was developed which has satisfactorily withstood thirty-three HEF-3 Vapor cycles.

Sealants based on Viton A and RTV silicones appeared to be serviceable after gamma irradiation up to 8.71×10^8 ergs/g.DC. A groove sealant based on a combination of Silastic LS-53U and Fluororubber 1F4 was crosslinked to a rubbery material at 8.71×10^7 ergs/g.DC. None of the materials which were studied as anti-rads were of any value in improving the radiation resistance of any of the sealant systems which were investigated.

B. F. Goodrich Co. Research Center. WADC TR 55-58, Part V.
A STUDY OF THE EFFECTS OF NUCLEAR RADIATIONS ON ELASTOMERIC COMPOUNDS AND COMPOUNDING MATERIALS. Dale J. Harmon. AF 33(616)-5646. September 1959. ASTIA Document No. AD 229865.

The research for this fifth year under the contract included the following: fundamental studies of the mechanism of radiation damage to high polymers, of the influence of molecular weight and copolymer ratios on the relative degree of radiation effects, and of the effect of anti-rads on chain scission and crosslinking rates; basic studies of the effect of state of cure on the radiation resistance of elastomers, and the role of anti-rads in protecting textile materials from radiation damage; further screening of anti-rads for other conventional and newer elastomers; a study of the radiation stability of some of the newer thermally stable elastomers; applied studies of the separate and combined effects of heat and radiation on the stress-strain properties of aircraft rubber compounds and the effect of irradiation in air and in alkyl diphenyl ether (C_{14}-C_{16}) on the compression set properties of aircraft compounds; irradiation and full-scale indoor testing of aircraft tires, with and without potential anti-rad protection.

This work which is reported thus included fundamental, basic, and applied research.

Armour Research Foundation. WADC TR 59-338. COMPOSITE INORGANIC RESILIENT SEAL MATERIALS. M. Sabanas. AF 33(616)-5793. October 1959.

Research was conducted on essentially inorganic seal materials exhibiting the desirable characteristics common to elastomers, combined with temperature and fluid resistance to at least 1000° F. Materials exhibiting this behavior are a prerequisite to the design concept of power transmission systems for future flight vehicles.

Experimental screening tests were performed on composite refractory materials to provide leads for new materials for ultimate static and dynamic seal applications. Of the numerous refractory material composites tested, the graphite and molybdenum disulfide combination approached the target objectives, but were rapidly oxidized in an air atmosphere at 1000°F.

Materials consisting of stainless steel and molybdenum fibers made into a bonded skeleton of the desired shape and impregnated with various soft ductile phases, were fabricated and their properties studied. The properties of these composites differ from the base metals from which they are made, since the geometrical configuration imparts an elastomeric like behavior to the composite. A longer period of research would be required for further experimental study to explore all the possibilities that these new composite materials offer.

Boeing Airplane Co. WADC TR 56-272, Part IV. DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS. George E. Trepus, Robert S. Roper, William R. Hickman. AF 33(616)-5722. October 1959. ASTIA Document No. AD 230658.

This is a continuation of a study initiated under Air Force Contract AF 33(616)-2867. The purpose of this study was to determine design criteria for O-Rings, back-up rings and other elastomeric seals. A survey of current literature concerning seals and seal materials was conducted with an emphasis placed on seals and seal materials for use in environmental extremes. Tensile properties of a number of silicone and Viton compounds are given. The effects of environment and groove configurations on the sealing force of an O-Ring were determined. Thermal expansion and compression modulus tests were performed on elastomeric materials at cryogenic temperatures. The functional tests included pneumatic seal tests at 400°F, hydraulic rod seal tests at 400°F using O-rings and back-up rings made from a variety of materials and in several configurations, static annulus tests at elevated temperatures using various groove configurations, and static seal tests using helium at cryogenic temperatures.

Certain physical properties were found to be of value to the life of a seal under specific environmental and/or mechanical conditions.

Boeing Airplane Company. WADC TR 59-428. DESIGN HANDBOOK FOR O-RINGS AND SIMILAR ELASTIC SEALS. Frank W. Tipton. AF 33(616)-5722. October 1959. ASTIA Document No. AD 230639.

This is a summation of the work done under Air Force Contracts AF 33(616)-2867 and AF 33(616)-5722. It is presented in the form of a handbook covering the mechanism of O-ring sealing, the relation of physical properties to sealing, the effect of cavity configuration back-up rings and adverse mechanical conditions on seal life, and the design of seals for specific systems. It also includes the test procedures and a bibliography of published articles pertinent to O-ring seals.

Southern Research Institute. WADC TR 59-301. DEVELOPMENT OF MATERIALS FOR IMPROVED EAR PLUGS AND FOR LIQUID-FILLED EAR PROTECTOR CUSHIONS. Edgar A. Verchot, Arthur E. Raeuber. AF 33(616)-5673. December 1959. ASTIA Document No. AD 233447.

This report describes research and development work directed toward finding materials suitable for fabrication and toward setting up specifications for V-51R design ear plugs and for liquid-filled ear-protector cushions. Section I of the report covers the work on ear plugs; Section II covers the work on ear-protector cushions.

Materials that met the initial specified requirements for ear plugs were found. Molds to make the extra-small size and the extra-large size V-51R design ear plugs were fabricated. Ear plugs with a range of collapsibilities were made from these molds and from the medium-size-ear-plug mold, made on a previous project (AF 33(616)-3488). The most desirable collapsibility measurement for ear plugs was then selected by an "in-service" evaluation program monitored by the project engineer. The changes needed to assure continued procurement of ear plugs with the most desirable properties have been suggested for the Military Medical Purchase Description Number 1, dated 9 January 1958.

Materials to meet the initial specified requirements for liquid-filled ear-protector cushions were studied. Although it was not found possible to get experimental quantities of the plastic film material judged to be best for liquid-filled ear-proctector cushions, it has been established that commercial quantities of such a plastic film would become available if a demand existed. On this basis, a number of liquid-filled ear-protector cushions were submitted for an "in-service" evaluation program monitored by the project engineer.

Boeing Airplane Co. WADC TR 56-272, Part V. DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS. George E. Trepus, Robert S. Roper, William R. Hickman. AF 33(616)-5722. March 1960.

This is a continuation of a study initiated under Air Force contract AF 33(616)-5722. The purpose of this study was to determine design criteria for O-rings, back-up rings and other elastomeric seals. A survey of current literature concerning seals and seal materials was conducted with an emphasis placed on seals and seal materials for use in environmental extremes. Physical properties of a number of silicone and Viton compounds are given. These properties, including thermal expansion, compression modulus, tensile strength and elongation, tensile and compression stress relaxation and compression set, were determined at temperature extremes in the range of from -425° to 600°F. Functional tests included pneumatic seal tests at 400°F and 500°F at 2000 psig, hydraulic rod seal tests up to 600°F at pressures from 1000 to 5000 psig, static annulus tests at 600°F using various groove and seal configurations, and static seal tests using helium at cryogenic temperatures.

Certain physical properties were found to be of value to the life of a seal under specific environmental and/or mechanical conditions. Hydraulic seal test results were more dependent on the effectiveness of the back-up ring than on any measured property of the O-ring material.

Chance Vought Aircraft, Inc. WADC TR 59-749. COMPOSITE ELASTOMER-METAL O-RING SEALS. Jack R. Bos, Eugene B. Weber. AF 33(616)-6182. April 1960. ASTIA Document No. AD 240165.

A new approach to O-ring sealing has been attempted in order to improve the physical and mechanical properties of conventional elastomeric O-rings and eliminate the use of anti-extrusion devices. Elastomer-metal composite O-ring seals have been fabricated with a metallic spring concentrically embedded in the elastomer. Special molds and molding processes were required.

Performance evaluation of the prototype composite seals in hydraulic fluids at elevated temperature revealed that rolling or spiralling, a failure frequently experienced with conventional O-rings, was virtually eliminated. The spring cored O-ring performed much better in static impulse tests than in dynamic reciprocating tests. Seal failure in cycling tests resulted from excess nibbling and friction wear which were attributed to the greater force exerted on the sealing surface by the elastomer-metal O-ring. A limited investigation of decreased squeese on the spring-cored O-ring seal showed improved cycling life.

Armour Research Foundation. WADC TR 59-338, Part II. COMPOSITE INORGANIC RESILIENT SEAL MATERIALS. M. Sabanas. AF 33(616)-5793. April 1960.

The principal objective of this research program is to investigate and develop new concepts for novel and unconventional material combinations which would have resilience, recovery, strength, and chemical resistance at temperatures up to 1000°F. Major emphasis was given to configurations that would enable these materials to be used as static and dynamic reciprocating shaft seals.

Composite material combinations consisting of stainless steel and molybdenum fibers impregnated with tin, indium, magnesium, silver, and polymeric materials were produced.

Composites made of molybdenum fibers impregnated with silver were evaluated as static seals and showed good ability to seal air heated to 1000°F and retained pulsating pressures from zero to 5000 psi.

The relationship between the fiber structure, impregnant, and final composite was studied.

Impregnation techniques and secondary work processes like machining are outline in this report.

Wyandotte Chemicals Corporation. WADC TR 55-492, Part V. RESEARCH AND DEVELOPMENT ON HIGH TEMPERATURE FUEL RESISTANT RUBBER COMPOUNDS. Raymond G. Spain, Wayne G. Lajiness, Eva Deck. AF 33(616)-5544. April 1960.

This report describes research directed toward the preparation of elastomeric vulcanizates resistant to high energy fuels (HEF) and hydrocarbon fuels.

Tests with HEF were made at temperatures up to 325°F, and tests involving the cycling of elastomeric vulcanizates in HEF and JP-6 utilized temperatures up to 450°F.

Tests of vulcanizates in JP-6 were made at temperatures up to 600°F using conventional testing procedures. Static and dynamic tests of experimental vulcanizates fabricated as O-rings were made at 400°F under simulated service conditions.

A variety of vulcanizing systems for fluoroelastomers was explored, and swelling studies of the vulcanizates indicated this technique to be applicable in the determination of the comparative degree of vulcanization of various specimens.

Firestone Tire & Rubber Company. WADC TR 56-331, Part IV.

RESEARCH ON HIGH TEMPERATURE RESISTANT RUBBER COMPOUNDS. Robert A. Hayes, Floyd M. Smith, Glenn A. Kidder, James C. Henning, Jack D. Rigby, George L. Hall. AF 33(616)-3953. April 1960.

Compounding of promising heat-stable elastomers to improve their physical properties has been continued. A representative series of 22 vulcanizates has been submitted to extensive high-temperature exposure conditions and tests which included: short term exposures, 3 or 4 minutes, in air at 600° and 800°F, in a nitrogen atmosphere with the sample in contact with a metal surface at 800°F and in nitrogen with the sample exposed to radiant heat from a metal surface 1/4" away at 1000°F; thermal conductivity measurements in the range of 100°F to 500°F; long-term aging, 300-500 hours at 300°F; and compression set determinations up to 500 F. Silicone, Viton, Kel-F 3700 and poly(ethyl acrylate) rubbers show outstanding thermal stability in many of these tests.

A method of constructing wirecord aircraft tires has been demonstrated and prototype tires of methacrylate and silicone rubber having excellent appearance were built. (Six additional methacrylate rubber tires were built under a separate WADC contract.)

The search for possible new high temperature elastomers has continued. A fairly heat-stable elastomeric polyester has been obtained from 5-pentoxyisophthalic acid and hexamethylene glycol and progress has been made in modifying this polymer to permit cures.

A new wholly aromatic polyamide fiber with outstanding high-temperature properties has been examined and methods for adhering it to various high-temperature vulcanizates were sought.

A bright aluminum coating applied by vacuum metallizing gave considerable surface protection to a resin-cured butyl vulcanizate exposed to 1000°F radiant heat in nitrogen.

Connecticut Hard Rubber Company. WADC TR 57-651, Part III.

RESEARCH ON RUBBERLIKE MATERIALS FOR APPLICATIONS INVOLVING CONTACT WITH LIQUID ROCKET PROPELLANTS. John H. Baldrige, Mark D. Inskeep. AF 33(616)-5572. May 1960.

Elastomeric materials resistant to hydrazine, unsymmetrical dimethyl hydrazine and JP-X fuel mixture have been found for service at room temperature and 160°F for immersion periods ranging from 21 days to 6 months. Among these is a new commercial polybutadiene elastomer. A semi-commercial elastomer resistant to n-propyl nitrate up to 3 months at 160°F has been found. Compounds which resist inhibited red fuming nitric acid up to 3 weeks at room temperature have been evaluated.

Several flexible plastics have been found resistant to nitrogen tetroxide (3 months), inhibiting red fuming nitric acid (3 weeks) at room temperature and liquid chlorine trifluoride at its boiling point (1 hour). No elastomers tested resisted nitrogen tetroxide or chlorine trifluoride.

Results are presented of fabrication studies, permeability tests and static immersion tests at 350°F and 400°F for resistant elastomers and plastics. A dynamic immersion test procedure has been developed, equipment assembled and a series of 24-hour evaluations run with several propellants.

WADD TN 60-154.

HIGH TEMPERATURE FUEL TANK SEALANTS. Phillip A. House. September 1960.

The Air Force has investigated the area of integral fuel tank sealants for advance design air weapons and has obtained materials which significantly advance the state-of-the-art. Fuel tank sealants are now available that will withstand considerable periods of aging in fuel and air at temperatures up to 600°F. A room temperature cure for fluoroelastomers has been discovered and efforts are now being directed toward suitable sealants using this curing system.

Electrical and Electronic Materials

Massachusetts Institute of Technology, Research Laboratory of Electronics, Cambridge. TR 122. U 18523; ATI-112708.

AN ELECTRONIC CORRELATOR. T. P. Cheatman, Jr. DA 36-039-sc-100 and W 36-039-sc-32037. March 28, 1951.

The role and application of correlation functions in the statistical theory of communication is described in terms of the important basic properties of autocorrelation and crosscorrelation functions. An electronic correlator constructed on the basis of (a) utilizing pulse-sampling techniques and (b) multiplication in time-amplitude coordinates is described in detail. General criticisms and suggestions for improvements, based on experience gained from the application of the correlator to several problems, are given. Some additional methods of computing correlation functions are outlined and discussed. The experimental application of the electronic correlator is illustrated by several studies that are concerned with the detection, analysis, or filtering of a desired signal in noise.

Chicago University. Chicago Midway Labs, Ill. Report No. CML-TN-55-F. 2-6.

A SURVEY OF FILTER THEORY AND ITS APPLICABILITY TO INSTRUMENTATION. R. N. Lewis. AF 33 (038)-25913. July 1955. ASTIA Document No. AD 69815.

This report gives an elementary review of the factors and the philosophy upon which fire control filter theory is based. A close analogy between fire control system sensors and classes of measuring instruments is maintained so that, while the theory of random noise filtering is developed, the difficulties of the measurement and evaluation of such filtering are indicated. The first section indicates the necessity for filtering of noisy signals, the second section develops the required theory, and the third section applies the theory to fire control system sensors and computers and to measuring instruments. The final section contains preliminary conclusions and recommendations for applying filter theory concepts in the selection of instrumentation for evaluating airborne fire control systems.

WADC TN 57-241.
ELECTRICAL AND ELECTRONIC MATERIALS FOR HIGH SPEED MISSILES AND AIRCRAFT. Dr. R. N. Evans. September 1957. ASTIA Document No. AD 131074.

The materials which go into the construction of electrical and electronic component parts for aircraft and missiles are discussed from a chemical point of view. Materials are divided arbitrarily into Organic and Inorganic types. Environmental hazards under which the materials of high speed aircraft and missiles must perform are listed. A blueprint of heteroatom polymer types and inorganic synthetic approaches is indicated representing the state-of-the-art effort to meet the severe thermal, nuclear, sonic requirements. The role of the materials engineer in the development of new electronic materials is discussed.

Westinghouse Electric Corp. WADC TR 59-337, Part I.
SYNTHESIS AND PURIFICATION OF DIELECTRIC MATERIALS. T. W. Dakin, R. N. Wenzel, W. C. Divens, D. H. Hogle, D. W. Lewis, P. A. Tierney. AF 33(616)-5979. October 1959. PB 161366. Order from OTS $2.75.

This report describes the progress during the first year on a research program undertaken to prepare pure dielectrics with improved properties for use as electrical insulation at 500°C. Effort has been concentrated on the materials: boron nitride, alumina and silica, and reconstituted mica.

Boron nitride, alumina and silica have been synthesized from purified chemicals. An improvement phase of the program has been the development of techniques for high temperature pressing and firing of these materials to preserve good high temperature dielectric properties. This work has yielded better dielectric properties at 500°C, with boron nitride, than have heretofore been reported for any material. Sintered pure alumina bodies have been prepared with high temperature properties which are approximately equal to those of the best single crystal sapphire available.

The dielectric properties of these materials prepared in many different ways have been measured over the temperature range 25°C - 500°C. Measurements are reported of d-c resistivity, a-c dissipation factor and dielectric constant at 60, 10^3 and 10^5 cycles per second.

It has been shown that little improvement in high temperature dissipation factor and resistivity of reconstituted mica can be achieved by purification of the mica particle suspensions through dialysis and electrodialysis. The high temperature dielectric properties are reported for a variety of preparations of three types of reconstituted mica: muscovite, phlogopite and synthetic fluorophlogopite. Work to improve the physical and electric strength characteristics of the paper and electrophoretic mica coatings is continuing.

Armour Research Foundation. WADC TR 59-303.
METHODS OF PURIFICATION OF METALS AND INTERMETALLIC COMPOUNDS. Sherman Susman. AF 33(616)-5895. November 1959. PB 161415. Order from OTS $2.25.

The mechanisms of impurity transport through a solid in a temperature gradient are listed and discussed. The heat of transport, electric field effects, distillation phenomena, and grain boundary effects must be considered. Usually, the drive toward uniform fugacity or chemical potential is the dominant force acting on impurities.

The physical and chemical properties of the silicon carbide, thorium oxide and zirconium oxide systems are considered in the context of diffusion phenomena and thermoelectric behavior at elevated temperatures. Resistivity values of both pure and ceri-doped thoria are measured over a range of temperatures from 700 to 1000°C. Values of the activation energy are computed and compared with the literature. Doping with ceria appears to yield minimal changes in the electrical properties of a thoria matrix. A calcia stabilized zirconia matrix gives a simple band structure; ceria stabilized material is more complex. Zirconia samples evidence considerably lower resistances than comparable thoria specimens.

Apparatus is described for the measurement of thermal conductivity by a relatively simple technique. The sources of error associated with the high temperature measurement of thermal conductivity by more common methods are eliminated.

Experimentally, inductive coupling and spectrometric diffusibility are used to investigate transport phenomena. Impurity distribution in polycrystalline silicon carbide is followed by spectrographic analysis. The data are analyzed in terms of an empirical working parameter, p, which describes the sum total of all purification effects in the thermal gradient for selected impurities. Purification trends appear for calium and aluminum. For polycrystalline silicon carbide $P_{Ca} = 32.8$ and $P_{Al} = 8.4$.

The spectrometric diffusibility technique is used to investigate single crystal silicon carbide. This method affords the possibility of establishing in a rapid fashion an index of the ease with which a given impurity can be transported through a solid matrix. The results obtained are discussed in terms of the time dependence of the concentration gradients and temperature gradients generated by the anode spot. The $J_t S_t / C_{oi}$ ratios for copper, calcium and aluminum are found to be 113, 194, and 58 respectively.

Nuclear Corporation of America. WADD TR 60-17.
PELTIER EFFECTS FOR CRYSTAL GROWING. Louis L. Thomas. AF 33(616)-6150. May 1960. PB 161985. Order from OTS $1.50.

Available literature on crystal growing and refining processes has been reviewed. In accordance with the results of the literature survey and analysis of the problem, the zone melting technique has been chosen for modification to study the potential of the Peltier effect for improving crystal properties. Zone melting apparatus, modified according to the criteria developed, has been fabricated, Germanium rods have been zone melted. Concurrent experimental work included measurement of resistivity, Hall effect, magneto-Seebeck effect at the melting point, and Peltier effect as applied to zone melting.

Hughes Aircraft Company. WADD TR 60-111.
ORGANIC SEMICONDUCTOR STUDY. J. B. Rust, F. A. Haak, J. P. Nolta. AF 33(616)-6024. June 1960. ASTIA Document No. AD 243913. PB 171177. Order from OTS $1.75.

This report covers the first fifteen months of a study on organic semiconductors under Air Force Contract AF 33(616)-6024. Experimental results for the temperature dependence of the resistivity are presented for thirteen organic compounds. Experimental results regarding the rectification properties of copper phthalocyanine and other metal derivatives of phthalocyanine are presented. A tentative theory based on the formation of a space charge due to the presence of an oxidizing agent in the system has been developed. Preparation for the study of the effects

of high electric fields on anthancene and phenanthrene are discussed and some qualitative results are presented.

WADD TR 60-364.
AN INVESTIGATION OF THE RESPONSE OF PHOTOMULTIPLIER TUBES. Gordon H. Griffith. August 1960. PB 171402. Order from OTS $0.50.

The response of a photomultiplier tube is related to its average sensitivity and can be improved by using only the most sensitive part of the photocathode. This report presents a number of graphs to illustrate the response of a photomultiplier tube as a function of the point on the photocathode surface that is illuminated and to indicate the improvement in symmetry of the isosensitivity lines that may be achieved by varying the shield voltage. While the observed sensitivities were due primarily to the geometry of the cathode-dynode configuration, they were also due partially to the nonuniform response of the cathode surface.

General Electric Company. WADC TR 59-469.
INVESTIGATION OF ORGANIC SEMICONDUCTORS. G. P. Brown, S. Afterbut. September 1960. PB 171340. Order from OTS $2.25.

The specific resistivity as a function of temperature has been determined for a variety of organic compounds including polyacrylonitrile, polyacrylonitrile containing additives, 1, 1-diphenyl-2-picrylhydrazyl, naphthalene and its 1-amino, 1-nitro-, 2-methoxy-, and 2-phenyl derivatives, 1, 4-diphenyl-1, 3-butadiene, 1, 8-diphenyl-1, 3, 5, 7-octatetraene, and several picrates. The resistivities were obtained on materials in various stages of purification, over a wide range of applied voltages, in nitrogen and in air, and in the solid and (whenever feasible) in the liquid state. A zone refiner was designed and used in the purification of several materials. The results obeyed the semiconductor equation, $P = P_o \exp(E/2kT)$; values for the energy gap E and the resistivity at infinite temperature P_o were calculated. A model for conductivity in organic compounds has been advanced postulating that the primary process responsible for conductivity consists in the formation of biradicals. The pertinent literature was surveyed and the available data were tabulated.

Westinghouse Electric Corporation.
WADC TR 59-337, Part II.
SYNTHESIS AND PURIFICATION OF DIELECTRIC MATERIALS. W. C. Divens, D. H. Hogle, D. W. Lewis, P. A. Tierney, T. W. Dakin, D. Berg. AF 33(616)-5979. September 1960. PB 171364. Order from OTS $2.00.

This report describes the progress during the second year on a research program undertaken to prepare pure dielectrics with improved properties for use as electrical insulation at 500°C. The effort has been concentrated on the materials: boron nitride, alumina, and boron phosphide.

Boron nitride has been prepared with better dielectric properties, at 500°C, than any other reported materials. The condition for the hot pressing of the boron nitride disc have been elucidated. Hot pressed boron nitride has been shown to possess electrical properties which are anisotropic.

Anodized aluminum oxide films have good 500°C dielectric properties. The films are polarity sensitive and have a resistance which decreases with increasing field.

The adhesion of the electrode metal to the anodized oxide film has been improved appreciably by first evaporating silicon monoxide in vacuum, and then evaporating gold on top of the silicon monoxide without having released the vacuum.

Dielectric measurements of aluminum oxide films formed by hydrolysis of aluminum isopropoxide have indicated inferior properties for the oxide made in this manner.

Boron phosphide has been synthesized by two means: (1) direct reaction of the elements, boron and phosphorus, and (2) by reaction of boron trichloride, phosphorus and hydrogen.

Fibrous Materials

Harris Research Laboratories, Inc.
WADC TR 58-602.
A SURVEY OF 18-OUNCE BLENDED SERGE FABRICS LABORATORY EVALUATION. John Menkart. AF 33 (616)-54-172; 55-82; 57-20. May 1959. ASTIA Document No. AD 214699. PB 151920. Order from OTS $1.25.

The development of alternates for the 18 ounce wool serge was the object of the investigation. Twenty experimental fabrics were made and their properties were evaluated, in comparison with those of the standard serge, at Quartermaster Research and Engineering Center, Natick, Massachusetts. On the basis of the laboratory data, tentative recommendations are made for the selection of alternates, resulting in a conservation of wool, with the least change in functional properties. Of the fabrics studied, a ternary blend of 70% wool, 20% viscose, and 10% nylon is indicated as providing the closest approach to the wool serge.

Goodyear Aircraft Corp. WADC TR 58-298, Part II.
EXPLORATORY INVESTIGATION OF INORGANIC FIBER REINFORCED INORGANIC LAMINATE. George Margo. AF 33(616)-5251. June 1959. ASTIA Document No. AD 219397.

Part I of WADC TR 58-298 covered the initial evaluation of several different types of laminating cements, primarily with glass fabric-asbestos felt reinforcement, and fabrication and test methods. This phase of the program was designed to complete the cement evaluation and to make a more extensive study of other reinforcements. The reinforcements included crocidolite asbestos fabric or met, amosite asbestos mat, ceramic fiber fabric, pure silica fibers and aluminum coated glass fibers.

It was found that the sodium silicate, magnesium oxysulphate, and aluminum phosphate cements came closest to meeting the program objectives. All other cements were eliminated on the basis of poor strength properties. The glass fabric-asbestos felt combination reinforcement provided the highest strengths, both at room temperature and 1000°F. The crocidolite and amosite materials were difficult to impregnate and showed considerable strength loss at elevated temperature, the ceramic fiber laminates did not exceed 2000 psi in flexural strength and the silica and aluminum coated fibers could not be effectively coated for protection against the corrosive effects of the cements. The highest strength value in flexure, 15,750 psi, was obtained with aluminum phosphate reinforced with acid-resistant glass fabric and asbestos felt. This laminate also had the highest flexural strength and flexural modulus of elasticity at 1000 deg F - 10,640 psi and 2.9×10^6 psi, respectively. The

magnesium oxysulphate laminates demonstrated the best resistance to moisture effects, retaining approximately 70 percent of their original strength. The sodium silicate laminates were second to aluminum phosphates in strength but were particularly sensitive to moisture effects, retaining only 10 to 20 percent of their original strength. The program objectives for flexural modulus of elasticity and specific gravity were met but the laminates fell short on ultimate flexural strength, strength retention after moisture exposure, water absorption, and impact strength. None of the systems had a good balance of the required properties and any selection would be a compromise, dependent on the intended application.

WADC TR 59-250.
INVESTIGATION OF A FUNGICIDAL TANNAGE. Lewellyn G. Picklesimer. June 1959. ASTIA Document No. AD 225706.

A new tannage for leather has been developed. The tanning material is the reaction product of tetrakis (hydroxymethyl) Phosphonium Chloride $(HOCH_2)_4$ PCl and primary or secondary amines. Leather tanned with this material is without the objectionable properties of leather tanned with the phosphonium compound alone.

The fluorinated diphenyl fungicides, 2, 2' dihydroxy 5, 5' difluoro biphenyl and 2, 2' dihydroxy 5, 5' difluoro diphenyl sulfide, have been condensed with various secondary alkyl amines without loss of fungicidal activity and with the advantage of being soluble in aqueous acid solutions.

The two modified fluorinated fungicides, 2 chloro 4 nitro aniline, and p-phenylazoaniline have been reacted with THPC and permanently bound to leather by tanning.

Arthur D. Little, Inc. WADC TR 59-155.
CANDIDATE MATERIALS FOR HIGH TEMPERATURE FABRICS. AF 33(616)-5880. September 1959. PB 161411. Order from OTS $3.00.

This program involved an evaluation of existing data relative to the development and use of high-temperature fabrics for re-entry parachutes. Anticipated environmental and aerodynamic factors of re-entry from satellite orbits were used as a basis for the selection of candidate materials. Among these factors were heating and deceleration rates, surface temperatures, erosion and corrosion rates, strength-to-weight properties at elevated temperatures, porosity, and flexibility. All types of high-temperature materials that could be made into fabric were evaluated, including metals, ceramics, glasses, inorganic and organic polymers, and single crystal systems (whiskers).

Emphasis was placed on a study of forming fibers from a melt, because of the limitations of present techniques with metals and high-melting ceramics. This report presents a new theory on the parameters that govern the ability of a substance to be melt spun into filaments and the maximum cooling time allowed for such spinning.

Although there was very little data on the behavior of fine filaments at the expected environmental conditions, we were able to select commercially available materials that could be used in a high temperature parachute cloth with a minimum of development work. We also defined the type of data necessary to the development of more sophisticated fabrics that would have greater reliability and an extended service temperature.

Department of Engineering Mechanics and Materials, Cornell University. WADC TR 58-684.
MASS TRANSFER COOLING OF PARACHUTE MATERIALS. Rodney H. Cornish, Charles W. Beadle, Franklin J. Ahimas, Kenneth Foster. AF 33(616)-5852. September 1959. PB 161293. Order from OTS $2.25.

Seventy three tests were made on different parachute ribbon materials and protective coatings. These tests were conducted at Mach 5 pressure altitude of 100,000 feet and stagnation temperatures of 700 to 900°F. Most of the effort was devoted to mass transfer cooling (sublimation and melting). Hexachloroethane, Chloroanthraquinone, Methyl-anthraquinone, Hydroxyanthraquinone, and Camphene were applied by various techniques in several thicknesses.

The results of these tests are plotted in the form of: lifetimes parameterized in terms of coating weight, temperature profiles, and heating rates. Tabular data is presented for all of the runs. This data indicates that coating weights on the order of the weight of the ribbons are required to give significant protection at the test conditions and that mechanical protection must be provided to the coatings to improve their efficiency.

Fabric Research Laboratories, Inc.
WADC TR 59-242.
A SURVEY OF 18-OZ. BLENDED SERGE FABRICS: STABILITY OF FABRICS TO MOISTURE AND R. H. MOVEMENT. Norman J. Abbott, Leo Barish, Milton M. Platt. AF 33(616)-54-172; 55-82; 57-20. October 1959. ASTIA Document No. AD 230982. PB 161901. Order from OTS $1.25.

The effect of blending a number of fiber types with wool on the behavior of 18-oz. serges under extremes of humidity was studied. The blended fibers included viscose, nylon, Dacron, Orlon, Acrilan, and Dynel, in amounts up to 30%. The results have shown that some differences exist between the effects of changes in humidity on the nine fabrics examined. With one exception, however, the differences were small, and it would require some practical experience with the fabrics to assess their importance.

The properties studied included dimensional change, fabric deformation, seam puckering, stiffness, moisture regain, and bagging tendency. The only relatively large change in any of these properties which was brought about by changes in humidity was the rather large increase in stiffness which took place in high humidity. This has been attributed to a jamming of the structure due to the swelling of the fibers, and was apparently not related to the type of fiber which was blended with the wool.

In other characteristics the differences which existed between the fabrics are sufficient to differentiate between the fibers, but in general are not considered to be of major practical importance.

Quartermaster Research & Engineering Command, U. S. Army. WADC TR 59-397.
DEVELOPMENT OF SHADE STANDARD AND TOLERANCES FOR USAF FLUE 85 SERVICE OVER COAT. John T. Walwood, Frank J. Risso, Constantine J. Megas, Aldo M. Crugnola. AF 33(616)-53-221. January 1960. PB 161684. Order from OTS $1.75.

The scope of work required by Call No. 5 of CSO&A (33-616)-53-221 involved development of a formulation of high order colorfastness properties, for Air Force Service Overcoat, practical for the Industry to apply routinely, and application of such formulation to the preparation of a new shade standard and a practical range of color tolerances.

The resultant studies here reported have fulfilled the stated requirements. The formulation is colorfast; the tolerance range is of a degree of spacing permissible in end use without apparent inconvenience to the industry in attainment. Colorfastness of the tolerance range relative to standard is such that uniformity in appearance throughout the useful life of the garment seems reasonably assured.

Scientific Oil Compounding Co. WADC TR 59-703.
TREATMENT OF COTTON FABRIC WITH FORMULATIONS OF 2, 2'-DIHYDROXY 5, 5-DIFLUORODIPHENYL SULFIDE. Charles C. Yeager, Jay C. Chapin. AF 33(616)-38797. January 1960. ASTIA Document No. AD 234782.

Formulations of the fungicidal compound 2, 2'-dihydroxy-5, 5'-difluoro-diphenyl sulfide (DDFDS) have been applied to fabric in a commercial textile finishing plant. The material treated was a standard 8.5 ounce corded cotton sateen and the colors used were natural, sage green, and olive green. Two levels of fungicide were applied. The lower amount varied from 0.75 to 1.24 percent by weight and the higher level varied from 1.91 to 2.18 percent by weight of the treated material. The fungicidal formulations applied were both water repellant and non-water repellant. Each combination of formulation was applied to approximately 250 yards of each color of fabric. A total of nearly 7000 yards of material were treated. Results show that the formulations are acceptable for commercial application procedures.

Textile Division, Massachusetts Institute of Technology. WADC TR 59-374.
AIR FLOW CHARACTERISTICS OF PARACHUTE FABRICS AT SIMULATED HIGH ALTITUDES. C. V. Seshadri, G. A. Brown, S. Backer, J. G. Krisik, D. M. Mellen. AF 33(616)-5864. March 1960. ASTIA Document No. AD 270928. PB 161902. Order from OTS $3.00.

The air flow characteristics of parachute canopy cloth have been measured over an unusually wide range of test conditions. High altitude simulated tests (up to 150,000 feet) have shown the cloth to have markedly low flow rates, as may be predicted from a nozzle flow analogy. A method of predicting high altitude behavior has been proposed. Permeabilities of four cloths have been shown to be significantly dependent on their state of stress at the time of air flow measurement. The magnitude of this relationship is observed to be determined by the biaxial stress-strain behavior of each fabric. The air stream deflection tendency of thick canopy material has been verified and its cause investigated. The role of pore geometry in influencing cloth permeability has been explored.

Cook Research Laboratories. WADC TR 59-694.
ANALYSIS OF WEBBING IMPACT DATA AND DETERMINATION OF OPTIMUM INSTRUMENTATION TO BE USED IN CONJUNCTION WITH THE IMPACTING OF WEBBING. R. B. Williams, R. J. Benjamin. AF 33(616)-6440. March 1960. PB 161756. Order from OTS $2.00.

Quantities of data have been obtained at Edwards Air Force Base, California, concerning the impact behavior of nylon webbing. The basic aims of this investigation are:

(1) To evaluate and analyze the methods used to obtain data acquired nylon webbing impact tests conducted at Edwards Air Force Base, California.

(2) To interpret these data and to judge their reliability.

(3) To recommend, if necessary, improved or modified testing methods and instrumentation techniques which would result in obtaining data of greater value in future tests.

Analysis and interpretation of the test data indicated that these data were of intermediate reliability. Certain trends were apparent, but relatively large experimental scatter existed. Possible causes of the scatter were investigated and recommendations were made for improvement of testing methods, equipment, data reduction technique, and data interpretation.

House Glass Corp. WADC TR 58-285, Part II.
DEVELOPMENT OF HIGH MODULUS FIBERS FROM HEAT RESISTANT MATERIALS. J. Frees Brossy, J. D. Provance. AF 33(616)-5263. March 1960.

Earlier work reported in WADC Technical Report 58-285, Part I, involved the investigation of high melting refractory oxide compositions to obtain continuous monofilaments having high modulus of elasticity and tensile strength at temperatures above 1000°F.

Work during the first two months of the period reported herein involved a continuation of effort seeking the development of continuous fiber drawing techniques. Thereafter, effort was devoted solely to composition evaluation and testing of hand-drawn fibers.

Use of hand-drawn fiber techniques allowed rapid fiberization trials and the evaluation of many compositions. However, variation in fiber properties with this technique does not allow specific, clear-cut conclusions. From this study however, several fiberizable oxide compositions have been found which merit further study.

Bjorksten Research Laboratories, Inc. WADC TR 59-699.
DEVELOPMENT OF TEXTILE TYPE VITREOUS SILICA YARNS. W. Wendell Drummond, Burns A. Cash. AF 33(616)-6255. March 1960. PB 161845. Order from OTS $1.50.

Experiments were performed leading to development of methods for producing vitreous silica textile yarns from fused quartz canes or rods by means of electrical heating devices. Both induction and resistance furnace devices were used, best performance being obtained from a resistance unit shielded against radiation losses and operating in a protective atmosphere. Power capabilities of available equipment were insufficient to permit production of a large number of continuous filaments simultaneously, but operation with five filaments were achieved. A limited amount of data on physical properties of filaments and strands were obtained.

Quartermaster Research and Engineering Command. WADD TR 60-294.
DEVELOPMENT OF SHADE STANDARD AND TOLERANCES FOR USAF TAN 505. Frank J. Rizzo, Constantine J. Megas, Alvin O. Ramsley, William B. Bushnell, Ruth J. Evans. May 1960. PB 171062. Order from OTS $1.75.

Shade standards and tolerances for USAF Tan 505 used in hot weather clothing have been developed in three fabric constructions using a formulation which has had considerable industrial application for a number of related shades.

The shade standardization procedure used was based on a technique developed at the Quartermaster Research & Engineering Command, following procedures which are well known in the industry.

The program was monitored by visual as well as colorimetric procedures. The tolerance spacing visually should provide for a reasonable approach to a monotine appearance in uniforms made from two different pieces of fabric in the same construction.

The shade characteristics of the three fabrics are such as to provide for use of any of the fabrics in a single ensemble. Whatever differences occur are more related to fabric texture than to specific differences in color. The colorimetric spacing obtained instrumentally does not coincide with the visual evaluation. The report discusses the reasons for this in detail.

The degree of colorfastness achieved is optimum for the depth of shade.

Quartermaster Research and Engineering Command, U. S. Army. WADD TR 60-150. DEVELOPMENT OF SHADE STANDARDS AND TOLERANCES FOR USAF BLUE 84 AND BLUE 584 SERVICE AND OPTIONAL WEAR UNIFORMS. Frank J. Rizzo, Constantine J. Megas, Alvin O. Remsley, William B. Bushnell, Ruth J. Evans. May 1960. PB 171089. Order from OTS $3.50.

The report covers the development of a number of shade standards and tolerances in USAF Blue 84 used by the USAF in the service uniform and also certain optional wear uniforms, and for Blue 584 used in special components. The standardization procedure developed by the Quartermaster Corps along concepts which have been common in the industry for many years was applied to this development.

As part of this work, an extensive study of dye formulations was accomplished from which new highly colorfast formulations were developed. These formulations, in addition to fulfilling the basic colorfastness requirements, have application characteristics suitable for both large and small industry.

The formulation selected represents somewhat of a compromise, a situation which is normal in the usual interaction between colorfastness properties, application characteristics, availability factors, and end use requirements.

The complete program was monitored by application of both standard visual techniques and by colorimetric analysis based on spectrophotometric data transposed in accordance with the tristimulus functions of the CIE standard observer for colorimetry.

The tolerance ranges which have been developed around each of the standards in the several different fabric constructions are visually well spaced, although the colorimetric spacing of the instrumental technique does not coincide with visual judgment. The reasons for this situation are analyzed, and it is concluded that a further review of the theory of color mixtures must be made for the type of blending operations which are involved in a program of shade standard and tolerances of the type being reported.

Carborundum Co. WADD TR 60-244. CONTINUOUS FILAMENT CERAMIC FIBERS. W. A. Lambertson, D. B. Aiken, E. H. Girard. AF 33(616)-6246. June 1960. PB 171061. Order from OTS $2.25.

Melting and fining of refractory glass compositions and drawing of continous filament ceramic fibers have been carried out in a dry, inert-atmosphere glovebox, which permitted the use of refractory metals, graphite, and boron nitride as crucible and heater materials, and prevented the adsorption of moisture by the filaments.

Several refractory glasses in the baria-alumina-silica, calcia-alumina-silica, and magnesia-alumina-silica systems were successfully determined at room and elevated temperatures. Values as high as 138,000 pounds per square inch at room temperature and 132,000 pounds per square inch at 1500°F (815°C) were obtained.

Wyandotte Chemical Corp. WADD TR 60-198. DEVELOPMENT OF A FABRIC TO PROVIDE PERSONAL PROTECTION FROM TOXIC MATERIALS. Raymond G. Spain, Alexander V. Sanger. AF 33(616)-6501. June 1960. PB 171058. Order from OTS $1.25.

This report describes research directed to the development of a coated fabric to resist the chemical effects of and be impermeable to high energy fuels (HEF).

This program included the design and fabrication of special test equipment and of a laboratory fabric coating apparatus.

A base fabric (Dacron) was selected on the basis of resistance to HEF, and the majority of the effort was directed toward the coating of this fabric with fluoroelastomer based compositions and the necessary subsequent testing.

A coated fabric was approved and 100 yds.^{2} of this material were prepared and delivered to the Nonmetallic Materials Laboratory, Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division.

Quartermaster Research and Engineering Command, U. S. Army. WADD TR 60-151. DEVELOPMENT OF SHADE STANDARDS AND TOLERANCES FOR SILVER TAN 193. Frank J. Rizzo, Constantine J. Megas, Alvin O. Ramsley, John T. Walwood, Ruth J. Evans. AF 33(616)-53-221 and 54-107. June 1960. PB 171011. Order from OTS $2.75.

Shade standards and tolerances have been developed for tropical and gabardine weave fabrics in all wool, wool/polyester and polyester/rayon blends. Tolerance spacing is such as to permit interchange of uniform components made for the blend fabrics with components made of all wool so long as the weave construction is common. The limiting factor to such usage is considered to be differences in appearance originating with the gloss of the fibers and not the chromatic and lightness deviations of the tolerance range. Colorfastness is good at 40 Standard Fading Hours for all fabrics, except for the polyester/rayon tropical where the vat dyed rayon permits a rating of good at 120 Standard Fading Hours. Dye formulations selected are practical for routing application and stable to finishing factors.

Fabric Research Laboratories, Inc. WADD TR 60-9. A POSSIBLE APPLICATION OF ORGANIC FIBELS IN HIGH TEMPERATURE ENVIRONMENT. Myron J. Coplan, Donald H. Powers, Jr., Leo Barish, Emery I. Valko. AF 33(616)-6234. June 1960. PB 171020. Order from OTS $2.75.

It is taken as a premise that fabrics used in re-entry drag parachutes (1) require typical textile properties up to deployment but (2) that they need not survive in their original chemical state subsequent to deployment provided the decay occasioned by re-entry conditions (heat, active oxygen, irradiation) does not interfere with the deceleration function. The possible utility of linear organic polymers as the principal chemical species in such materials is examined.

Details of re-entry environment conditions, as applicable to the problem, are developed. Some of the textile qualities required of the parachute fabric up to development are reduced to quantitative criteria. Typical organic, metallic and ceramic materials are contrasted and the advantages of the first class are defined.

Recognizing that the principal deficiency of the organic polymer fibers resides in their thermal and thermochemical sensitivity, a survey of the causes of these deficiencies is given. Some speculative arguments are advanced relative to means of overcoming the deficiencies. In addition, a broad concept for a means of employing the organic materials is proposed wherein thermal degeneration is to be anticipated and even used to advantage.

A detailed account of the principles of the so-called "metamorphic" fiber system is given. A number of specific thermochemical reaction schemes and polymer systems are proposed.

Unqualified conclusions regarding the feasibility of the basic principle and any of the specific schemes are not possible in the light of the documented need for further experimental effort.

Quartermaster Research & Engineering Command. WADD TR 60-295.

DEVELOPMENT OF SHADE STANDARD AND TOLERANCES FOR USAF BLUE 157 NYLON RAYON POPLIN AND POLYVINYL BUTYRAL COATED NYLON TWILL. Frank J. Rizzo, Constantine J. Megas, Alvin O. Ramsley, Ruth J. Evans. AF 33(616)-53-221 & MIPR (33-616)56-15. July 1960. PB 171153. Order from OTS $1.25.

Shade standards and tolerances for Blue 157 have been developed in two separate fabric constructions used in USAF rainwear, namely, the cloth, nylon-rayon poplin and the cloth, rayon, twill, PVB coated. The formulation used for the former fabric was one prescribed by the USAF. The formulation of the latter was one which was selected after a rather extensive study of colorants. The durability of the formulation of the nylon-rayon poplin to degradative factors is dependent upon the effectiveness of the durable water repellent treatment. The formulation used on the nylon twill has inherently good colorfastness. The data reported show that good colorfastness was obtained in both instances. The tolerance spacing and general standardization procedure utilized was one developed by the Quartermaster Research and Engineering Command following procedures which are well known in the industry.

The program was monitored by both instrumental and visual methods and the tolerance spacing provided is such as to be in harmony with the essential military requirements of good appearance and the recognized capabilities of the industry. The tolerance spacing on the nylon-rayon fabric is slightly wider than that on the coated fabric. This was occasioned by the greater complexibility of the dyeing system required for an ortho blend against a single fiber system. In addition, coating tends to a narrowing of the shade range from the precoat condition. These tolerances as spaced colorimetrically are different from their visual appearance. The latter has been accepted as the important consideration in light of the fact that the colorimetric theory is under question. Some of the possible reasons for these deviations are enumerated in the report.

WADD TR 60-385

FLAME BARRIER CHARACTERISTICS OF TEXTILE FIBERS. Edward L. McLeod. July 1960. PB 171136. Order from OTS $1.25.

Methods of sample preparation and testing were developed to determine the relative performance of various textile fibers as impedients to applied flame. Tests were conducted on randomly oriented compressed fiber pads.

It was found that residual products of ash-forming fibers continued to obstruct applied flame which temperatures detected on the opposing side increased at a varying rate with time. Melting and non-ash forming fibers receded from applied flame without an increase in detected temperature until the point of complete flame penetration. In some instances, nylon 66 showed some departure from the normal pattern of other melt type fibers. It was established that increased weight resulted in increased temperature lag time while the most favorable density for each fiber occurred at some intermediate degree of concentration.

Fabric Research Laboratories, Inc. WADD TR 60-511, Part I.

INVESTIGATION OF THE HIGH SPEED IMPACT BEHAVIOR OF FIBROUS MATERIALS. PART I. Design and Apparatus. Chauncey C. Chu, Robert J. Coskren, Henry M. Morgan. AF 33(616)-6321. September 1960. PB 171311. Order from OTS $1.00.

A high speed impact test machine has been designed, constructed, and calibrated to test parachute components at high rates of loading. This instrument is capable of rupturing materials of up to 10,000 pounds static breaking strength at velocities of from 200 to 750 feet per second. The impacting force is applied by a free flying missile launched by a gas gun utilizing either nitrogen or helium gas at moderately low pressures. The gun has a bore of 2.5 inches and fires missiles weighing up to 10 pounds.

Pertinent data are obtained by means of multiple exposure photography using a multi-microflash lighting source which provides a maximum of fifteen separate flashes spaced at predetermined intervals of between 10 and 10,000 microseconds. The resulting photograph records the specimen and the impacting missile before, during and after the impact. Measurement of the distances between successive exposures yields information such as the breaking strength, the extension to rupture, and the energy absorbed by the specimen.

Narricot Corporation. WADD TR 60-252.

THE DESIGN AND EVALUATION OF HEAT STABILIZED TAPES AND WEBS. Jacob Serbin, Herman Becker. AF 33(616)-39447. October 1960. PB 171486. Order from OTS $2.00.

The basic purpose of this work was to design and evaluate a series of 13 representative webbings, tapes and ribbons, which would maintain maximum breaking strength and minimum shrinkage when subjected to a temperature of 250°F for a period of five hours

Each of the 13 samples were to be made from:

1. Dupont Type 330 or 700 nylon.
2. Chemstrand Type RHB nylon.
3. Dupont Type 51 or 52 Dacron

A heat stabilization process was found necessary to accomplish the desired shrinkage. An experimental pilot program, involving three types of stabilization processes, indicated that a "heat stabilized" continuous method (HSCM) using a dry air oven was the most practical method.

Investigation has shown that, with due consideration given to the strength per weight ratio, a woven end item of nylon or Dacron can be stabilized with a measureable degree of success. Additional information was sought

to ascertain strength and shrinkage of all items at room temperatures, and also after five hours at 300°F. All items were checked for seam efficiencies in the natural state and after exposure to 250°F and 300°F for five hours.

Fifty (50) yards of each of the 39 samples were delivered to WADD for evaluation. The decision was made to use nylon materials. The complete set of thirteen (13) samples were finally selected from among the Dupont or Chemstrand nylon webbings, tapes and ribbons.

Southern Research Institute. WADD TR 60-510. DETERIORATION OF TEXTILE MATERIALS BY ULTRAVIOLET LIGHT. William S. Wilcox, Charles V. Stephenson, James C. Lacey, Jr., Bobby C. Moses. AF 33(616)-6565. October 1960. PB 171417. Order from OTS $3.00.

This is the report on a project to determine the effects of ultraviolet radiation on polyethylene, Teflon, nylon, Mylar, and Acrilan. These materials have been irradiated in a vacuum and in an inert atmosphere of nitrogen.

Deterioration of these materials is faster for irradiation with short wavelength ultraviolet than it is for long wavelength radiation. Deterioration of polyethylene, nylon, Mylar, and Acrilan is faster in a nitrogen atmosphere than it is in a vacuum. However, Teflon deteriorates faster in a vacuum.

Experimental evidence as to the processes of deterioration are discussed in this report and mechanisms are proposed by which deterioration could take place.

WADD TN 60-253.
ULTRA VIOLET RADIATION RESISTANT MATERIALS FOR DECELERATION. Joyce C. McGrath. December 1960. ASTIA Document No. AD 258595. Order from OTS $1.00.

Two improved types of nylon yarns (1) Type 330, 30 denier, a higher strength than the conventional nylon yarn, were developed by the E. I. duPont de Nemours and Company, Incorporated, to be used in materials for personnel and other type decelerators.

Cloth, woven of the two types of nylon yarn, in both natural and International Orange were exposed to (1) outdoor weathering at the Naval Auxiliary Air Station, El Centro, California for periods of 1, 2, 3, 4, 5 and 6 weeks, (2) Accelerated aging in a Weather-O-Meter, Type X1A, for periods of 20, 40, 60, 80, 100 and 150 hours and (3) a temperature of 300°F for 8 to 16 hours.

Data obtained indicates that the Type 330 nylon cloth in natural color retains a very high percentage of strength under all three exposure conditions while the Type 330, International Orange and the Type 300 in both natural and International Orange deteriorated at a very rapid rate after exposure to both outdoor weathering and accelerated aging. The Type 300, natural showed the greatest loss in strength after heat exposure of 8 to 16 hours.

(It was determined, in supplemental tests, by the E. I. duPont de Nemours Company that the type of dye used to obtain the International Orange color, was the chief factor in strength loss of the dyed Type 330 cloth. The Type 300 was not developed as a heat or ultra-violet light resistant yarn).

TR 4609.
INVESTIGATION OF AIRCRAFT CLEANING COMPOUNDS. Howard Packer. February 1941.

TR 5636.
STORAGE CHARACTERISTICS OF FUELS UNDER SEVERE AND MODERATE CLIMATIC STORAGE CONDITIONS. Robert W. Altman. September 1947.

TR 5895 (R).
THE INFLAMMABILITY CHARACTERISTICS OF LIQUID FUELS. L. T. Taylor, Captain. July 1949.

TR 5944.
ANALYTICAL METHODS FOR DETERMINING SOLUBILITY OF WATER IN HYDROCARBONS. Bernard Rubin and Robert J. Burger. May 1950.

Shell Development Company. TR 6625.
STABILITY OF JET (TURBINE) FUELS IN STORAGE. C. A. Cole and A. C. Nixon. AF 33(038)-7277. January 1952.

The general objective of this investigation is to increase the permissible storage life of JP-3 jet fuels by employing methods which will result in the utilization of the simplest possible refinery treatments and which will result in maximum availability. This work is being done in two phases, in the laboratory using accelerated and hot room aging methods and under desert storage conditions at El Centro, California.

The early laboratory work was done under Navy Contract No. NOa(s)9970 and was designed primarily to survey possible experimental methods for investigating stability of jet fuels. On the basis of this work it was concluded that reasonable correlations could be established between accelerated and hot room aging and that the extent of deterioration could best be measured by use of the 500°F steam jet gum procedure. This procedure was found to give much more reproducible and consistent results than the 400°F air jet procedure for determining gum. Subsequently a variety of fuels have been tested to determine the effect of fuel properties, treatments, contaminants, composition and minor components on the storage stability. Numerous compounds have been tested for their effect as oxidation inhibitors. Some data have been accumulated on the effect of gum formation on the freezing point of the fuel. The problem of predicting stability from laboratory data has been given some consideration.

About 30 samples of jet fuels of various types were obtained from the major crude sources of the world (excluding Russia) and were put in storage at the El Centro Naval Air Station, El Centro, California. Containers used for these fuels consist of regular black iron 55 gallon drums and 5 gallon black iron cans. The effect of water contamination was studied in the 5 gallon cans by including similar series with distilled and sea water. Parallel experiments with vented and unvented cans have been run. Some fuels were tested also in 5 gallon galvanized iron and aluminum containers. The extent of deterioration in all these samples has been observed and correlated with the laboratory results.

The results of this study may be summarized as follows: the stability decreases with an increase in the

end point of the fuel and an increase in the proporation of cracked components, high boiling cracked components being more deleterious than low boiling. Thermally cracked materials are more detrimental than catalytically cracked, particularly in the gas oil range. Soluble copper has an adverse effect on stability in thermally cracked and some straight run but not in catalytically cracked fuels. The presence of metallic copper is adverse in some SR fuels and in blends containing thermally cracked components. Generally speaking, water, either sea or distilled, has a beneficial effect. Caustic treatment, particularly of catalytically cracked components, and acid treating show some favorable effect (particularly on inhibitor susceptibility) although the effect is not as marked as with gasoline. Some sulfur and nitrogen compounds catalyze the rate of gum formation. Color develops more rapidly than gum or aging.

Conventional gasoline inhibitors have, generally, little beneficial effect on the stability of these fuels. This includes the three inhibitors permitted in the MIL-F-5624 and 5161 specifications. A number of compounds have been found which show promise as inhibitors, particularly on the minimizing of insoluble gum. Dispersants have some beneficial effect in dispersing insoluble gum.

Only limited data are available for the effect of gum formation on freezing point but in two cases the freezing point has been increased significantly by the formation of rather moderate amounts of gum. However, the effect seems to be quite variable.

Gum forms in jet fuel as either soluble or insoluble gum and the latter may be either flocculant or adherent, depending on its physical nature. It is found that the soluble gum can reach quite high values (of the order of 1%) without resulting in the formation of any significant amount of insoluble gum. On the other hand, insoluble gum has been found in fuels containing less than 0.1% total gum. Accelerated conditions generally produce a greater proportion of insoluble gum than do normal aging conditions. Insoluble gum contains appreciably more oxygen than does soluble gum. It is not known if the soluble gum is a precursor of insoluble gum or not but it is possible that they are formed by different mechanisms. Both types of gum always contain more S and N_2 than does the substrate.

Elevated temperatures and oxygen pressures (such as are used in the specification test) are slightly less and more severe (respectively) than they are with cracked gasolines.

Correlations between accelerated, hot room and desert aging for the present fuels indicate that the 16 hour aging period in the MIL-F-5624 specifications is equivalent to more than 3 years of desert storage. The previous work (with U. S. samples only) indicated it was equivalent to about 2-1/3 years. In either case the specification appears unduly restrictive, in view of the present storage requirements of 3 years temperature zone bulk and 90 days desert drum storage.

This work is continuing with particular emphasis on the effect of inhibitors and on methods of minimising the formation of insoluble residues. The effect of the introduction of JP-4 specifications will be assessed. It has not been possible to analyse, as yet, all the data which have been obtained during the course of the present investigation. This will be done and the results presented in the next report.

United States Department of the Interior. TR 52-35.
RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT FUELS. G. W. Jones, M. G. Zabetakis, and J. K. Richmond. AF 33(038)-50-1293-E. June 1952. PB 127339. Order from LC, Mi $5.70, Ph $16.80.

The results of limit of flammability, limit of ignitibility, and ignition temperature tests conducted on aircraft fuel vapor-air mixtures by the U. S. Bureau of Mines Gaseous Explosions Laboratory between February 19, 1950 and February 19, 1952 are presented. Two aviation gasolines grades 100/130 and 115/145, and two jet fuels grades JP-1 and JP-3, were investigated. A limited amount of work was done on the ignitibility of JP-1 mists and sprays, and on the ignition temperatures of aircraft hydraulic fluid AN-O-366.

In addition to the above results, sections are included on definitions and theory, and apparatus used for the investigation is described.

Northrop Aircraft, Inc. TR 52-100.
IMPROVEMENT OF JET ENGINE DESCALING PROCEDURE. G. M. Bryan. AF 33(038)-23310. August 1952. PB 110879. Order from LC, Mi $3.00, Ph $8.75.

X-ray diffraction analysis of the Inconel scale on combustion tube inner liners revealed that it is composed of nickel oxide, the major component, and lesser amounts of chromium oxide and iron oxide. Metallographic examination demonstrated that heavy scale is associated with precipitation, possibly carbides at the grain boundaries, a fact which can explain intergranular corrosion of scaled Inconel in acid solutions.

Inconel scale could not be taken off in neutral or alkaline solvents but several acid solutions were found which remove most of the scale without seriously attacking the base metal. Oxidizing pretreatments, particularly with the alkaline permanganate solution in current use by the Air Force, were shown to promote efficient acid pickling.

Physical tests of Inconel specimens descaled with the nitric acid-ferric chloride solution revealed that high temperature pickling (160°F) caused a severe loss in tensile strength whereas room temperature pickling caused no appreciable loss in tensile strength.

A full scale test of the nitric acid-ferric chloride solution was performed at Norton Air Force Base, San Bernardino, and satisfactory results were achieved.

Southwest Research Institute, San Antonio, Texas. TR 52-102.
DEVELOPMENT OF TEST METHODS FOR ANTI-SEIZE COMPOUNDS. J. W. Cunningham. AF 33(038)-22805. September 1952.

Armour Research Foundation. TR 52-53.
STUDIES TO DETERMINE THE ELECTRICAL AND PHYSICAL PROPERTIES OF AVIATION FUEL. C. C. Petersen. AF 33(038)-3793. September 1952. PB 110968. Order from LC, Mi $7.00, Ph $25.00.

Shell Development Company. TR 6625, Suppl.
STABILITY OF JET (TURBINE) FUELS IN STORAGE. C. A. Cole and A. C. Nixon. AF 33(038)-7277. October 1952.

This is the final report under Supplemental Agreement Number One (May 1 to August 15, 1951) of Contract No. AF 33(038)-7277 on the storage stability of jet (turbine) fuels. This report briefly discusses proposed work to be carried out under the second supplemental agreement as well as recent progress made under the first extension.

Accelerated aging tests on four emergency fuels,

produced in accordance with the requirements of the Military Petroleum Advisory Board Questionnaire, show them to be relatively stable with respect to total gum formed, although significant quantities of insolubles comprised part of the total. Data are also presented which confirm earlier results showing that iron and aluminum as container materials have no significant effect on fuel stability. A rapid filtration test designed for routine work to show relative filter clogging tendencies of fuels is discussed. Water and iron in combination appear to promote the formation of insoluble gum under hot-room conditions of aging although total gum is reduced. Further study of the correlations between desert and accelerated aging shows that accelerated aging tests are relatively more severe on cracked fuels than straight run fuels in comparison with the effect of desert storage.

Foster D. Snell, Incorporated. TR 53-147.
NONTOXIC SOLVENT FOR CLEANING AIRCRAFT ENGINES AT EXTREMELY LOW TEMPERATURES. Bernard Berkeley and Isidore Shafiroff. AF 33(616)-36. November 1953.

Two aircraft engine degreasers consisting essentially of methylene chloride and aliphatic petroleum hydrocarbon were recommended on the basis of laboratory evaluations as being suitable for use in arctic operations. Toxicity, flash point, vapor pressure and stability data on known lowmelting point solvents were compiled and screened for the purpose of selecting basic and additive ingredients of the degreasers. A correlation between evaporation rates at -65°F and 77°F was established for pure compounds. The results of previous government field tests dealing with the evaluation of low temperature engine degreasers were proved to be misleading and incorrect. Synthetic soiling mixtures comparable to actual engine soil were created for this project on the basis of field reports and laboratory tests with engine soil. The recommended degreasers were evaluated for detergency properties, flash point, corrosive action on metals, effect on accessory equipment, toxicity and evaporation rates.

Shell Development Company. TR 53-63.
STORAGE STABILITY OF JET TURBINE FUELS. C. A. Cole and A. C. Nixon. AF 18(600)-37. November 1953.

This investigation was initiated to ascertain the variables influencing the storage stability of jet fuels and to determine the simplest and most practical methods of controlling these variables for a resultant increase in the permissible storage life of the fuels. The work is being done both under laboratory aging and desert field storage conditions. An early program under Navy sponsorship (Contract No. (N0a(S)9970) surveyed and developed the various methods of determining and measuring stability of jet fuels. With development of the 500°F steam jet gum evaporation unit and the use of the accelerated aging procedure, it was decided that reasonable predictions of stability in terms of field storage could be made if correlations between field and laboratory aging conditions were established. A subsequent Air Force Contract (No. AF 33(038)-7277) led to the limited determination of these relationships as well as the study of various factors influencing stability, such as fuel type, contamination, inhibition, treatment, storage temperature and minor components. During the present contract, work on all of these phases of study was continued and extended to include the effects of gum on fuel filterability, dielectric constant and blending compatibility.

The results of the present study may be summarized as follows: Both soluble and metallic copper have an adverse effect on stability. Sea water in the presence of iron appears to promote insoluble gum formation; however, water, generally, reduces soluble gum formation. High boiling materials, contribute more to the instability of a blend than do low boiling materials. The average thermally cracked fuel is more unstable than the average catalytically cracked fuel. Some sulfur and nitrogen compounds catalyze gum formation.

A large number of compounds have been tested as potential jet fuel inhibitors. None tested, including the specification inhibitors, have shown consistent stabilizing action.

The freezing point of a fuel can change with aging of the fuel, but no correlation exists between the quantity of gum formed and the freezing point change. No simple relationship was observed between stability and unsaturation, conjugated diolefins, nitrogen, sulfur or peroxide content.

Correlations between accelerated and desert aging for all fuels tested indicate that 5 hours' aging (at 100°C and 100 psig O_2) is about equivalent to two years of desert drum storage. Fuels correlated on the basis of type show wide variations from this relationship, however.

A method for determining the constant flow filterability characteristics of jet fuels at any practical temperature has been developed which required only 100 ml of fuel. Although only scant data are available as yet, the method appears to give good correlation with large scale results.

On the basis of present data, soluble gum does not appear to affect filterability characteristics. Water, if not present above the saturation point of the fuel, does not affect filterability at temperatures as low as -30°C (no data are available with excess water). Insoluble gum is important in determining the filter clogging tendencies of a fuel, but no relationship exists between the quantity of insolubles present and the tendency of the fuel to clog a filter.

It was found that in some cases quite small quantities of insoluble gum could markedly influence the filter clogging effect of a fuel. This effect was strongly accentuated by lowering the temperature.

Moderate quantities of gum do not affect the dielectric constant of a fuel significantly.

Compatibility of various components of a blend is not affected in most cases by a moderate amount of aging before blending.

This investigation is continuing with emphasis on methods of improving stability by inhibition and treatment utilizing practical jet fuel components and blends and model compounds.

United States Department of the Interior. TR 52-35., Suppl.
RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT FUELS. G. W. Jones, M. G. Zabetakis, G. S. Scott and A. L. Furno. AF 18(600)-151. January 1954. ASTIA Document No. AD 27722. PB 1273395. Order from LC, Mi $3.90, Ph $10.80.

The results of limit of flammability, limit of ignitibility, and ignition temperature tests conducted on aircraft fuel vapor-air mixtures, with various quantities of added nitrogen and carbon dioxide, by the U. S. Bureau of Mines Gas Explosions Branch between 1 February 1952 and 1 February 1953 are presented. Two aviation gasolines grades 100/130 and 115/145, and three jet fuels grades JP-1, JP-3 and JP-4 were used in the investigations.

Southwest Research Institute. TR 53-197.
DEVELOPMENT OF TEST METHODS FOR ANTISEIZE COMPOUNDS. John W. Cunningham. AF 33(038)-22805. February 1954. PB 134774. Order from LC, Mi $8.40, Ph $28.80.

Initial development of a tapered pin seizure test method and testing device to evaluate antiseize compounds (WADC TR 52-102, May 1952) indicated the need for modification of both method and device in order to increase sensitivity, determine the effect of certain variables on the effectiveness of antiseize compounds, simplify the comparison of individual test results, and determine the validity of the tapered pin test method as a means of predicting antiseize compound performance under actual field conditions. To meet these requirements, the tapered pin seizure tester was modified to provide a progressively increasing contact pressure. Tests were conducted to determine the relationship to the effectiveness of antiseize compounds of varied time and temperature exposure, different specimen materials, various specimen surface finishes, specimen assembly design modifications, and vibration at varying frequencies and amplitudes. Visually plotted averages of individual test results were transposed to rectangular coordinates. Antiseize compounds applied to both tapered pin and threaded specimen assemblies of identical materials were tested under identical time and temperature conditions to determine the extent to which the test results were correlative.

In general, the modified and improved tapered pin seizure test method provides more extensive information of greater validity regarding the effectiveness of antiseize compounds. It is believed, however, that the validity and reliability of the data obtained could be greatly increased by further development to facilitate torque measurement, to permit the direct recording of test data on rectangular coordinates, to assure more accurate transmission of thrust load, and to provide a continuous increase in contact pressure.

Shell Development Company. WADC TR 53-63 (Sup. 1).
STORAGE STABILITY OF JET TURBINE FUELS. C. A. Cole, A. C. Nixon. AF 18(600)-37. July 1954.

The present phase of the investigation of jet fuel stability has been concerned with the evaluation of practical methods of improving stability through the employment of existing conventional methods of treatment and inhibition. In addition to this, studies to determine the effects of storage variables on gum formation and the influence of aging on such fuel characteristics as filterability and freezing point have been continued.

In the treatment study attention has been focused primarily on gas oil components since previous work has demonstrated that the stability of the final blend is often dominated by the properties of this fraction. The results of this work may be summarized as follows: Relatively severe treatments with sulfuric acid, caustic soda, clay, combinations of the three or mild hydrogenation did not improve the inhibitor susceptibility of any of the fuels tested. Inhibitor response was observed under accelerated aging conditions in the case of full range fuels in which catalytically cracked gas oil made up the sole heavy component. This response, however, became less marked, or negligible, under milder conditions of aging such as in the oven at 70°C, or in the hot room at 110°F.

Considerable improvement in stability was observed in the case of the more unstable cracked blends which had received sulfuric acid, or combinations of treatments including sulfuric acid or hydrogenation. In cases of improved stability, following treatment, significant reductions in maleic anhydride values, mercaptan sulfur, total sulfur and total nitrogen values were observed. Little change in olefin content was obtained following sulfuric acid treatment singly or in combinations of treatments but large reductions in this value were effected by hydrogenation. Hydrogenation appeared superior from the standpoints of stability improvement and fuel loss due to treatment. Treatment improves filterability of ovenaged samples in most cases only in so far as it improves stability with respect to flocculant insoluble gum.

Oxygen availability in desert aging containers has been shown to be important in determining the extent of fuel degradation during storage. The oxygen content of the vapor spaces of drum samples following the summer of 1952 ranged from 0 to 6% for cracked fuels and 1.5 to 17% for straight run fuels. Samples with fuel to air ratios of 9 have shown much lower gum values than the same fuels aged at 1/1 ratios. The JP-4 fuels have a greater inherent instability than their JP-3 equivalents due to their lower vapor pressure and the consequent greater partial pressure of oxygen in the vapor space of the storage containers. Although fuel insoluble gum levels have shown changes during the last year of desert storage from a decrease to an increase, the filter clogging tendencies of the fuels have in most cases increased. Decreases in MAV values greater than 2 meq/liter with these fuels have usually been accompanied by significant increases in the quantities of soluble and insoluble gum formed. Several inhibitors which appeared promising in the laboratory have not been effective in reducing gum formation on further evaluation in desert storage tests.

Supplemental data on the effect of fuel aging on dielectric constant have confirmed earlier work and show that neither aging time nor soluble or insoluble gum has a significant effect on this value.

United States Department of the Interior. WADC TR 52-35 (Sup. 2).
RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT FUELS. G. W. Jones, M. G. Zabetakis, G. S. Scott, A. L. Furno. AF 18(600)-151. August 1954. ASTIA Document No. AD 47525. PB 127339s2. Order from LC, Mi $3.30, Ph $7.80.

The results of limit-of-flammability, limit-of-ignitibility, limit-of-propagation and ignition temperature tests conducted on aircraft fuel and pure hydrocarbon vapor-air mixtures, with various quantities of added nitrogen and carbon dioxide, by the U. S. Bureau of Mines, Gas Explosions Branch between 1 February 1953 and 1 February 1954 are presented. Two aviation gasolines, three jet fuels and six pure hydrocarbon fuel components were used in the investigations.

Shell Development Company. WADC TR 54-328.
CHROMATOGRAPHIC DETERMINATION OF GUM IN FUELS. A. C. Nixon, T. Skei, H. S. Knight. AF 18 (600)-37. September 1954.

A chromatographic method of determining gum in small samples has been developed in order to solve problems in the investigation of jet fuel stability being carried out for the Air Force. Although the method was primarily intended for use with experimental fuels and components of limited availability, the simplicity, rapidity and ease of application may make it attractive under other circumstances.

The present introductory report deals with the use of this method with jet and diesel fuels and light fuel oils where its agreement with current procedure is adequate. Preliminary data suggest it may also be

applicable to gasolines. Further investigation of the method is in progress to establish optimum procedures.

AF TR 5636 (Suppl. 1).
THE STORAGE CHARACTERISTICS OF FUELS UNDER SEVERE AND MODERATE CLIMATIC STORAGE CONDITIONS. Robert W. Altman. December 1954.

This program comprised a study of the stability of conventional reciprocating engine type fuels and jet propulsion fuels in an attempt to obtain correlation with the results of desert fuel storage presented in the basic report.

Sixty-one reciprocating engine fuels were aged under moderate outdoor climatic conditions at Wright-Patterson Air Force Base, and eighteen jet engine fuels were subjected to aging in a hot room maintained at 130°±2°F. Subsequent to aging under these conditions for various interim periods, these fuels were tested in the laboratory for their residue content by the ASTM air-jet evaporation method.

A majority of the reciprocating type fuels after 59 months under moderate climatic storage conditions had a gum content above the 6.0 milligrams per 100 milliliters of fuel level which was adopted at the outset of the test as the maximum allowable gum content. At 12 months the jet fuels in hot room storage exhibited such good stability that no further tests were conducted until the 35th month of this storage. At that time seven exceeded the 6.0 milligrams per 100 milliliters of fuel level.

AF TR 5713 (Suppl. 1).
THE DEVELOPMENT AND EVALUATION OF PAINT REMOVER USED BY THE U. S. AIR FORCE.
Sam Collis. January 1955.

Paint and lacquer remover formulations containing solvents other than methylene chloride were investigated. Materials evaluated were those submitted by manufacturers, and commonly available solvents and chemicals.

A number of solvents were evaluated for their ability to remove paint and lacquer coatings from metal. Materials for use as surface active agents, activating agents, thickeners, and evaporation retardants were evaluated.

The best experimental formulation developed was nitromethane-toluene-ethanol solvent mixture with a dibutyl amine - moniosopropyl amineadipic acid thickener, a surface active agent, and a paraffin evaporation retardant. This formulation is considered unsatisfactory for Air Force use due to slow paint removal and poor storage stability.

Many of the materials tested were not developed or intended by the manufacture for the conditions to which they were subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

U. S. Dept. of the Interior. WADC TR 52-35 (Suppl. 3).
RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT FUELS. G. W. Jones, M. G. Zabetakis, G. S. Scott, A. L. Furno. AF 18(600)-151. February 1955. PB 127339 s.3. Order from LC, Mi $2.70, Ph $4.80.

The results of limits of flammability, limit of propagation and ignition temperature tests conducted on aircraft oil and on aircraft fuel components in air by the U. S. Bureau of Mines, Gas Explosions Branch, between 1 February 1954 and 1 October 1954 are presented. One aircraft oil and a number of pure paraffin and aromatic hydrocarbons were used in the investigations.

Shell Development Co. WADC TR 53-63 (Part 2).
STABILITY OF JET (TURBINE) FUELS IN STORAGE. Charles A. Cole, Harry B. Minor, Alan C. Nixon, Thurston Skei, Roy E. Thorpe. AF 18(600)-37. February 1955.

Various methods for improving jet fuel stability have been investigated at the Emeryville Research Center of Shell Development Company. Mild hydrogenation of cracked gas oil components appears capable of increasing fuel availability. No effective gum inhibitors have yet been found for desert storage conditions. The effects of other factors on desert storage are summarized. Electron microscopic examination showed filterability to be related to the type of insoluble material formed in aged fuels. The effects of inhibitors, dispersants and fuels were studied in a laboratory test which measured filter clogging tendencies under conditions simulating those in aircraft fuel-oil heat exchangers. Fractionation by distillation and chromatography of a catalytically cracked gas oil into its type components showed that stability under mild aging conditions generally decreased with increasing boiling point and with increasing olefinicity. For this phase of the program, a chromatographic method (chromatagum) was developed for determining gum in small samples.

This investigation is continuing.

Shell Development Company.
WADC TR 54-328 (Part 2).
CHROMATOGRAPHIC DETERMINATION OF GUM IN FUELS. H. S. Knight, T. Skei, A. C. Nixon, S. Groennings. AF 18(600)-37. March 1955. PB 111861. Order from OTS $0.50.

A novel method for the determination of soluble gum in jet fuel, gas oil and possibly also in gasoline was introduced in a previous report. The gum content is related to the length of a brown zone observed when the fuel is displaced over silica gel in a small chromatographic column with α-methylnaphthalene as solvent and acetone as eluent. This method is superior to the conventional procedure involving evaporation at elevated temperatures in a jet of steam because it is much simpler and permits the use of very small samples which is advantageous in research work on fuel stability.

The present, supplementary report deals with improvements of this "chromatogum" method. The purification of α-methylnaphthalene has been made more effective, the necessity of using a closely specified amount of sample has been eliminated, and a narrower column has been designed to improve the reliability even though smaller samples are employed. Also, the method appears to be applicable to insoluble gum from jet fuels and many gas oils.

WADC TR 54-596.
SPECTROPHOTOMETRIC - CUPRETHOL METHOD FOR THE QUANTITATIVE DETERMINATION OF COPPER IN AVIATION FUELS. O. M. Ballentine. May 1955. PB 111888. Order from OTS $0.50.

Minneapolis-Honeywell Regulator Company.
WADC TR 55-219.
INVESTIGATION OF THE ELECTRICAL AND PHYSICAL CHARACTERISTICS OF AIRCRAFT FUELS.
Richard E. Johnson. AF 33(616)-2504. January 1956. ASTIA Document No. AD 92585. PB 132224. Order from LC, Mi $5.70, Ph $16.80.

Continued use of the capacitance type of fuel-quantity gage in modern aircraft has necessitated a study to determine whether significant fuel characteristics are changing for the older fuel grades and to determine the characteristics of the newer fuel grades.

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The Minneapolis-Honeywell Aeronautical Division has completed such a study. Measurements were made of dielectric constants, densities, and dissipation factors on 144 samples of aviation fuel, grades 91/96, 100/130, 115/145, and JP-4. This report summarizes the results of the investigation and includes a statistical analysis of the data.

It is recommended that the calibration data currently specified by MIL-G-7817 be retained for use in calibrating noncompensated capacitance-type, fuel-gage systems. A new optimum response line and appropriate calibration data are recommended for use in calibrating the compensated systems.

United States Department of the Interior. WADC TR 52-35 Sup 4.

RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT FUELS. Micheal G. Zabetikis, George W. Jones, George S. Scott, Aldo L. Furno. AF 18(600)-151. January 1956. ASTIA Document No. AD 91875. PB 12733954. Order from LC Mi $5.40, Ph $15.30.

A summary of the theory of combustion, explosion and ignition is presented as well as the results of limit-of-flammability, limit-of-ignitibility, limit-of-propagation, ignition energy and ignition temperature tests on current Air Force fuels, fuel components and hydraulic fluids. These data were obtained in air, and carbon dioxide and nitrogen-enriched air at various simulated altitudes by the Bureau of Mines and other laboratories. Most of the experimental results were obtained by the members of the Bureau of Mines Gas Explosions Branch during the period February 1950 to June 1955. The combustibles investigated include the paraffins, aromatics, blends of these, JP-1, JP-3, and JP-4 jet fuels, 100, 130 and 115/145 aviation gasolines, MIL-O-5606 hydraulic fluid and several experimental hydraulic fluids. The data are presented in graphic and tabular form.

WADC TR 55-493.

EVALUATION OF COMPOUNDS FOR USE IN HIGH TEMPERATURE ANTI-SEIZE APPLICATIONS. Gary G. Winters, 2/Lt, Edward Ogletree, R. C. Zurbrigg. August 1956. ASTIA Document No. AD 97213.

A laboratory mechanical evaluation test method has been developed by this Center for evaluating anti-seize compounds for use in high temperature applications such as threaded fasteners. Data obtained from a typical gas turbine engine application have established that the developed test method will qualitatively predict the suitability of anti-seize compounds for use at temperatures up to 1500°F. On the basis of the results of this progress, several commercially available anti-seize compounds are recommended for use at high temperature. A recommendation is also made that a performance specification be written for high temperature anti-seize compounds incorporating the developed mechanical evaluation test as a requirement.

The purpose of this report is to evaluate the performance of a number of products for a specific application. Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

Shell Development Company. WADC TR 53-63 Part 3.

STABILITY OF JET TURBINE FUELS. Harry B. Minor, Alan C. Nixon, Roy E. Thorpe. AF 33(616)-2707. February 1957. ASTIA Document No. AD 118084.

The Emeryville Research Center of Shell Development Company has been investigating the effect of hydrocarbon type, minor constituents, additives and treatment on the storage and high temperature stability of jet turbine fuels.

A heat exchanger test developed here has been used for high temperature stability testing. Although most pure hydrocarbons are more stable in this test than commercial fuels, α-methyl naphthalene is equally unstable. Small amounts of chromatographic polar fractions reduced the high temperature stability of a stable straight-run fraction; olefin and olefin-diolefin fractions were inert. Dispersants are effective in prolonging filter plugging time although the rate of deposit accumulation on the filter is not changed. Silica gel treatment had a beneficial effect on high temperature performance, but not sodium treatment.

The one successful storage inhibitor was again found effective under mild conditions of aging. Metal deactivator is effective in preventing catalytic action by copper-containing steels but is only partially effective against copper surfaces. The deactivator increases the rate of solution of copper by a factor of about five. With dissolved copper, at least 2.0 times the stoichiometric amount of deactivator is required. The final results of the five year desert storage program are discussed briefly with respect to the effect of fuel type and source, oxygen availability and filterability.

Chromatographic separation of catalytically cracked, thermally cracked and straight run gas oils into type components shows that conjugated diolefins, aromatic olefins and "resin fractions," consisting largely of nitrogen bases and phenols, are responsible for instability and lack of anti-oxidant susceptibility under mild aging conditions. Objectionable olefinic constituents are not appreciable in straight run gas oils. They are more prevalent in thermally cracked than in catalytically cracked gas oils.

Shell Development Company. WADC TR 53-63 Pt IV.

STABILITY OF JET TURBINE FUELS Part IV. Thermal and Oxidative Stability. Harry B. Minor, Alan C. Nixon, Roy E. Thorpe. AF 33(616)-2707. February 1957. ASTIA Document No. AD 118085.

The Emeryville Research Center of Shell Development Company has been investigating the effects of hydrocarbon types, minor constituents, additives and treatment on the storage and high temperature stability of jet turbine fuels.

Previously it was shown that conjugated diolefins, conjugated aromatic olefins, alkyl thiophenes, and "resin" fractions were responsible for instability and lack of anti-oxidant susceptibility under mild aging conditions. To provide a better understanding of the adverse nature of the "resins," this fraction has been separated into its basic and acidic components and a neutral fraction consisting in part of an unknown polymeric heterocyclic fraction and partly of aromatic hydrocarbons and thiophenes, similar to the main hydrocarbon fraction. The nitrogen bases were resolved into aniline homologs, and pyridines-quinolines. The nitrogen bases do not affect stability adversely; the natural phenols have a small effect only; but the heterocyclic neutral "resin" fraction is extremely unstable.

The nitrogen picture was completed by isolating and evaluating the pyrroles which, together with the bases, comprise the bulk of the nitrogen compounds. Pyrroles are markedly unstable, forming large amounts of insolubles.

With respect to the high temperature performance of the above components, aromatic olefins in combination with aromatics, phenols, nitrogen bases and pyrroles decrease the stability of an aromatic-free solvent. An anilines-rich fraction had a moderate adverse effect whereas pyridines-quinolines appeared to exert a pronounced stabilising action. Prior aging of jet fuels causes a large decrease in thermal stability which cannot be anticipated from the small increases in gum and peroxide content. Of several additives tested only one offered some benefit. A number of other additives are being examined to offset the effect of storage; a few compounds showed some immediate beneficial action with respect to heat exchanger fouling. The effective keto-imine storage stabilizer had an immediate adverse effect on the thermal stability of 3 of 6 CRC fuels tested although it reduced gum formation in the same 3 fuels (and one other) during storage. In the one case where this compound improved high temperature performance somewhat, there was an adverse effect on the formation of insoluble gum during storage. In other fuels the keto-imine generally has no effect on high temperature stability although it may significantly reduce formation of gum on aging.

A few compounds in addition to the keto-imine and its analogs have shown some favorable effect in reducing gum formation in fuels. The beneficial action of the keto-imine may be related to its ability to deactivate soluble copper. The latter has been found in most jet fuels in amounts ranging from about 0.05 to 0.25 ppm. The conventional commercial metal deactivator has not always been effective in reducing gum formation.

Repeated exposure (recycle) of jet fuels to high temperature conditions (ca 450°F) is more severe than once-through from the standpoint of rate of deposition on both filters and preheaters, but once-through is generally more severe on the rate of filter plugging (ΔP).

WADC TR 55-138.
ACCELERATED STORAGE STABILITY OF AVIATION FUELS. R. W. Sneed, O. M. Ballentine, Capt. J. H. Winterhaulter. March 1957. ASTIA Document No. 118109. PB 131369.

This work is a study of aviation fuels of various base stocks, containing several different additives, and stored under accelerated conditions in two different type containers; the purpose was to obtain data on the deterioration which may be expected during the storage of aviation fuels at desert temperature.

Wide variations were found in the rates of deterioration of the fuels. Phenylene diamine appears to be the best oxidation inhibitor and the C_2 and C_4 alkylate base stock is more stable than the straight run fuel; copper definitely accelerates gum formation.

Shell Development Company. WADC TR 53-63 Part V.
STABILITY OF JET TURBINE FUELS Part V. Effect of Nuclear Radiation. Alan C. Nixon, Roy E. Thorpe. AF 33(616)-2707. April 1957. ASTIA Document No. 118230.

The Emeryville Research Center of Shell Development Company is studying the effects of ionising radiation on jet fuels and their components, particularly with respect to radiation-induced changes in thermal stability. This investigation is progressing in three directions:

1. Evaluation of radiation damage in terms of thermal and oxidative stability.
2. Determination of the chemical changes by spectroscopy and physical separations.
3. Evaluation of the feasibility of "radiation protectors" (e.g., aromatic compounds).

Differences are noted between the damage suffered by various jet fuels, both with regard to chemical changes and stability effects. On the basis of the infrared spectra of the irradiated fuels, three general radiation reactions occur in jet fuels:

1. Formation of carbonyl compounds (in air).
2. Formation of olefins (in both air and nitrogen).
3. Destruction of benzene and naphthalene (particularly in nitrogen).

Irradiation of jet fuels to 1×10^8 roentgens increases soluble gum (as measured by chromatogum, with and without prior aging) to about the same level in either air or nitrogen. The insoluble gum level is not altered appreciably in either case.

Of three typical jet fuels, two (a special kerosene and a JP-5 fuel -- Los Angeles Basin crude) improved in thermal stability at 450°F as a result of irradiation in air to 10^8 roentgens. A JP-4 fuel -- San Joaquin crude -- on the other hand, was affected adversely, as judged by filter-plugging tendencies. Filter-plugging time was reduced by a factor of 2 to 3. A start was made toward relating the thermal stability of these fuels to composition changes.

The results of initial studies of "radiation protectors" indicate that the aromatic compounds investigated thus far do not generally extend sponge-type protection to jet fuels; instead they themselves react in substantial amounts on irradiating, in nitrogen or air, jet fuels containing 2.5 to 20%w of added aromatic compounds.

At the present time the best choice for a radiation-stable jet fuel would appear to be one similar to the special kerosene, RAF-99, referred to above.

Shell Development Company. WADC TR 53-63 Pt VI.
RESEARCH ON DETERMINATION OF THE STABILITY OF JET ENGINE FUELS. Alan C. Nixon, Roy E. Thorpe, Harry B. Minor, Ted R. Lusebrink. AF 33(616)-3888. May 1958. ASTIA Document No. AD 151193.

The Emeryville Research Center of Shell Development Company has continued the investigation of the effects of environment and composition on the thermal and radiation stability of jet fuels. During the past year the major effort has been devoted to the study of radiation damage as a function of dosage and fuel composition. The relation between most properties and log dosage follows an exponential curve in the range 10^7-10^9 r, but the thermal stability undergoes a sinusoidal type of change, the greatest degradation occurring at 10^7 r or below with an improvement over base occurring at about 10^8r, followed again by degradation at higher dosages. An apparent major degrading effect of combined neutron-gamma flux (in SPT) now appears to be mainly due to the gamma flux level and to solution of iron. The latter (and also copper) has a marked degrading effect.

Some antioxidants and dispersants have been found to have a beneficial effect on thermal stability even after storage, but the effect of radiation has not yet been completely determined. It appears that metal deactivators (in a metal-containing fuel) cannot be relied upon to eliminate the adverse effect of the metal on thermal stability.

In view of the degrading effect of aromatics on thermal stability, a completely saturated fuel seems to be the best choice for a fuel to be used in a radiation environment, although there is some evidence that fuels containing a high proportion of C_5 rings should be avoided.

Shell Development Company. WADC TR 59-108. RESEARCH ON THE EVALUATION OF NEUTRON AND GAMMA IRRADIATION EFFECTS OF SIX HYDROCARBON-TYPE FUELS. A. C. Nixon, T. R. Lusebrink. AF 33(616)-6052. September 1959.

Previous work with three fuels indicated that neutrons in a mixed gamma-neutron field had a strikingly deleterious effect on thermal stability. Since the experimental conditions could have contributed to this, recent additional experiments have been done in an effort to recheck the results obtained. Five hydrocarbon fuels were irradiated in the Convair reactor at various levels up to 18^8 rads. Examination of the irradiated fuels confirmed that neutrons were more deleterious to thermal stability than a pure gamma field, although the neutron flux amounted to only 7% of the total and the change in the usual inspection properties was no greater than would be expected from an equal amount of gamma-radiation.

From the behavior of the various fuels which were irradiated, it appears that saturated fuels have the best thermal stability properties under these conditions of irradiation and appear adequate to at least 1.1×10^8 rads neutron-gamma and 3×10^8 rads gamma radiation. However, while the refinery-produced fuels which were examined appear to have adequate radiation resistance, the present state of knowledge does not allow us to choose or specify operational fuel with certainty. It is evident that additional work will have to be done in order to develop adequate fuels and specifications for their procurement. It is recommended that such work be initiated with pure compounds and simple mixtures in order to develop exact characterization of suitable fuels in terms of chemical composition as well as physical inspection properties as related to radiation and thermal stability.

WADC TR 59-139.
A HIGH TEMPERATURE X-RAY DIFFRACTOMETER SPECIMEN MOUNT. Lt William L. Baun. October 1959.

A high temperature diffractometer specimen mount is described. This specimen mount makes possible the examination of materials at temperatures up to 1900°C in high vacuum or inert atmosphere. Diffraction patterns of crystalline materials may be recorded in the scanning range 17° to 122° using this specimen mount. Realignment of the sample at temperature may be accomplished from each point of the three point sample suspension system. A power regulator is described which permits automatic temperature regulation by means of a sensing device that maintains an accurate pre-set temperature.

Bureau of Mines. WADC TR 59-663. FLAMMABILITY CHARACTERISTICS OF HIGH TEMPERATURE HYDROCARBON FUELS. J. M. Kuchta, S. Lambiris, H. E. Perlee, D. R. Halagan, M. G. Zabetakis. DO 33(616)-57-4. February 1960.

Data are presented on the flammability and spontaneous ignition characteristics of various aircraft fuels. Limits of flammability were determined in air at atmospheric pressure and elevated temperatures under essentially static conditions. Spontaneous ignition temperatures and corresponding ignition delay times were determined under static and dynamic flow conditions. In the static tests, an oxidizing atmosphere of air was used at both reduced and elevated pressures. The dynamic tests were conducted at elevated pressures employing various nitrogen-air concentrations. Other physical properties of these fuels are also presented. In addition, data are reported on the spontaneous ignition of fuel vapor-air mixtures contained in cylindrical and spherical vessels which were heated either uniformly or non-uniformly.

Consolidated Electrodynamics Corporation. WADD TR 60-461.
AN ANALYZER FOR MOISTURE AND SOLIDS IN JET FUELS. Michael Czuha, Jr., Kenneth W. Gardiner. AF 33(616)-6588. September 1960. PB 171303. Order from OTS $1.00.

An instrument for continously and simultaneously measuring total water, suspended water, and dissolved water in jet fuels was developed in conjunction with the anti-icing program at WADD. Studies were also made of photometric techniques for solid contamination detection and of the adaptability of a nephelometric method to the moisture analysis equipment to form a unitised contaminants analyzer.

The continuous coulometric analysis of total and dissolved water in JP-4 and RP-1 fuels furnished by WADD was effected over several ranges in a dual analyzer and the suspended water was determined by the difference in the two readings. The instrument response was correlated with Karl Fischer analyses using a polarized electrode end point indicator.

A positive interference with the instrument reading was observed with methyl carbitol in the fuel as an anti-icing additive used in early studies at WADD.

An optical system utilizing forward scattering of light and multiplier phototubes in a ratio measuring circuit was incorporated into the instrument to detect solid contaminants in the range 0-10 ppm. The response of the analyzer was determined by adding test dust to the fuel and correlating with gravimetric determinations using Millipore filters.

Southwest Research Institute. WADD TR 60-908. SERVICE TEST EVALUATION OF FILTERABILITY AND WATER SEPARATION CHARACTERISTICS OF JET FUEL VOLUME I AND II. Robert K. Johnston, J. P. Cuellar, Jr. AF 33(600)-39425. January 1961.

A study was made to determine to what extent fuel corrosion inhibitors interfere with fuel filtration and water separation. Performance was rated on contaminated JP-4 fuel in a single-element filter-separator test facility, using filter housings designed for each of five commercial element types to simulate flow conditions in the corresponding full-scale units. Standard coarse A/C test dust and water were used as contaminants; a brief study was also made using iron oxides.

The corrosion inhibitors interfered with filtration and coalescence, the effects ranging from slight to very severe. The various elements types differed in efficiency and in modes of failure. Fuel aromatics content and gravity, within the JP-4 range, had little effect on performance.

The CRC water separometer, a bench-scale coalescing test apparatus, gave a general correlation with the single-element results. Interaction of additives with fuel components caused severe repeatability problems in early work, but apart from this the separometer repeatability was satisfactory. Preliminary work on a separometer reference fluid indicated that an "odorless solvent" (alkylate) may be suitable.

The Carborundum Company. WADC TR 57-491 Pt I.
SiC-GRAPHITE BODY FOR ELEVATED TEMPERATURE SERVICE. Carl F. Cline. AF 33(616)-3538. October 1957. ASTIA Document No. AD 142022.

A graphite body bonded by silicon carbide has been developed which has physical properties superior to graphite alone. Data are presented fro transverse rupture strength, density, chemical analysis, and tensile strength. One composition has an average transverse rupture strength of 12,000 pounds per square inch at 1500°C as compared to 5,400 pounds per square inch for ATJ graphite.

A dynamic high temperature test was developed which utilized the high current arc as its heat source. Data are presented on the stability of the developed bodies as compared with ATJ graphite when the materials are subjected to a preliminary version of the high temperature arc test.

National Carbon Company. WADC TR 57-602. GRAPHITE-BASED MATERIALS FOR HIGH TEMPERATURE APPLICATIONS. M. Janes. AF 33(616)-3537. February 1958. ASTIA Document No. AD 150962.

Recent aerodynamic developments require a material which will endure under conditions of high heat flux with consequent high surface temperatures and high flow of an oxidizing gas relative to a body of the material. Surface temperature rise may be quite rapid, so that the material must possess resistance to thermal shock. Graphite has outstanding high temperature strength, a high sublimation temperature, and excellent thermal shock resistance in comparison to other agglomerate bodies. However, it is quite reactive with oxygen or other oxidizing gas at high temperatures and high relative gas flow rates.

This report summarizes the results of a first year of a program to compare the oxidation, with accompanying erosion, of graphite prepared with variation in raw materials or processing, and of combinations of graphite with metals, carbides and compounds such as calcium fluoride.

Three different procedures for testing oxidation-erosion resistance, varying in principle and in severity, have been developed and used. Two procedures for evaluating relative thermal shock resistance are described.

Graphite materials prepared by the variations in raw materials and processing so far evaluated, show relatively minor differences in oxidation-erosion rate. Removal of catalytic impurities substantially reduces the rate of oxidation if any portion of the specimen is subjected to relatively low temperature oxidation.

Combinations of graphite with various carbides such as those of boron and titanium together with silicon carbide, with the carbide incorporated throughout the body, have shown considerably improved oxidation resistance in an intermediate temperature range (to about 1700°C). It appears possible that further development will extend the stability of these materials to higher temperatures.

Armour Research Foundation. WADC TR 58-152. STUDY OF THE MECHANISM OF FAILURE OF ROCKET MATERIALS. Yehuda Baskin, Thomas A. Greening. AF 33(616)-3925. August 1958. ASTIA Document No. AD 155804.

Twenty-five different materials were evaluated as nozzle inserts in rocket motors utilizing typical solid propellants. Evaluation of each material consisted of its exposure to the high temperature sonic gas flow of solid propellant engines under conditions of high internal chamber pressure for relatively short periods. Some of the nozzle materials were incorporated in subsequent trials at various combinations of chamber pressure and duration. Unfired and fired nozzles were examined, utilizing X-ray diffraction, microscopic, chemical, metallographic, and other analytical techniques. Chemical changes taking place in the nozzle as a consequence of firing are described. The mechanisms considered responsible for deterioration of the various materials are discussed in detail.

National Carbon Company. WADC TR 58-395. FURTHER STUDIES OF GRAPHITE-BASED MATERIALS FOR HIGH TEMPERATURE APPLICATIONS. M. Janes. AF 33(616)-3537. November 1958. ASTIA Document No. 204216

Recent aerodynamic developments require a material which will endure under conditions of high heat flux, with consequent high surface temperatures and high velocity flow of an oxidizing gas relative to a body of the material. Surface temperature rise may be quite rapid so that the material must possess resistance to thermal shock. Graphite has outstanding high temperature strength, a high sublimation temperature, and excellent thermal shock resistance in comparison to other agglomerate bodies. However, it is quite reactive with oxygen, or other oxidizing gas, at high temperatures and high relative gas flow rates.

This report summarizes the results of a 6-month period of a continuing program to study the behavior of graphite- or carbon-based materials under conditions of exposure to high heat flux with consequent high surface temperatures at high relative gas-flow rates of an oxidizing gas. Materials which showed promise in earlier work previously reported have been more intensively studied and tested at higher relative air-flow rates. Carbon and graphite materials prepared with normal variations in carbon technology, such as the raw materials for filler component and binder, particle sizing, method of forming, etc., continue to show relatively minor differences in oxidation-erosion rate. Materials of promise, even under the relatively more severe conditions of air-flow rate, include graphite coated with pyrolytic carbon, graphite impregnated with AlF_3, and graphite coated with silicon carbide or silicon nitride. Combinations of graphite with carbides, such as boron carbide and silicon carbide, with the carbide incorporated throughout the body, show substantially improved oxidation-erosion resistances at the more severe conditions of high relative air-flow rate. However, comparatively poorer behavior is to be anticipated at surface temperatures above about 1700°C.

Armour Research Foundation. WADC TR 58-503. GRAPHITE TECHNOLOGY. Samuel W. Bradstreet. AF 33(616)-5185. January 1959. ASTIA Document No. 208321. PB 151682.

The two-fold purpose of this investigation has been to correlate the properties of small, multicrystalline graphite specimens with the factors believed to influence them, and to assess the probability that graphite can conform to engineering specifications.

The technique of differential thermal analysis was found applicable to investigating binder pyrolysis. Three promising binder materials were selected for future work: coal-tar pitch, furfuryl alcohol, and phenol-benzaldehyde.

The most illuminating properties of experimental mixtures were found to be: apparent (water immersion) density, dynamic (sonic) modulus, volume electrical resistance, and flexural strength. Measurements of modulus, strength, and thermal expansion have been carried out to temperatures exceeding the creep threshold of graphite, between 2100 and 2250°C.

Solvent mixing techniques for insuring homogeneous distribution of the binder were found to improve reproducibility, in all cases some change in the rheology of the system was observed with time. Maximum density and strength in pitch-bonded coke was achieved after "aging" the mix at 125°C under partial vacuum. Maximum density consistent with good thermal shock resistance was obtained by adding Thermax to the coke flour. With 14 percent by weight of Thermax specimens were made having densities exceeding 1.8 gm/cc with room-temperature strengths of about 2500 psi.

Dynamic flexural moduli were measured for specimens heated and cooled between room temperature and 2650°C. Specimens cut from a block of commercial, impregnated, molded graphite increased 175 percent in flexural strength when heated to 2000°C. Such heating caused a significant permanent change in dimension, modulus, and volume resistance.

It is concluded that small graphite specimens can be made to conform to engineering specifications.

Armour Research Foundation. WADC TR 58-152 Sup 1.
STUDY OF THE MECHANISM OF FAILURE OF ROCKET MATERIALS. Yehuda Baskin, Thomas A. Greening. AF 33(616)-3925. January 1959. ASTIA Document No. AD 207779.

Twenty-five different materials were evaluated as nozzle inserts in rocket motors utilizing typical solid propellants. Evaluation of each material consisted of its exposure to the high temperature sonic gas flow of solid propellant engines under conditions of high internal chamber pressure for relatively short periods. Some of the nozzle materials were incorporated in subsequent trials at various combinations of chamber pressure and duration. Unfired and fired nozzles were examined, utilizing X-ray diffraction, microscopic, chemical, metallographic, and other analytical techniques. Wear parameter data, shadowgraphs, and macrophotographs are presented.

The Carborundum Company. WADC TR 59-10, Part I.
DEVELOPMENT AND EVALUATION OF MATERIALS FOR HIGH TEMPERATURE APPLICATIONS. Charles E. Schulze, Frank A. Saulino, William T. Adams, Gustave E. Maagsen, James C. Andersen, Howard W. Emmons. AF 33(616)-5542. May 1959. ASTIA Document No. AD 214427.

Various preparational techniques have been used to fabricate graphite-based bodies which have potential resistance to high velocity, high temperature air. Materials have been evaluated in a high intensity arc testing facility. Results are reported herein.

At least ten compositions have been developed which are superior to either ACHESP graphite or to a graphite-silicon carbide body on the bases of tests imposed.

Carbon Research Laboratory, University of Buffalo. WADC TR 59-660, Part II.
INVESTIGATION OF ELASTIC AND THERMAL PROPERTIES OF CARBON-BASE BODIES. S. Mrozowski, J. F. Andrew, N. Juul, J. Okada, H. E. Strauss, D. C. Wobschall. AF 33(616)-5186. March 1960. PB 161733. Order from OTS $2.25.

In continuation of the work, four basic types of carbon were prepared, namely from (1) soft filler and soft binder, (2) soft filler and hard binder, (3) hard filler and soft binder, and (4) hard filler and hard binder. Elastic moduli, permanent set, shrinkage and density, electric resistivity and thermal expansion coefficient were investigated in dependence on heat treatment temperature and also the variation of the heat conductivity and electrical resistivity on ambient temperature in the range 1200-3000°C. An appartus for direct determination of the heat diffusivity in the temperature range 100-700°C was set up and reasonably good date for the diffusivity coefficient obtained. Studies of ultrasonic attenuation show that the decay in intensity of transmitted waves is due to: (1) energy losses due to hysteresis effect present for all wavelengths, (2) scatter of waves by pores, observable only when the wavelength becomes shorter than 10 particle diameters. A discussion of the merits of various types of carbons closes the report.

National Carbon Company. WADC TR 59-789.
DEVELOPMENT OF GRAPHITE AND GRAPHITE BASE MULTI-COMPONENT MATERIALS FOR HIGH TEMPERATURE SERVICE. W. W. Losier. AF 33(616)-5563. April 1960.

This report describes graphite-type materials and their evaluation in various test facilities. Arc torches of various characteristics were employed.

Materials found promising in their oxidation resistance in previous contract work were confirmed and subjected to more severe tests with higher sample temperature and greater heat flux. At sample temperatures of 3000°K or above, none of the variations showed ablation as high as 1600 B.t.u./lb.

One material which has been found to show unique behavior is highly oriented graphite deposited by pyrolytic decomposition of a hydrocarbon gas. These pyrolytic graphites have been made and tested in various shapes and sizes. Because of their highly anisotropic conductivity, the pyrolytic coatings have exhibited a marked ability to retard heat penetration into and temperature rise of the substrate material. However, the pyrolytic graphite has shown no conclusive advantage in ablation resistance under the conditions of the tests. Seeming advantages in some cases may turn out to be due to indirect effects such as a different distribution among various heat dissipation mechanisms.

A new method developed for the measurement of reflectance and emissivity using the carbon arc image furnace has given new information on the spectral emissivity of graphite and lampblack at temperatures ranging from 500°K to 3800°K. It has also permitted a more accurate determination of the temperature of the "pyrometric" carbon arc which is widely recognized and used as a high temperature standard.

Armour Research Foundation. WADC TR 59-786.
INVESTIGATION OF GRAPHITE BODIES. S. W. Bradstreet, L. M. Atlas, S. A. Bortz, H. H. Lund, H. H. Nakamura. AF 33(616)-6143. April 1960. PB 161802. Order from OTS $2.50.

The objectives of this work have been to correlate the properties of small multicrystalline graphite specimens with factors known to influence them, and to use this correlation to guide the preparation of strong, nearly isotropic, non-impregnated graphite specimens having reproducible properties.

The techniques of differential thermal analysis, "brittle ring" tensile testing, and dynamic and static flexural loading to temperatures above 5000°F developed as part of this work have been reported at the fourth biennial conference on Carbon.

The effects of fine aggregate packing on density, modulus, and strength are discussed, and these properties are given for experimental graphites. Bodies have now been made with coal tar pitch binders which fail in tension at above 3500 psi at room temperature and above 5000 psi at 2000°C; furfuryl alcohol-bonded bodies with a density of 1.80 gm/cc of nearly equal strength at room temperature exceed 8000 psi in flexural strength at 2250°C.

University of Buffalo. WADC TR 58-360, Part III. INVESTIGATION OF ELASTIC AND THERMAL PROPERTIES OF CARBON BASE BODIES. S. Mrozowski, J. F. Andrew, N. Juul, H. E. Strauss, D. C. Wobschall. AF 33(616)-5186. February 1961. ASTIA Document No. AD 258531. Order from OTS $1.75.

In continuation of the work the elastic properties and permanent set were investigated at room temperature for carbons made of graphitized filler (soft and hard) and for carbons impregnated in the baked or graphitized state in dependence on the heat-treatment temperature. An apparatus for study of deformation at high temperatures (1000-3000°C) was built and calibrated and the first results for the variation of Young's modulus with ambient temperature were reported. Another greatly improved apparatus for studies of heat conductivity at high temperatures was finished and also put into operation. Further studies of corrections to the heat conductivity due to convection by gas across the pores were made and it is shown that such corrections are necessary even in the case of quite dense carbons. Transverse heat diffusivity was investigated in the temperature range 700-2800°C by a transient state method, using a newly constructed apparatus; good fit with the previously found data in the low temperature range is obtained. A general discussion of the aims of the project concludes the report.

Hydraulic and Heat Transfer Fluids

Pennsylvania State College. TR 5561 (R). HYDRAULIC FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. Dr. M. R. Fenske. NOrd 7958(B) (Joint Air Force-Navy). March 1947.

Pennsylvania State College. TR 5655 (R). HYDRAULIC FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. Dr. M. R. Fenske. NOrd 7958(B) (Joint Air Force-Navy). December 1947.

Pennsylvania State College. TR 5756 (R). HYDRAULIC FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. Dr. M. R. Fenske. NOrd 5958(B) (Joint Air Force-Navy). January 1949.

TR 5746 (R). POLYHALO ORGANIC COMPOUNDS AS LESS FLAMMABLE AIRCRAFT HYDRAULIC FLUIDS. James C. Mosteller. January 1949.

Pennsylvania State College. TR 5756, Suppl. 1 (R). HYDRAULIC FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. Dr. M. R. Fenske. NOrd 7958(B) (Joint Air Force-Navy). September 1949.

Purdue Research Foundation. TR 5763, Suppl. 1 (R). CHLORINE AND FLUORINE CONTAINING ORGANIC COMPOUNDS FOR NON-FLAMMABLE MATERIALS. E. T. McBee, et al. W33-038-ac-19024. May 1950.

Pennsylvania State College. TR 6665 (R). DIBASIC ACID ESTERS AND ESTER-TYPE FLUIDS AND LUBRICANTS. Merrell R. Fenske, Ph.D. NOrd 7958(B) (Joint Air Force-Navy). October 1951.

The work performed by the Petroleum Refining Laboratory, Pennsylvania State College, was directed toward the investigation of lubricants and fluids. These studies included oxidation stability, corrosion resistance, low temperature stability, lubricity of fluids and lubricants, thermal stability, shear stability, development of synthetic base stocks, lubricant additives, non-flammable fluids, viscosity index improvers, and fluid volatility.

California Research Corporation. TR 6685 (R). DEVELOPMENT OF HIGH-TEMPERATURE AIRCRAFT HYDRAULIC FLUIDS. Neil W. Furby. AF 33(038)-9831. November 1951.

The requirements for a non-flammable hydraulic fluid possessing favorable viscosity, volatility, and lubricity characteristics led to the investigation of the organo-silicates as possible new base materials to meet this demand. The studies conducted by the California Research Corporation have resulted in the synthesis of a number of siloxanes, disiloxanes, and organo-phosphorus compounds in an effort to develop a fluid that would meet the requirements as outlined in the three phases of their program. Data presented on mechanical test evaluation, viscosity-volatility relationships, viscosity-temperature characteristics, and fluid stability, indicate the potentiality of the organo-silicates as possible aircraft hydraulic fluids.

California Research Corporation. TR 6685, Suppl. 1 (R). DEVELOPMENT OF HIGH-TEMPERATURE AIRCRAFT HYDRAULIC FLUIDS. Neil W. Furby. AF 33(038)-9831. October 1952.

A high temperature silicate base hydraulic fluid capable of operating from -65°F to over 400°F was formulated from a mixed monomer orthosilicate base stock, a silicone viscosity index improver and a basic amine inhibitor to retard oxidation and hydrolysis. Various additives were evaluated for their effect on oxidation, viscosity, hydrolysis, lubricity, and flammability properties of silicate formulations. Further synthesis work was done on silicate and phosphonate esters as new base materials. Bench-scale wear tests and gear and piston type hydraulic pump tests were carried out on experimental hydraulic fluids.

Purdue Research Foundation. TR 52-118 (R). CHLORINE AND FLUORINE CONTAINING COMPOUNDS FOR NON-FLAMMABLE MATERIALS. Ogden R. Pierce, Earl T. McBee. W33-038-ac-19024. March 1953. ASTIA Document No. AD 9152.

For use as possible base stocks for non-inflammable hydraulic fluids or additives thereof, the following classes of halogen containing compounds were prepared: Sulfides,

Sulfones, Alcohols, Esters, Acids, Alkanes, Alkenes, Silanes, Phosphonites, and Phosphonates.

A review of the relationship between molecular structure and physical properties is presented. It was concluded that extension of this work into the field of fluorine-containing compounds is not practicable at this time, with the exception of spectrographic investigations.

A study of the rates of hydrolysis of the ethyl esters of mono-, di-, and trifluoroacetic acid has shown that the rate of hydrolysis increases with an increase in the fluorine substitution alpha to the carbonyl group.

An outline of future work is presented.

California Research Corporation. TR 6685, Suppl. 2.

DEVELOPMENT OF HIGH-TEMPERATURE AIRCRAFT HYDRAULIC FLUIDS. Neil W. Furby. AF 33(038)-9831. September 1953.

Progress for the year 1952 in the development of a high temperature aircraft hydraulic fluid for operation from -65°F to 400°F is described. Compounds synthesized for investigation as base materials included silicates, trisiloxanes, silicon-phosphorus compounds, phosphonates, and miscellaneous compounds. Diethyl and methylphenyl silicones as well as polymethacrylates were investigated as V. I improvers for silicate base fluids. Additives were evaluated for effects on high temperature stability, hydrolytic stability, and antiwear properties. Four-ball wear tests and 3000 psi Vickers and New York Airbrake piston pump tests were conducted on several experimental fluids to determine lubricating properties.

Purdue University. TR 53-462.

CHLORINE-AND FLUORINE-CONTAINING COMPOUNDS FOR NONFLAMMABLE MATERIALS. O. R. Pierce, E. T. McBee. AF 18(600)-128. April 1954. ASTIA Document No. AD 32732. PB 127881. Order from LC, Mi $9.30, Ph $31.80.

Flash point, fire point and autogenous ignition temperature were determined for mixtures of 25 bromine compounds with hydraulic fluid MIL-0-5606. None of the additives raised the autogenous ignition temperature of the hydraulic fluid appreciably. Flash point and fire point were increased by the addition of bromo-form, 1,1,2,2,-tetrabromoethane, 1,3-dibromopropane and carbon tetrabromide.

A series of alkylphosphonosilanes and alkylphosphonoethoxysilanes was prepared from alkylchlorosilanes and appropriate phosphorus compounds. The entire group exhibited good viscosity and resistance to burning but relatively poor hydrolytic stability. The compound $[(n\text{-BuO})_2P(O)]_2SiEt_2$ (code No PS-52) was found to possess superior viscosity properties and good non-flammability.

Fluorine-containing polyethers were obtained from $F_3CCH{-}O{-}CH_2$ and sodium ethoxide. A fraction of the polymer, presumably $H(-OCH(CF_3)-CH_2-)_2OEt$, was unsatisfactory in all respects as hydraulic fluid.

The ester $(CF_3CF_2CF_2CH_2O)_4Si$ was prepared from silicon tetrachloride and heptafluorobutanol. It has a high flash point, fire point and autogenous ignition temperature.

Fluorine-containing esters and acids were prepared as intermediates from polyfluoroaldehydes by use of the Reformatsky reaction with α-bromo esters. The ionization constants of the acids were determined and interpreted in terms of electronic effects. Both saturated and unsaturated esters were converted to alcohols through reduction.

Fluorinated phosphonate esters $RPO(OCH_2CF_3)_2$ were obtained from bis-trifluoroethyl chlorophosphate by action of Grignard reagents or from tris-trifluoroethyl phosphite and alkyllithium compounds and air oxidation of the resultant alkyl phosphonites. The compounds were found to have poor hydrolytic stability.

Perfluoralkyllithium compounds were prepared by lithium-halogen interchange at low temperature and were utilized in addition reactions with carbonyl compounds.

The reaction between ethyl orthosilicate and heptafluoropentylmagnesium bromide was further investigated. The compounds $(C_3F_7CH_2CH_2)_4Si$ and $(C_3F_7CH_2CH_2)_2Si(OEt)_2$ were separated from the products and purified. The latter was polymerized by heating with hydrochloric acid. A distilled fraction of the polymer showed good resistance to burning (high AIT) but very poor viscosity properties.

Fluorine-containing alcohols were prepared as intermediates from fluoroalkylmagnesium halides by reaction with oxygen and paraformaldehyde. By direct oxidation of these primary alcohols the corresponding acids were produced in good yield. The degradation of their silver salts with bromine furnished fluoralkyl bromides. The esters of the fluorinated acids were converted to secondary alcohols by reactions with primary alkylmagnesium halides and isopropylmagnesium bromide.

Standard Oil Company of California. WADC TR 54-191.

THE DEVELOPMENT OF A HIGH TEMPERATURE AIRCRAFT HYDRAULIC FLUID. N. W. Furby, R. O. Bolt, R. L. Peeler, J. M. Stokely. AF 33(038)-9831. December 1954. PB 132108. Order from LC, Mi $5.70, Ph $16.80.

The requirements for a high temperature hydraulic fluid possessing favorable viscosity, volatility, lubricity and thermal stability led to the investigation of silicon containing compounds as possible new base materials to meet this demand. The studies conducted by the California Research Corporation have resulted in the synthesis of a number of siloxanes and disiloxanes in an effort to develop a finished fluid that would meet the requirements set forth in the program. Data are presented on compounds synthesized and on evaluation of fluid blends.

Monsanto Chemical Company. WADC TR 54-532.

HIGH TEMPERATURE HYDRAULIC FLUIDS. Edward S. Blake, James W. Edwards, William C. Hammann. AF 33(616)-2623. March 1955.

An extensive literature search and thermal stability screening of available chemicals were conducted as a basis for planning a research program for the development of a high-temperature (700°F) hydraulic fluid. Esters of organic and inorganic acids offer the best possibilities from the standpoint of ease of synthesis, fluidity and viscosity. Phenolic esters have better thermal stability but poorer hydrolytic and viscosity properties than esters of aliphatic alcohols. Research effort therefore should be directed toward the improvement of the thermal stability of the aliphatic esters and toward the improvement of the viscosity and hydrolytic stability of the aromatic esters. Suggestions for a research program have been made and the thermal stability screening test data are reported for 42 compounds.

WADC TR 55-62.

A SONIC SHEAR METHOD FOR DETERMINATION OF SHEAR BREAKDOWN ON HYDRAULIC FLUIDS AND LUBRICATING OILS. A. J. Gironda, Earl B. Essing, Bernard Rubin. March 1955.

A procedure has been devised which, within short test periods, will evaluate the shear stability of Hydraulic Fluids and Lubricating Oils containing polymeric materials. This procedure utilizes the Raytheon, 10KC, Magnetostrictive Oscillator Model DF-101. The test fluids are subjected to sonic vibration for a specified length of time, after which viscosity measurements are taken in order to determine the extent of molecular decomposition. Data of typical shear-time relationship of several hydraulic fluids are presented.

WADC TR 55-89 Part 1.
PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF EXPERIMENTAL SILICATE BASE HIGH TEMPERATURE HYDRAULIC FLUIDS Part 1, Monsanto Fluid, OS-45. Oliver M. Ballentine, Donald C. Trop, Harry M. Schiefer, 1st Lt., USAF. October 1955.

A physical and chemical evaluation has been conducted by the Wright Air Development Center, Directorate of Research, Materials Laboratory on an experimental high temperature hydraulic fluid, OS-45 developed by the Monsanto Chemical Company. This silicate base fluid was evaluated against proposed requirements for a -65° to 400°F hydraulic fluid for long life piloted aircraft. This fluid conformed to such proposed requirements as spontaneous ignition temperature, pour point, hydrolytic stability at 200°F and foaming tendency. The oxidation and corrosion characteristics of the fluid at 400°F were good in all respects with the exception of a high acid number signifying some oxidation of the oil. The viscosity of OS-45, 1.2 cs at 400°F, failed to meet the desired specification value of 2.5 at 400°F. Additional hydrolytic stability tests at 400°F have shown that the fluid will break down in the presence of very small percentages of water. This instability at high temperatures seems to be one of the major efficiency of this type of fluid. It is apparent from the mock-up and high temperature test data available, that the present hydrolytic stability test at 200°F needs revision. Fluid stability testing above 400°F indicated that OS-45 has marginal resistance to both oxidation and thermal stability, especially when in the presence of metal catalysts. However this fluid is comparable to other silicate fluids evaluated under these requirements.

WADC TR 55-89 Part 2.
PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF EXPERIMENTAL SILICATE-BASE HIGH-TEMPERATURE HYDRAULIC FLUIDS Pt. 2, Hollingshead Fluid, No. 71852-B. O. M. Ballentine, D. C. Trop, H. M. Schiefer, 1/Lt. USAF. November 1955.

A physical and chemical laboratory evaluation has been conducted by the Materials Laboratory, Directorate of Research, Wright Air Development Center, on an experimental high temperature hydraulic fluid, 71852-B, which was developed by the Hollingshead Chemical Corporation. This silicate base fluid was evaluated against proposed requirements for a -65° to 400°F hydraulic fluid for long life piloted aircraft. This fluid conformed to proposed requirements as spontaneous ignition temperature, pour point, hydrolytic stability at 200°F, oxidation-corrosion, foaming, and viscosity-temperature requirements. The fluid however, failed to meet proposed requirements covering rubber compatibility. Insufficient fluid was available to perform the more significant 400°F hydrolytic stability test. This fluid had properties comparable to other fluids evaluated under the procedures contained in this report.

Monsanto Chemical Company. WADC TR 54-532 Part 2.
HIGH TEMPERATURE HYDRAULIC FLUIDS Pt. 2 Development of Base Stock. Edward S. Blake, William C. Hammann, James W. Edwards, Thomas Reichard. AF 33(616)-2623. January 1956.

Fifty compounds have been screened for thermal stability.

Tert-alkylcarbinyl sebacates have been shown to be remarkably more stable (104-122°F) than n-alkyl sebacates. One new tert-alkylcarbinol, 2, 2, 4-trimethyl-1-pentanol, and eight new esters were synthesized. A polyaryl-phosphonate as a V. I. improver for thermally stable trialkylphosphate appeared to degrade on heating. A sufficient number of aromatic ethers were made and tested to reaffirm the high thermal stability of this chemical class and to indicate a high probability that the melting points of the aromatic ethers can be sufficiently lowered, without sacrifice in their thermal properties, to make them promising as base stock for high temperature functional fluids.

WADC TR 56-25.
EVALUATION OF CHLOROPHENYL PHOSPHATES AS POTENTIAL BASE STOCKS FOR HIGH TEMPERATURE HYDRAULIC FLUIDS. Dr. J. C. Dacons, H. M. Schiefer, 1/Lt. May 1956. PB 121446. Order from OTS $0.75.

Three phosphate ester fluids, all containing phenyl and/or chlorophenyl groups have been evaluated for potential high temperature hydraulic fluid application. These fluids are so chosen that they represent three different classes of chlorophenyl phosphate fluids and results obtained form a basis for predicting the success or failure of the chlorophenyl phosphates in general for this application. The fluids were tested extensively for thermal and oxidative stability since these are the areas in which they were particularly controversial. Other tests include viscosity, pour point, flash point, fire point, and foaming tendency determinations. Results indicate good thermal stability in glass apparatus only and poor thermal and oxidation stability particularly at high temperatures in the presence of metals. The flash points, fire points, and foaming tendencies are good, but the viscosity-temperature relationships are poor. Some wear data were obtained but are inconclusive due to the non-availability of equipment for testing at high temperatures. Such equipment is in the process of being developed. Low temperature wear test results indicate that these fluids would probably be poor in lubricity at high temperatures, but may have possibilities as extreme pressure additives.

Denver Research Institute. WADC TR 56-207.
DEVELOPMENT OF "CHAIN TYPE" POLYPHENYL COMPOUNDS FOR USE AS HIGH TEMPERATURE LUBRICANTS AND HYDRAULIC FLUIDS. Josef J. E. Schmidt, John A. Krimmel, John R. Hobaugh. AF 33(616)-2939. October 1956. ASTIA Document No. AD 110420.

A comprehensive literature survey on biphenyl and other "chain type" polyphenyl compounds was conducted and abstracted data stored in a central card file and on IBM punch cards. Based on the collected data and correlations thereof a series of biphenyl and terphenyl compounds were synthesized and tested for thermal stability and other physical properties.

Alkyl derivatives of biphenyl and of m-terphenyl showed considerable thermal stability. It is suggested that, among the other alkyl-m-terphenyl derivatives, compounds with good thermal stability and wide liquid range may be expected.

WADC TR 56-634.
HYDRAULIC FLUIDS FOR 400°F. TEMPERATURE SYSTEMS. H. M. Schiefer, 1/Lt., Bernard Rubin. February 1957. ASTIA Document No. AD 118073.

The properties and some of the performance characteristics of a group of silicate-ester compositions evaluated as -65° to 400°F hydraulic fluids are described.

The silicate-ester fluids have good viscosity-temperature characteristics, low volatility, and adequate thermal stability for long term use at 400°F; pour points and -65°F viscosity are within range for -65°F use. However, these formulations are hydrolytically unstable in the presence of free water, and show lower lubricity in the 300° to 400°F range than the presently used mineral oil fluid (Specification MIL-O-5606) has in the 200°F to 250°F range. However, mock-up hydraulic systems, adequately engineered and designed, and hydraulic pumps have operated for extended periods at temperatures of 400°F.

Specification MIL-H-8446 has been prepared and issued. Producers of silicate-ester fluids have been requested to submit samples for qualification.

Effects of the silicate-ester fluids on elastomers, protective coatings, fabrics, and plastics are reported to aid in engineering selection of fluid resistant materials.

General Electric Company. WADC TR 56-168.
SILICONE FLUID RESEARCH FOR THE DEVELOPMENT OF HIGH TEMPERATURE HYDRAULIC FLUID AND ENGINE OILS. Edgar D. Brown, Norman G. Holdstock, John M. McGuire. AF 33(616)-2899. February 1957. ASTIA Document No. AD 118045.

The evaluation of chlorophenyl siloxane systems showed that this class of silicones showed some improvement in lubricity over previously evaluated dimethyl silicones and phenyl methyl silicones of comparable viscosity-temperature characteristics and offered promise in the development of 700°F hydraulic fluids and high bulk oil temperature turbine engine oils. However, improvements were required in thermal stability, oxidation stability, lubricity, and foaming tendencies. Development effort based on improvements of the chlorophenyl silicone Versilube F-50 (No. 81406) by additives has shown that: (a) Reaction of F-50 with tin compounds, such as stannic chloride, yields a product with greatly improved lubricity at 300°to 700°F temperature range over that of the original silicone. However, viscosity-temperature properties are much poorer; (b) Addition of iron octoate improves the oxidation resistance of F-50 at 500°F to within the contract requirements for turbine oil development; (c) Perfluoro acids and amines greatly reduce the foaming tendency. However, these materials are too volatile within the desired temperature limits for turbine oil use; and, (d) F-50 type chlorophenyl silicones become thermally unstable and undergo rearrangement and degradation in the 600° to 620°F range. None of the materials studied showed any improvement in thermal stability over the initial starting material.

These additive investigations indicate that a -30° to 600°F hydraulic fluid can be prepared from a suitably modified chlorophenyl silicone of Versilube F-50 structure. The basic compound, F-50, has -65° to 550°F potential. Further development of chlorophenyl silicone formulations will be aimed at improving this performance.

WADC TR 55-89 Pt. IV.
PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF EXPERIMENTAL SILICATE BASE HIGH TEMPERATURE HYDRAULIC FLUIDS Part IV, MLO 8200. H. M. Schiefer, 1/Lt., R. J. Benzing. March 1957. ASTIA Document No. AD 118123. PB 131042. Order from OTS $0.75.

A physical and chemical evaluation has been conducted by the Materials Laboratory on an experimental high temperature hydraulic fluid, MLO 8200, which has a disiloxane base fluid. It was evaluated against the requirements of Specification MIL-H-8446 (USAF) and for higher temperature applications to 550°F. It passes all the requirements of MIL-H-8446 with the exception of rubber swell.

Some additional tests were performed on this fluid which indicated that, as for other silicate fluids tested, the hydrolytic stability at 400°F seems to be one of the major deficiencies of this type of fluid. It is apparent from mock-up and 400°F laboratory hydrolytic stability tests that the present hydrolytic stability test at 200°F needs revision. Some wear data were obtained with this fluid at 167° and 400°F which indicated that the fluid should have sufficient lubricity to lubricate high temperature pumps. Some minimum spontaneous ignition temperatures obtained with the fluid when sprayed from varying pressure levels into a glass vessel at atmospheric pressure showed that the ignition temperature of this fluid decreases to approximately 510°F at high pressures. This phenomenon is also present in other silicate base fluids tested under these conditions.

Monsanto Chemical Company. WADC TR 54-532 Pt III.
HIGH-TEMPERATURE HYDRAULIC FLUIDS Part III. Development of Aromatic Ethers as Base Stock. Edward S. Blake, James W. Edwards, William C. Hammann, Thomas Reichard. AF 33(616)-2623. April 1957. ASTIA Document No. AD 118179.

Over fifty alkyl-substituted and unsubstituted aromatic ethers have been synthesized, many of which are new compounds. The melting point, pour point, vapor pressure, viscosity, and thermal stability of most of these ethers have been measured from which correlations have been made by which the boiling point, 210°F viscosity and 210-400°F ASTM slope, and thermal stability of a new ether can be predicted. From the data at hand it should be possible to synthesize aromatic ethers stable to 700°F, liquid at 0°F, viscosity $<$ 550 cs at 32°F and with a vapor pressure $<$ 600 mm/700°F.

Denver Research Institute. WADC TR 56-207 Pt 2.
DEVELOPMENT OF "CHAIN TYPE" POLYPHENYL COMPOUNDS FOR USE AS HIGH TEMPERATURE LUBRICANTS AND HYDRAULIC FLUIDS. Josef J. E. Schmidt, John A. Krimmel. AF 33(616)-2939. May 1957. ASTIA Document No. AD 118295.

This report describes the synthesis of alkyl biphenyl and terphenyl derivatives and their intermediates. These compounds have been synthesized in the course of investigations for the development of thermally-stable fluids. Modifications for the synthesis of isopropyl-m-terphenyl have been introduced for the achievement of higher yields.

In the terphenyl series the polyalkylation of m-terphenyl has been investigated and isomerization of m-terphenyls attempted.

Thermal stability tests on isopropyl-m-terphenyl and amylisopropyl-m-terphenyl have been carried out by heating over a longer period of time at elevated temperatures. These tests indicated that among m-terphenyls substituted in the 4 position, the isopropyl derivative is more stable than the corresponding amyl derivative.

Bureau of Mines. WADC TR 57-151.
RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT HYDRAULIC FLUIDS. Michael G. Zabetakis, Aldo L. Furno, Joseph J. Miller, Jr. AF 18(600)-151. May 1957. ASTIA Document No. AD 130764. PB 131143.

The results of minimum spontaneous ignition temperature tests conducted on seven hydraulic fluids while in contact with seven surfaces found in aircraft under conditions likely to be encountered in practice are presented here. These tests were conducted by members of the Branch of Gas Explosions, Division of Explosives Technology, U. S. Bureau of Mines between 1 November 1955 and 31 October 1956. Hydraulic fluids Esso Univis J-43, conforming to specification MIL-O-5606, MLO 53-446 (General Electric GE 81406), MLO 54-540 (Monsanto OS-45), MLO 54-581, MLO 54-645 (85% Oronite 8200 + 15% Plexol), MLO 54-856 (Hollingshead 72073C), and MLO 8200 (Oronite 8200) were tested while in contact with heated aluminum, beryllium-copper, copper, magnesium, pyrex glass, stainless steel and titanium surfaces. The effects of both test chamber pressure and injection pressure variations were investigated.

WADC TR 57-266, Pt I.
EFFECTS OF NUCLEAR RADIATION ON ORGANIC FLUIDS Part I. Gamma Radiation Stability of Certain Mineral Oils and Diester Fluids. William L. R. Rice, 1/Lt, James H. Way, 1/Lt. June 1957. ASTIA Document No. AD 130807. PB 131222.

A study was made of the stability to gamma radiation of certain refined petroleum oils and diester fluids. Testing of the irradiated fluids by means of lubricant evaluation procedures, such as viscosity, flash point, and oxidation-corrosion resistance, indicated that for the tests conducted, the mineral oils of the type studied should have general resistance to gamma radiation up to a dosage of about 1×10^8 roentgens. The diester fluid di-2-ethylhexyl sebacate (Plexol 201) had very poor stability to gamma radiation over the same range. Addition of 0.5% phenothiazine improved the radiation resistance of this base fluid, except for the loss of oxidative stability experienced at dosage levels of 1×10^7 roentgens and lower.

Examination was made of the test data for the irradiated fluids to determine if any property changes followed the radiation exposure history. This was to discover if either the mineral oil or diester type fluids could be used as secondary reference standards for radiation calibration of non-uniform geometries exposed to nuclear radiation, such as bearing assemblies or circulating fluid loops. The changes offering the most promise were the increase in neutralisation number of the diester fluid and the gas evolution of the mineral oils.

California Research Corporation. WADC TR 57-119.
DEVELOPMENT OF A HYDROLYTICALLY STABLE HIGH TEMPERATURE HYDRAULIC FLUID. Robert L. Peeler, Steve A. Kovacich. AF 33(616)-3476. July 1957. ASTIA Document No. AD 130915. PB 131380.

This report covers the first year's work on a contract to improve the hydrolytic stability of silicate based hydraulic fluids operating in the -65°F to 400°F temperature range.

A literature search was made of the mechanism and factors affecting the rate of hydrolysis of alkoxysilanes. Steric effects were found to be the most important factor.

Silanes, disiloxanes, trisiloxanes, and miscellaneous silicates, having primary, secondary, and tertiary alkoxy, alkyl, and phenyl groups, were synthesized. These compounds were evaluated for hydrolytic stability at 400°F. Of the compounds tested to date, silanes with two tertiary butoxy and two secondary alkoxy groups have the greatest hydrolytic stability. Di(tert-butoxy)-di(5-ethyl-2-nonoxy) silane and di(tert-butoxy)di(2-octoxy)silane were the two most stable compounds prepared.

Disloxanes containing secondary alkoxy and two tertiary butoxy groups have not shown stability comparable to the silanes.

Two carboxylic acid esters and four silicones were evaluated for hydrolytic stability at 400°F for reference purposes. Contrary to the good hydrolytic stability shown by these synthetic materials at 200°F, they showed considerable deterioration at 400°F. Thus, the best silanes prepared in this program are more stable in this test than other types of synthetic base materials usually assumed to have satisfactory stability.

A small-scale, 400°F hydrolytic stability test method was developed which shows good correlation with the standard bomb method. Diesters, mono-esters, and hydrocarbons have been found which are more effective swelling agents for Neoprene than di(2-ethylhexyl) sebacate. Dimethyl silicones were found to be the most effective viscosity index improvers of those tested, although other high viscosity silicones, polymethacrylates, and polyvinyl ethers were also usable. With di(tert-butoxy) di(2-octoxy)silane, a fluid with 2500 cs viscosity at -65°F and over 1.0 cs at 400°F can probably be prepared. Oxidation tests at 400°F indicated the need for better inhibitors. Four-Ball Wear properties of the new hydrolytically stable base stocks were equivalent to the currently used hexa(2-ethylbutoxy)disiloxane.

WADC TR 55-89 Pt III.
PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF EXPERIMENTAL SILICATE BASE HIGH TEMPERATURE HYDRAULIC FLUIDS. H. M. Schiefer, 1/Lt. July 1957. ASTIA Document No. AD 97101.

A physical and chemical laboratory evaluation has been conducted by the Materials Laboratory on an experimental high temperature hydraulic fluid, MLO 54-645, blended at Wright Air Development Center in such a manner to pass the MIL-H-8446 (USAF) Specification rubber swell test. This silicate-diester base fluid was evaluated primarily against the requirements outlined in the above specification for a -65° to 400°F (200-500 hours) non petroleum base hydraulic fluid. This fluid passes all specification tests with the exception of the oxidation test at 400°F in which the fluid gave a marginal neutralisation number increase and viscosity decrease.

Some additional tests were performed on this fluid, which indicated that, as for other silicate fluids tested, the hydrolytic stability at 400°F seems to be one of the major deficiencies of this type of fluid. It is apparent from mock-up and 400°F laboratory hydrolytic stability tests, that the present hydrolytic stability test at 200°F needs revision. Some wear data were obtained with this fluid at 167° and 400°F which indicated that the fluid should have sufficient lubricity to lubricate high temperature pumps. Some minimum spontaneous ignition temperatures obtained with the fluid when sprayed from varying pressure levels into a glass vessel at atmospheric pressure, showed that the ignition temperature of this fluid decreases to approximately 510°F at high pressures. This phenomenon is also present in other silicate base fluids tested under these conditions.

WADC TR 55-89 Pt V.
PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF SILICATE BASE HIGH TEMPERATURE HYDRAULIC FLUIDS Part V. Room Temperature Hydrolysis Studies. George Baum. July 1957. ASTIA Document No. AD 118341.

Silicate-esters are being considered for use in 400°F hydraulic systems. Previous study had established evidence of the hydrolytic instability of the silicate-ester formulations.

The behavior of four silicate-ester base formulations toward extended storage with varying quantities of water was studied. The formulations presented varying degrees of hydrolytic stability. Solids formation was encountered with all formulations. However, the disiloxane formulation (MLO 8200) produced significantly lower quantities of solids than the other three formulations. Addition of a diester to this disiloxane formulation resulted in a large increase in the amount of solids formed. The solids formed were not a linear function of water present. Instead, maximum solids occurred at intermediate water concentrations.

WADC TR 57-167.
HIGH TEMPERATURE HYDRAULIC FLUID DEVELOPMENT STATUS AND ENGINEERING DATA. George Baum, Robert J. Benzing. August 1957. ASTIA Document No. AD 131009. PB 131512.

A review of the status of extreme high temperature hydraulic fluid development is presented. Progress in research and development for fluids with a high temperature capability of 550°F to 700°F and a low temperature capability of 0°F to -65°F is described in detail. The fluids within this temperature range and described in this report fall into the following classes of compounds; Organosilicates, disiloxanes, silicones, mineral oils and silanes. The available data on properties of these research fluids, important to hydraulic system design are reported. These data include the following; viscosity-temperature, density-temperature, thermal stability, data for heat transfer calculations, compressibility, vapor pressure, and flammability measurements.

The large amount of design data on -65°F to 400°F type fulids obtained by industry and the military services is compiled and reported. This includes both previously published and unpublished data.

Sundstrand Aviation Division. WADC TR 57-2.
HIGH TEMPERATURE FLUID EVALUATION TEST STAND. Donald B. Reinke. AF 33(616)-3126. September 1957. ASTIA Document No. AD 131080.

A hydraulic fluid test stand with the capability of evaluating fluids at temperatures up to 700°F and pressures up to 3000 psi has been constructed. A survey of the industry showed several areas in which reliable aircraft type components were not available, and in such instances the best available non-aircraft components were adapted to complete the test stand.

This report describes the fluid evaluation parameters, the finalized test procedures to be used for fluid analysis, the selection of components, and the results of initial check-out tests. Evaluation of hydraulic fluids at the temperature level for which this stand is built represents a potential fire hasard. Safety and automatic control provisions are noted.

WADC TR 57-573.
GAMMA RADIATION STABILITY OF OS-45 AND OS-45-1 HYDRAULIC FLUIDS. William L. R. Rice, 1/Lt. December 1957. ASTIA Document No. AD 142262. PB 131661.

The hydraulic fluids OS-45 and OS-45-1 were exposed to gamma radiation over the range 4.36×10^8 ergs/gm C to 8.71×10^{10} ergs/gm C. Changes in the physical and chemical characteristics of the fluids were determined following the static irradiations. It was observed that major changes took place in most of the properties at the higher exposure levels. Relationships were derived between the change in certain properties and the gamma dosage. It was concluded that although the effects of gamma radiation on the fluids indicated possible difficulty in their use in a radiation field, dynamic in-source testing in a mock-up in their use in a radiation field, dynamic in-source testing in a mock-up hydraulic system would be necessary to establish their use limitations.

Monsanto Chemical Company. WADC TR 57-437.
HIGH-TEMPERATURE BASE STOCK FLUIDS. Edward S. Blake, James W. Edwards, William C. Hammann, Thomas E. Reichard. AF 33(616)-3819. December 1957. ASTIA Document No. AD 142188.

A limited study was made on the variables involved in the production of a quantity of bromine-free non-crystallizable mixed bis-(methylphenoxy)benzene, by the Ullmann reaction.

Thermal stability and other physical data are reported on 61 compounds which include seven classes not previously reported, namely amides, sulfones, aromatic amines, ketones, silicon fluorides, triaryl derivatives of Group V-B elements below nitrogen and ferrocenes. Twenty chemical classes are compared in thermal stability, and the useful life for eight potential base stocks is estimated.

The limits of error for the WADC TR 55-449 oxidation and corrosion test were determined using esters without antioxidants as test fluids. This test was used to determine the oxidation susceptibility of certain compounds and substituent groups in aromatic ethers. An attempt was made with some success to adapt a modified Warburg apparatus for simplified oxidation studies.

Some eutectic and crystallization studies on various aromatic ethers are reported. Numerous syntheses were made.

General Electric Company. WADC TR 57-294.
RESEARCH ON LIQUID METALS AS POWER TRANSMISSION FLUIDS. Richard H. Blackmer. AF 33(616)-3698. February 1958. ASTIA Document No. AD 151001. PB 131743.

The eutectic alloy of sodium (23 weight-percent) and potassium (77 weight-percent), known as NaK 77, has been determined from a technical survey of liquid metals and salts as the most feasible liquid known for 10°F to 1000°F hydraulic system applications. A single cylinder test pump in an inert atmosphere glove-box has pumped NaK-77 up to 3600 psi at 100°F and up to 2000 psi at 1000°F. A total of about 100,000 cycles at 1 cycle per second and an average pressure of 1500 psi have been accumulated. Results of literature survey, consultation, and laboratory tests are included in this report.

General Electric Company. WADC TR 56-168 Pt II.
SILICONE FLUID RESEARCH FOR THE DEVELOPMENT OF HIGH TEMPERATURE HYDRAULIC FLUID AND ENGINE OILS. Edgar D. Brown, Jr., Norman G. Heidstock. AF 33(616)-2899. February 1958. ASTIA Document No. AD 150988. PB 131926. Order from OTS $3.00.

Continued study of the chlorophenyl silicone formulation has resulted in development of an additive which greatly improves the lubricity of the base fluid (F-50)

without impairing other desirable features such as viscosity-temperature characteristics and oxidation stability below 500°F. This additive is a straight chain linked tin-carbon-silicon-oxygen polymer and is soluble in the base fluid at all temperatures down to -65°F. The tin polymer-silicone formulation has been designated silicone fluid 81717 and will allow for prolonged use in the -65°F to 575°F temperature range. Under appropriate conditions of high pressure and wet atmosphere, hydraulic fluid use in the 650°F-700°F range is possible.

Silicone fluids undergo thermal rearrangement above 600°F which causes large changes in viscosity properties. However, a change in structure to silicone carbon linkage has yielded fluids with excellent 700°F thermal stability. Further syntheses and evaluation study is required to produce fluids of the desired lubricity level.

The fluid with the best over-all oxidation stability is still the base-stock, F-50, with iron octoate as oxidation inhibitor. This is now available as silicone fluid 81644.

WADC TR 57-168.
LABORATORY EVALUATION OF SILANE FLUIDS AS POTENTIAL BASE STOCKS FOR HYDRAULIC FLUIDS AND LUBRICANTS. Harold W. Adams, George Baum. March 1958. ASTIA Document No. AD 142141. PB 131889.

A new class of potential base stocks for high temperature turbojet engine oils and hydraulic fluids has been developed at this laboratory. The physical and chemical properties of the alkyl and alkylaryl silanes show them to possess good thermal stability up to 650°F, good lubricity and marginal oxidative stability at 500°F.

An overall assessment of the properties of the silanes evaluated indicates that they are promising base materials for a 0 to 500°F engine oil or a 0 to 700°F hydraulic fluid.

Olin Mathieson Chemical Corporation. WADC TR 57-581.
RESEARCH AND DEVELOPMENT ON THE SYNTHESIS OF TETRAALKYL SILANES. Edward E. Harris, Frank J. Milnes, Robert J. Rayner, Roman Dombachewsky. AF 33(616)-3430. March 1958. ASTIA Document No. AD 151074.

One-gallon samples of nine tetraalkyl silanes and one diaryl dialkyl silane have been synthesized for testing as high-temperature lubricants. The diaryl dialkyl silane and three of the tetraalkyl silanes have been produced in five-gallon quantity. Finally, fifteen-gallon quantities of diphenyl di-n-octyl silane and di-n-octyl silane have been produced. A Grignard process has been used for all syntheses, both of intermediates and final products.

Denver Research Institute. WADC TR 56-207 Pt III.
"CHAIN TYPE" POLYPHENYL AND POLYNUCLEAR AROMATIC COMPOUNDS AS BASE MATERIALS FOR HIGH TEMPERATURE STABLE AND RADIATION RESISTANT LUBRICANTS AND HYDRAULIC FLUIDS. Josef J. E. Schmidt, John A. Krimmel, Thomas J. Farrell, Jr. AF 33(616)-2939. April 1958. ASTIA Document No. AD 151166.

This report describes the preparation and investigation of alkyl-biphenyl and alkylterphenyl derivatives and their intermediates. In particular, homologous series of 4' -alkyl-m-terphenyl and 3-alkyl-m-terphenyl compounds have been prepared in connection with a closer investigation of the correlations between chemical structure of these classes of compounds and their physical properties, thermal stability, and their feasibility as thermally stable and radiation resistant fluids.

Two half-gallon samples of the more promising fluids, 4' -n-nonyl-m-terphenyl and 3-n-heptyl-m-terphenyl, have been prepared for WADC's Materials Laboratory under contractural terms for the purpose of additional testing.

Midwest Research Institute. WADC TR 58-84.
DEVELOPMENT OF HIGH-TEMPERATURE STABLE LIQUID UREAS AND AMIDES. Cecil C. Chappelow, Jr., Robert N. Clark. AF 33(616)-5129. May 1958. ASTIA Document No. AD 151191.

A program for the development of thermally stable liquid ureas was undertaken. This program consisted of the syntheses and evaluation of new compounds which contain only carbon groups, nitrogen atoms and organic radicals. The first class of compounds to be investigated has been the tetra-substituted ureas.

During the syntheses program, a total of 26 tetra-substituted ureas were prepared, 23 of which are new compositions of matter not reported in the literature. These compounds are representative of the following types of tetra-substituted ureas: tetraalkylureas, dialkyldiarylureas, tetraalkarylureas, diaklaryldiarylureas, N-(substituted phenyl)triphenylureas and tetraarylureas.

During the evaluation program, primary emphasis was placed upon thermal stability. As a result of this work, it was found that the basic urea structure was very stable. Also, it was found that the following substituent groups were thermally stable: diphenylene, phenyl, 4-methylphenyl, 4-biphenylyl, 4-methoxyphenyl, 2,4-dimethylphenyl, 1- and 2-naphthyl, n-octadecyl, methyl, 4-phenoxyphenyl, n-butyl.

On the basis of preliminary experimental data, tetra-substituted ureas as a class of compounds show promise as base materials for high-temperature hydraulic fluid and lubricant applications.

Bureau of Mines. WADC TR 57-151 Suppl. 1.
RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT HYDRAULIC FLUIDS. Michael G. Zabetakis, Agnes C. Imhof, Frank W. Lang. DO 33(616)-57-4. May 1958. ASTIA Document No. AD 155510. PB 131193s. Order from OTS $1.00.

The results of spontaneous ignition temperature tests conducted on JP-4 and seven hydraulic fluids in air while in contact with seven surfaces found in aircraft are presented. Tests were conducted at reduced and elevated pressures using both low and high velocity injection techniques. They were conducted by members of the Branch of Gas Explosions, Division of Explosives Technology, U. S. Bureau of Mines between 1 November 1956 and 31 October 1957. Hydraulic fluids Esso Univis J-43 (Spec. MIL-O-5606), MLO 53-446 (General Electric GE 81406), MLO 54-540 (Monsanto OS-45), MLO 54-581, MLO 54-645 (85% Oronite 8200/15% Plexol), MLO 54-856 (Hollingshead 72073C) and MLO 8200 (Oronite 8200) were tested while in contact with heated aluminum, beryllium-copper, copper, magnesium, pyrex glass, stainless steel and titanium surfaces. In addition, the flash and fire points of these fluids were determined in a standard ASTM apparatus.

California Research Corporation. WADC TR 57-119 Pt II.
DEVELOPMENT OF A HYDROLYTICALLY STABLE HIGH TEMPERATURE HYDRAULIC FLUID. Robert L. Peeler,

Steve A. Kovacich. AF 33(616)-3476. July 1958. ASTIA Document No. AD 155712.

This report covers the second year's work on a contract to improve the hydrolytic stability of silicate ester hydraulic fluids for operation in the -65°F to 400°F temperature range.

Significantly improved hydrolytic stability was found for the following classes of compounds: (1) tetraalkoxysilanes containing two or three tert-butoxy groups; (2) alkyltrialkoxysilanes containing alkyl groups higher than methyl and one tertiary alkoxy group; (3) hexaalkoxydisiloxanes containing four tertiary alkoxy groups; and (4) dialkyltetraalkoxydisiloxanes containing two tertiary alkoxy groups with the alkyl groups preferably ethyl or higher. Compounds which hydrolyzed at 400°F but which did not form significant quantities of insolubles were (1) alkyltrialkoxysilanes containing ethyl or higher alkyl groups and (2) dialkyltetraalkoxydisiloxanes containing ethyl or higher alkyl groups. Glycol silicates, trisiloxanes, and disilylethane derivatives did not show the required hydrolytic stability. Considering all known properties of these compounds, 1,3-diethyl-1, 1,3,3-tetra(2-ethylhexoxy) disiloxane was chosen as the best available base stock for the final hydraulic fluid.

Rate studies with H_2O^{18} showed that the siloxy groups were more easily hydrolyzed than the corresponding alkoxy groups, indicating a limitation of the polysiloxane structure.

Formulation work on the best disiloxane base stock let to a choice of low viscosity methyl phenyl silicone as V. I. improver and isodecyl pelargonate as rubber plasticizer. The new formulations tested greatly exceeded 8515 fluid in hydrolytic stability and had better shear stability. Oxidation stability was poorer, indicating need for further formulation work on oxidation inhibitors. Other properties tested were comparable to 8515 fluid.

Armour Research Foundation. WADC TR 58-171 Pt I.
DEVELOPMENT OF HIGH TEMPERATURE OXIDATION-CORROSION INHIBITORS TO IMPROVE STABILITY OF HIGH TEMPERATURE HYDRAULIC FLUIDS AND LUBRICANTS. Robert Brunier. AF 33(616)-5101. September 1958. ASTIA Document No. AD 155883.

The objective of this research program was to synthesize and evaluate selected organic compounds as high temperature oxidation-corrosion inhibitors in mineral oils, synthetic ester lubricants, and silicone and silane fluids. As a result of this and continued studies, specific inhibitors will be recommended for use.

During this 12-month period, known inhibitors were appraised and rated. On the basis of this appraisal, organic structures and functional groups effective in corrosion inhibition were determined and organic molecules incorporating these structures and groups designed to overcome the limitations found in known inhibitors. The synthesis of the selected compounds was started.

Simultaneously, outside sources were checked for compounds not yet tested as oxidation-corrosion inhibitors, and some of these compounds were selected for screening. These selections were made on the basis of structures related to those found to be effective by the appraisal program and also structures with potential use as inhibitors.

The compounds were evaluated by a micro oxidation-corrosion test developed at Wright Air Development Center. The testing was directed mostly toward evaluating additives in silane and ester base fluids. Compounds on hand were first submitted to a screening test, followed by specification testing of the most promising compositions.

Some of the compositions evaluated were very active in the silane fluids, and the specifications for a 500-700°F lubricant can probably be filled by such mixtures. Specifically, a mixture of 1,10-diaminodecane and isonicotinic acid thioamide and a mixture of 1,10-diaminodecane, isonicotinic acid thioamide, and triphenylarsine were the most effective inhibitors in the silane fluid. No composition tested in the ester was more active than the ester alone, and more work will have to be done to stabilize this lubricant for use above 500°F.

Stanford Research Institute. WADC TR 58-335 Pt I.
SYNTHESIS AND EVALUATION OF HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC HYDRAULIC FLUIDS AND LUBRICANTS. R. M. Silverstein. AF 33(616)-5276. January 1959. ASTIA Document No. AD 207525.

During the period covered by the current report, candidate antioxidants have been screened in the following lubricating fluids: F-50 silicone fluid, paraffinic mineral oil MLO 57-574, silane fluid MLO 57-461, silane fluid MLO 57-628, and naphthenic mineral oil MLO 57-573.

A number of additives at 0.2% concentration were found to be effective in retarding high temperature oxidative degradation (i.e. gelation) of F-50 silicone oil. Condensed aromatic ring structures containing three or more rings were effective as a class.

Several of the effective additives were soluble enough in F-50 silicone fluid at low temperatures to warrent their consideration for practical use. Fluoranthene at 0.2% concentration, for example, did not come out of solution at -65°F. Solubility studies are still in progress. A mechanism study was initiated to determine how condensed aromatic ring structures inhibit gelation of F-50 silicone fluid.

Following the lead uncovered by the effectiveness of N-phenylferrocene-carboxamide, a number of ferrocene derivatives were prepared and evaluated. None of these compounds showed pronounced antioxidant activity.

None of the additives evaluated to date showed any antioxidant activity in the silane fluids or in the mineral oils.

Bureau of Mines. WADC TR 57-151 Pt II.
RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT HYDRAULIC FLUIDS Part II. Explosion Hazards of Aircraft Hydraulic Fluids and Fuels. Michael G. Zabetakis, George S. Scott, Agnes C. Imhof, Sotirois Lambiris. DO (33-616)-57-4. March 1959. ASTIA Document No. AD 210715.

The results of limit of flammability and spontaneous ignition temperature tests conducted on six high density fuels and JP-6 are presented. The former tests were conducted at atmospheric pressure and elevated temperatures and the latter at reduced and elevated pressures with the fuels in contact with pyrex and stainless steel surfaces in air and oxygen. In addition, spontaneous ignition temperature tests were conducted at atmospheric pressure on seven hydraulic fluids in oxygen and oxygen-enriched air with the fluids in contact with a pyrex surface. All tests were conducted by members of the Branch of Gas Explosions, Division of Explosives Technology, U. S. Bureau of Mines between 10 December 1957 and 30 June 1958. Monoisopropylbicyclohexyl (HTF-27), monoisopropylbiphenyl (HTF-28), tetralin (HTF-30), bicyclohexyl (HTF-31), decalin (HTF-32), and methylcyclopentadiene (HTF-33), and hydraulic fluids Esso Univis J-43 (Spec. MIL-H-5606A), MLO 59-446 (General Electric GE 81406), MLO 54-540 (Monsanto OS-45), MLO 54-881, MLO 54-645 (85% Oronite 8200 + 15% Phenol), MLO 54-896 (Hollingshead 72073C) and MLO 8200 (Oronite 8200) were tested.

WADC TR 58-213.
SILICONE FLUIDS AS HIGH TEMPERATURE JET ENGINE OILS AND HYDRAULIC FLUIDS. George Baum, Harold W. Adams. April 1959.

A physical and chemical laboratory study of several silicone fluids has been conducted. These fluids were investigated for possible use as high temperature jet engine lubricants and/or hydraulic fluids. Properties of the silicones are compared with the requirements of Specification MIL-L-9236, the target high temperature jet engine oil specification, and with general high temperature hydraulic fluid requirements.

Significant improvement in the lubircating properties of silicone fluids has been accomplished by the incorporation of an organo-tin silicon polymer in General Electric Company's Versilube F-50.

Both a methyl chlorophenyl and a methyl phenyl silicone are being considered for jet engine lubricants, based on MIL-L-9236 requirements. The use of silicone fluids as gas turbine lubricants depends primarily upon solving the problem associated with carbon seals. The problem presents itself in the form of deposits in carbon seal areas.

Silicone fluid formulations based on General Electric's Versilube F-50 possess many properties required for a wide temperature range hydraulic fluid. The choice of a high temperature hydraulic fluid will be dictated by comparative properties such as thermal stability, oxidative stability, and anti-wear characteristics as required by the system.

General Electric Company. WADC TR 57-294 Part II.
RESEARCH ON LIQUID METALS AS POWER TRANSMISSION FLUIDS. Robert C. Kumpitsch. AF 33(616)-3698. May 1959. ASTIA Document No. AD 214694. PB 151876. Order from OTS $3.00.

The behavior of liquid metal NaK-77 (autectic alloy of sodium 23% by weight and potassium 77% by weight) as a hydraulic fluid was observed over a temperature range of 80°F to 1000°F and at cyclic pressures of 15 psia to 3000 psia. An evaluation was made of several bearing and construction materials and several types of static seals to determine their effectiveness, in NaK-77 under such conditions of operation. Capillary and orifice flow characteristics were obtained with this fluid and a correlation was made of these test results with the calculated results.

A single piston test pump operating in an inert atmosphere "glove box" has pumped NaK-77 to pressures of 3000 psi at 1000°F and delivered a cyclic flow rate of 0.02 G.P.M. A total of 368 hours of NaK pumping have been accrued, two and one-half hours of which were at temperature and pressure. Various material combinations, clearances, orifice characteristics and static seals were successfully evaluated.

Materials compatibility tests were conducted with friction and wear test equipment in NaK-77 at 1000°F. A total of 19 tests were conducted, nine for the calibration of the test equipment, and ten tests to evaluate the five most promising material combinations.

Test results essentially confirmed previous work conducted by others in which carbide materials appear to be the most compatible combinations in NaK-77 at 1000°F ambients.

A high pressure (3000 psi) and temperature (1000°F) test loop was also designed to circulate NaK-77 at a flow rate of 1 G.P.M. for purposes of evaluating the feasibility of using liquid metals as a hydraulic fluid for power transmission and control systems applications. The design of this test loop also included the design of a 1000°F, 3000 psi 1 G.P.M. continous flow, staged gear pump as a prime mover.

General Electric Company. WADC TR 56-168 Pt III.
SILICONE FLUID RESEARCH FOR THE DEVELOPMENT OF HIGH TEMPERATURE HYDRAULIC FLUIDS AND ENGINE OILS. Edgar D. Brown, Jr., Norman G. Holdstock, J Merle Nielsen. AF 33(616)-5118. May 1959. ASTIA Document No. AD 213606.

The past year's work has been concentrated on both thermally stable fluids and improved oxidative stability. The most promising materials for thermal stability were the silphenylene polysiloxane fluids. They show no siloxane rearrangement as more conventional silicone fluids do and have stabilities up to the temperature range of 800-850°F. Polymer modifications gave good low temperature properties indicating a wide fluid range. Evaluation of various types of oxidation inhibitors showed that iron type additives still do the best inhibiting of silicone fluids. A new processing of 81644 has produced a fluid which is clear at room temperature and does not sludge after prolonged exposure at 500°F.

Armour Research Foundation. WADC TR 58-171, Part II.
DEVELOPMENT OF HIGH TEMPERATURE OXIDATION-CORROSION INHIBITORS TO IMPROVE STABILITY OF HIGH TEMPERATURE HYDRAULIC FLUIDS AND LUBRICANTS. Robert Brunier. AF 33(616)-5101. June 1959.

The objective of this research program was to synthesize and evaluate selected organic compounds as high temperature oxidation-corrosion inhibitors in paraffinic mineral oils, synthetic ester lubricants, methylchlorophenyl silicone, and diphenyldidodecylsilane.

After promising results were obtained with a compound containing a thionamide function attached to a heterocyclic ring, more substances from the same chemical class were selected and tested. A few of these compounds showed a certain degree of antioxidant action.

The compounds were evaluated by a micro oxidation-corrosion test. The results confirmed previous observations which indicated that the test method lacks sufficient reliability to permit conclusive and dependable evaluation of all the compounds tested.

Selected compositions were submitted to this oxidative test and in this case the oxygen uptake was also measured. This procedure indicated that the oxidation can be reduced to a limited degree with some of the mixtures used. However, no composition was able to satisfy all the specifications set as the goal of this program.

Olin Mathieson Chemical Corp. WADC TR 58-407 Part I.
FORMULATION OF SILANE BASE HIGH TEMPERATURE HYDRAULIC FLUID. Arthur W. Sawyer, Edward E. Harris. AF 33(616)-5292. June 1959. ASTIA Document No. AD 226379.

This report covers efforts to formulate (1) a silane-based high temperature hydraulic fluid suitable for use in the 0° to 700°F range and (2) a silane-based engine oil suitable for use at 450° to 500°F bulk oil temperature.

A large number of viscosity improver additives were evaluated. These included commercial and synthesized materials. None yielded the target viscosities. To date, the combination of thermal stability and viscosity improvement of a petroleum resin (Kendex) have not been surpassed.

Oxidation-corrosion does not appear to be a major problem under the test conditions of 24 hours at 550° and 700°F, in presence of 95% N_2-5% O_2. However, none of the antioxidants were satisfactory under engine oil test conditions which include 1 liter dry air per hour through a 25 ml. sample of fluid held at 500°F for 24 hours.

Olin Mathieson Chemical Corp. WADC TR 58-407, Part II.
FORMULATION OF SILANE BASE HIGH TEMPERATURE HYDRAULIC FLUID. Arthur W. Sawyer. AF 33(616)-5292. June 1959. ASTIA Document No. AD 225683.

During the period covered by this report, emphasis was placed on synthesis and evaluation of viscosity improver additives for a silane base 700°F hydraulic fluid and of an effective anti-oxidant for a silane base 450 to 500°F bulk engine lubricant.

A polymer prepared from para cymene was effective in increasing the viscosity of silane at 700°F to the target value of 1.2 centistokes, but both 0°F and 700°F target viscosities cannot be met with a single fluid. Attempts to produce effective, thermally stable, linear polymers of the following types have not yet been successful: polymerized petroleum resin, polyethers, and polybenzyl and polysilyl compounds.

Alkal metal amides have proven effective as antioxidants under silane engine oil test conditions. The most consistently effective materials found in this current work were the sodium and potassium amides produced from a mixture of secondary amines (aromatic). Viscosity increases of silane under oxidation test conditions typically range above 200%; 2% alkali metal amide markedly reduces this increase. In diphenyl di-n-dodecyl silane, 2% of the amide commonly limits the viscosity increase to below 30%, and in some duplicate tests, results have shown less than 10% change. This effect of these additives is less pronounced in octadecyl tri-n-decyl silane.

Alkylated acridine compounds were produced but were not very effective as antioxidants in silanes. Some metal-organic chelates were prepared; these were either insoluble, or ineffective as antioxidants, in silanes.

Several types of commercial antioxidant candidates were evaluated and found to be ineffective or insoluble in silanes.

Samples of an engine oil and two hydraulic fluid formulations were submitted at the end of this period.

WADC TR 58-287, Part I.
STORAGE STABILITY OF FLUIDS AND LUBRICANTS (DESERT STORAGE OF AIRCRAFT OILS). John B. Christian. June 1959. ASTIA Document No. AD 215546.

Six samples of oil were stored under desert conditions for five years. Withdrawals were made periodically for the purpose of determining the effects of desert storage on the oil's properties.

At the termination of the five years of storage, it was found that a number of the properties of the oils varied from the initial sample. Though these variations were evident, the properties of the oils generally met specification requirements. The mineral oil base jet turbine oil may be considered satisfactory. The diester turbo-jet cannot be properly evaluated from a use viewpoint since a key test was not included in this evaluation.

WADC TN 59-160, Part I.
HIGH TEMPERATURE EVALUATION PROCEDURES FOR LUBRICANTS. Vernon A. Lauer, Donald C. Trop. August 1959. ASTIA Document No. AD 212224 PB 151961. Order from OTS $2.50.

This report describes the design, development and instrumentation of a high temperature aluminum block bath capable of attaining and controlling temperatures up to 1000°F ± 5°F. This report also describes two high temperature viscosity baths capable of attaining and maintaining 400°F and 700°F respectively.

California Research Corp. WADC TR 59-252.
DEVELOPMENT OF NUCLEAR RADIATION-RESISTANT HYDRAULIC FLUIDS. N. W. Furby, S. R. Calish, M. A. Pino, D. R. Wilgus. AF 33(616)-5583. October 1959. ASTIA Document No. AD 230655.

Synthesis work during this contract period had several parts. A synthetic base fluid, isopropyl-1, 9-diphenylnonane, having an excellent combination of viscosity-temperature properties and stability against the common types of degradation, was developed. Gallon quantities of this material were prepared for formulation studies and evaluation tests.

Various polyalkylaromatics were synthesized for evaluation as viscosity-temperature improvers. A large laboratory batch of the C_{14-16} alkyl diphenyl ether-p-xylene copolymer was prepared for use in the final fluid formulation, CALRESEARCH 59R-439. Starting with the base fluid, isopropyl-1, 9-diphenyl-nonane, this fluid was prepared after extensive formulation studies.

CALRESEARCH 59R-439 has substantially better oxidative, thermal and hydrolytic stability, a greater low temperature capability, and superior radiation resistance than CALRESEARCH 216. CALRESEARCH 216 was developed under the previous contract effort, and is composed of a C_{14-16}-alkyl diphenyl ether base fluid plus additives.

New leads, related to the in situ formation of polymeric materials in α, ω-diarylalkanes by treating with aluminum chloride, may make possible the development of superior fluids. This opens a fertile field for future research.

New experimental fluids were tested before and after irradiation. Much valuable knowledge pertaining to factors contributing to stability against radiation, thermal, oxidative, shear, and hydrolytic damage was gained. A radiation process which enhances shear stability of thickened fluids was discovered.

Sundstrand Aviation Division. WADC TR 59-760.
HIGH TEMPERATURE FLUID EVALUATION TEST STAND. Luke J. Gabrovic. AF 33(616)-3126. April 1960. ASTIA Document No. AD 244691.

A hydraulic fluid test stand with the capability of evaluating fluids at temperatures up to 700 F and pressures up to 3000 psi, has been constructed. A survey of the industry showed several areas in which reliable aircraft type components were not available, and in such instances, the best non-aircraft components were adapted to complete the test stand.

The report describes the fluid evaluation parameters, the finalized test procedures to be used for fluid analysis, and the selection of components.

The test results of two fluid evaluations are included in the report.

WADD TR 60-250, Part I.
HIGH TEMPERATURE HYDRAULIC FLUIDS - PART I. Chemical Degradation in a High Temperature, High Pressure Circulation Loop. Robert J. Benzing, Leslie R. Drane, Jr. May 1960. PB 161965. Order from OTS $0.50.

A study was made to observe the nature and intensity of chemical degradation resulting from severe mechanical shearing stresses at elevated (400°F) temperature. The degradation of these four fluids (two silicones, a silane, and an alkoxy disilosane) were also studied under static thermal conditions. The physical and chemical properties, particularly viscosity, neutralization number, and flash point showed changes in direction of values with time. Possible mechanisms of break-down to explain observed differences between the static and dynamic tests are discussed.

Lockheed Nuclear Products. WADD TR 59-592. GAMMA IRRADIATION OF AN ELECTRO-HYDRAULIC SERVO TEST LOOP USING ORONITE 8515 HYDRAULIC FLUID. A. MacCullen. AF 33(600)-32055. May 1960.

This summary technical report of the irradiation of an electro-hydraulic servo test loop dynamically operated in a gamma environment includes a brief historical background, the object and scope of the program, and the procedures and results. The test was conducted for 260 hours with an average dose rate of 1.1 x 10 ergs/gm-hr (C). The fluid temperature was maintained at 275°F, and the nominal pressure was 3000 psi.

WADD TR 60-352.
PERFORMANCE OF PETROLEUM BASED HYDRAULIC FLUIDS IN A PUMP LOOP AT 3000 PSI AND 275°F. Robert N. Johnson. June 1960. PB 171063. Order from OTS $2.25.

Fluid breakdown and pump wear were investigated in a pump loop at 275°F and 3000 psi on five petroleum based hydraulic fluids.

The mechanical shearing of these fluids resulted in excessive loss of viscosity, although their resistance to oxidation was not appreciably changed. The wide variations in the wear data of these fluids indicated their difference in lubrication characteristics in the particular system employed. Bench wear data did not correlate with the pump wear results.

Vickers Incorporated. WADD TR 60-394. INVESTIGATION OF THE LUBRICATING CHARACTERISTICS OF HIGH TEMPERATURE POWER TRANSMISSION FLUIDS. Bud N. Barclay. AF 33(616)-6498. July 1960. ASTIA Document No. AD 247171.

This program was conducted to test and determine the feasibility and potential of high temperature power transmission fluids for use in 550 F hydraulic systems. These fluids were tested for a particular purpose, and the results do not reflect on the ability of the fluids to operate satisfactorily under other conditions. The following fluids were tested under contract AF 33(616)-6398:

1. Mineral Oil MLO 7460 - A supperrefined mineral oil.
2. Silicone MLO 59-250 - A halogenated methyl phenyl silicone.
3. Silane MLO 59-92 - Diphenyl d-n-dodecyl silane.
4. Silane Mixture - 50% Silane MLO 59-92 and 50% Silane MLO 59-91 (MLO 59-91 is essentially the same as MLO 59-92, but contains a lesser quantity of viscosity index improver).
5. TMP Ester MLO 59-699 - ester derivative of trimethanol propane.
6. Silicone-Ester Blend MLO 59-700 - 50% TMP ester MLO 59-699 and 50% chlorinated phenyl methyl silicone.
7. M-Bis (m°Phenoxyphenoxy) Benzene MLO 59-692.

The test fluids were subjected to 550°F testing at 3000 psi in a Vickers axial-piston pump designed for high temperature operation. Of the fluids tested, the silane-base formulations, Silane MLO 59-92 and Silane Mixture (MLO 59-92 and MLO 59-91), exhibited the best apparent potential for 550°F operation, enabling pump runs of 50 and 100 hours respectively.

The low viscosity of MLO 7460 at 550°F resulted in pump bearing failures; nevertheless, this fluid appears to offer a good potential for high temperature applications. The MLO 59-692 fluid, m-Bis(m-Phenoxyphenoxy) Benzene, exhibited excellent thermal stability, but the 550 F viscosity was too low to enable continued operation of the test pump.

Silicone MLO 59-250, TMP Ester MLO 59-699, and Silicone-Ester Blend MLO 59-700 did not maintain adequate chemical stability at 550°F. The consequent increase in acid content resulted in pump damage and necessitated early termination of tests.

The results of this program indicate that operation of a hydraulic pump on existing hydraulic fluids at 550°F is feasible. It is further concluded that the present high temperature pump design can be modified to enable extended operation on some of the fluids tested.

WADD TR 60-467.
SHEAR STABILITY OF HYDRAULIC FLUIDS BY SONIC SHEAR. Donald C. Trop. June 1960. ASTIA Document No. AD 245677.

This report presents conclusive proof that shear stability data obtained on hydraulic fluids by sonic irradiation compares quite favorably with data obtained by the present shear stability test procedure (pump-testing) in the MIL-H-5606 Specification. The sonic irradiation method should be used to replace the present pump testing procedure since it reduces test time from hours to minutes and sample quantities from gallons to milliliters.

WADD TR 60-583, Part I.
LUBRICITY OF EXPERIMENTAL HIGH TEMPERATURE POWER TRANSMISSION FLUIDS - PART I. Pump Wear at 275°F. Leslie R. Drane, Jr., R. J. Benzing. September 1960.

The lubricity of several experimental high temperature power transmission fluids of widely varying chemical types was investigated. A New York Air Brake Company pump was used at 275°F as a means of studying the wear behavior. Four-Ball wear tests also were run. The fluids consisted of a silicone-ester blend, and ether and two disiloxanes. In general, these high temperature fluids gave comparable lubrication to that afforded by normal temperature petroleum fluids. In most cases the desired five hundred hours of operation were successfully completed. Such performance demonstrates the feasibility of the experimental fluids. No correlation could be obtained between the Four-Ball wear tests and the pump wear tests.

WADD TR 60-795, Part I.
EXTREME TEMPERATURE RANGE ORGANIC COOLANTS - PART I. -80°F to 400°F Temperature Range Fluids. Lt. Dale A. Barsness. December 1960. ASTIA Document No. AD 258597. Order from OTS $1.00.

The increased performance demands of advanced aerospace systems dictate the requirement for new and improved heat transfer materials capable of operating over extreme temperature ranges. A review of the status of extreme temperature range (ETR) coolants for electronic equipment is presented. The fluids considered for use over a -80°F to 400°F temperature range are described in this report and fall into the following class of compounds: amines, chlorofluoro-carbon oils, fluorochemicals, silicones, silicate esters, and silcanes. Results show that the silicone fluids comply most closely to the physical properties required for ETR electronic coolants. It was observed that they are the only group of materials that possess high boiling points and thermal stabilities above the 400°F bulk operational temperature, and still display reasonably low viscosity values within the extreme temperature range. The electrical properties of these materials are also very desirable.

The data on the new experimental research fluids, reported herein, list properties important to future coolants for electronic components. These data include the viscosity temperature relation, vapor pressure-temperature properties, electrical characteristics, thermal properties and flammability characteristics.

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Midwest Research Institute. WADD TR 60-855. LUBRICATION BEHAVIOR AND CHEMICAL DEGRADATION CHARACTERISTICS OF EXPERIMENTAL HIGH TEMPERATURE FLUIDS AND LUBRICANTS. Vernice Hopkins, Andrew D. St. John. AF 33(616)-6854. January 1961. ASTIA Document No. AD 258563.

The results of seven 100-hr fluid shear stability experiments, six at 550°F and one at 275°F, indicate that MLO 56-834 (chlorinated phenyl silicone), and MLO 57-637 (di-n-dodecyldiphenylsilane) resist degradation from high shear stresses. MLO 59-98 (50-50 blend of phenyl methyl silicone and ester of TMP), and MLO 8200 (hexa-2-ethyl butoxydisiloxane) experienced a molecular change which substantially altered their properties. MLO 7277 (superfined mineral oil) experienced a slight molecular change, and its properties were slightly changed. An experiment is described wh.re the wear of various lubricated contacts can also be studied along with the shear stability of experimental fluids.

An apparatus for studying lubricant behavior in rolling and rolling-sliding contacts is described and results for initial runs are presented. The initial wear-life results of solid film lubrication of spherical surfaces are presented and discussed. Based on data from a plane on a cylinder configuration, these results were lower than expected. Steps taken to make the high pressure viscometer a self-contained working unit are outlined and the pressure limitation resulting from bellows configuration anf fluid compressibility is discussed.

Results for extreme pressure behavior of lubricants are presented for a series of runs at 600°F and 275°F. Mating surfaces of M-10 tool steel were used for the 600°F runs while 52100 steel was used for the 275°F

Lubricants and Solid Film Lubricants

TR 5685.
APPARENT VISCOSITY OF GREASES. Bernard Rubin. April 1948.

Shell Development Co. WADC TR 59-173. NUCLEAR RADIATION RESISTANT HIGH TEMPERATURE LUBRICANTS. C. L. Mahoney, E. R. Barnum, W. S. Saari, K. J. Sax, W. W. Kerlin. AF 33(616)-5617. September 1951.

Polyphenyl ethers are very promising materials for further development as radiation-resistant high-temperature lubricants. The unsubstituted polyphenyl ethers are far more stable than presently-used lubricants and can be classed with the most resistant types of aromatic compounds (polyphenyls, aromatic silanes, etc.) with respect to radiation, oxidation and thermal stability. Furthermore, these ethers have much lower melting points, better physical properties and much better lubrication characteristics than the other aromatic materials. Unsubstituted meta-linked polyphenyl ethers having pour points of 5°F and 40°F have been prepared. Initial thermal decomposition temperatures of these ethers are 830°F or higher.

Selected alkyl-substituted polyphenyl ethers, cumyl and tertbutyl derivatives, although less stable than the unsubstituted compounds are much more resistant to radiation, oxidation and thermal decomposition than typical antioxidant-containing oils. The alkyl derivatives are also good lubricants as judged by bearing and gear tests.

Although the development of polyphenyl ethers has been guided largely by predicted requirements of future turbo-jet engines their properties indicate that they should also find application in other fields where extreme conditions are encountered. Suggested applications include high-temperature hydraulic fluids and heat-transfer media.

Armour Research Foundation. WADC TR 52-22. LOW TEMPERATURE LUBRICATING OIL ADDITIVES. G. Gavlin, S. P. Jones, Jr., E. A. Swire. AF 33(038)-1644. September 1952. PB 112118. Order from LC, Mi $2.25, Ph $5.00.

Armour Research Foundation. TR 52-220. STUDIES TO DETERMINE THE ELECTRICAL AND PHYSICAL PROPERTIES OF AIRCRAFT LUBRICATING OILS. Joseph L. Radnik. PO 33(038)-3793. September 1952. PB 135096. Order from LC, Mi $7.50, Ph $24.30.

Armour Research Foundation, Illinois Institute of Technology. TR 53-11.
LOW-TEMPERATURE LUBRICATING ENGINE OIL ADDITIVES. Gilbert Gavlin, Edwin A. Swire, Sam P. Jones, Jr. AF 33(038)-1644. April 1953. PB 119093. Order from LC, Mi $11.10, Ph $58.85.

Purdue University. TR 53-79 (R).
CHLORINE AND FLUORINE CONTAINING COMPOUNDS FOR NON-FLAMMABLE MATERIALS. Ogden R. Pierce, Earl T. McBee. W33-038-ac-19024. May 1953. PB 139025. Order from LC, Mi $8.40, Ph $28.80.

A number of bromine-containing compounds were evaluated as snuffer additives in MIL-O-5606 fluid. The most effective materials for this purpose were found to be brominated aliphatic hydrocarbons of short chain length.

For the studies of new base stock oils several new classes of fluorine and/or silicon containing materials were investigated. A series of compounds containing both silicon and phosphorus were synthesized and were found to possess good viscosity and non-flammability properties. Various fluorine-containing crotonyl and phosphonate ester systems were studied and the preparation and reactions of n-heptafluoropropyllithium investigated. The synthesis and evaluation of a fluorine-containing polyether and fluorine-containing silicate ester were also carried out.

Pennsylvania State College. TR 53-25 (R).
DEVELOPMENT WORK ON LUBRICANTS FOR AIRCRAFT TURBINE ENGINES. E. Erwin Klaus, Merrell R. Fenske. AF 33(038)-18193. May 1953.

Work on the synthetic ester type of gear lubricant has been continued. An experimental gear lubricant PRL 3161 has been proposed as an improvement over PRL 3059 gear lubricant. PRL 3161 has been prepared to give improved oxidation and corrosion stability under the severe conditions of the turbine bearing. Advantages of PRL 3161 over di-2-ethylhexyl sebacate and Specification AN-O-3M mineral oil in viscosity-temperature characteristics are illustrated.

Comparison of the four-ball wear data with the turbo-prop gearbox results indicates some agreement between (a) good anti-wear properties and a high point of incipient seizure in the wear tests, and (b) satisfactory operation in the turbo-prop engine and gear box. The four-ball wear tests do not evaluate the viscosity function of a lubricant.

Fourteen different organic phosphates have been evaluated as anti-wear and E.P. additives in the four-ball machines. The wear characteristics imparted by the phosphate or acid phosphates additives are substantially the same for alkyl, aryl, alkyl-aryl organic constituents.

Oxidation and corrosion characteristics of experimental synthetic ester gear lubes have been measured by means of six different procedures using aluminum, magnesium, steel, copper, cadmium-plated steel, silver-plated steel, and lead-indium coated silver-plated steel as metal catalysts in some of the tests.

A survey of the thermal stability of various esters has been made to 500°F under a nitrogen atmosphere.

Wear characteristics of several ester type gear lubes have been determined after subjecting the fluid to various oxidation and corrosion tests. These values are then compared with similar values for the unused fluids. These results are indicative of the persistence of the anti-wear and E.P. additives under conditions of severe accelerated use.

PRL 3161 fluid has been service tested for a total of 237 hours in a T-38 Allison turbo-prop engine. The lubrication problems encountered in this test are outlined briefly. Properties of the used PRL 3161 fluid taken from this test are discussed. The properties evaluated include viscosity, neutralization number, wear and lubrication, and oxidation and corrosion stability. PRL 3161 exhibits excellent stability in service use.

Many of the studies in this report are basic on fluids using di-2-ethylhexyl sebacate as the base stock. An additional investigation has been made on other esters that are important commercially for the preparation of PRL 3161 type fluids.

A specification has been suggested for use in the commercial procurement of synthetic gear lubricants of the type discussed in this report.

Engineering Research Institute, University of Michigan. TR 53-45 (R).
SYNTHETIC LUBRICANTS. D. W. McCready. W33-038-ac-21457. June 1953. PB 130818. Order from LC, Mi $5.40, Ph $15.30.

This project was established to study the possibility of synthesizing a pure compound to be used as a synthetic lubricant in aircraft engines operating at ambient temperatures ranging from -65 to 160°F. The syntheses of the 84 compounds prepared are described in this report. The viscosity-temperature characteristics and melting points of the compounds have been determined. Other physical properties such as density, vapor pressure, specific heat, thermal conductivity, etc., have been determined on some of the compounds.

None of the compounds synthesized meets the viscosity specifications of 65 cSt. at 210°F and 3000 cSt. at -65°F. The compounds which most nearly meet this specification and which show the greatest promise of meeting the specification are compounds having the "dumbell" structure with terminal cyclopentyl rings along with the thioether structure.

Standard Oil Company. TR 53-83.
DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASES. Cecil G. Brannen, Edward A. Swakon. AF 33(038)-23687. July 1953.

In the work directed toward the development of an aircraft grease suitable for use over a wide temperature range, emphasis has been placed on the development of thickeners for silicone oil and on the evaluation of silicone-oil greases at high temperatures. Studies were made on one hundred and one silicone-oil greases containing as thickeners representatives of ten classes of materials that might be expected to produce thermally stable greases. Each composition was subjected to simple laboratory tests for preliminary evaluation, and twenty-nine compositions were run in the ABEC-NLGI bearing tester. The greases containing inorganic thickeners showed poor performance in the bearing tester, all failing in less than one hundred hours at 450°F. Several tests of two hundred hours or more were obtained with copper phthalocyanine, calcium acetate, and urea greases. One test of six hundred and fifty hours was obtained with a grease containing p-carboxydiphenylurea as thickener. This test satisfies the high-temperature bearing-test requirement of the contract.

University of Virginia. TR 53-293 Part I.
HIGH-TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC-BASE OILS PART I - TESTING METHODS AND PRELIMINARY RESULTS. James W. Cole, Jr., Arthur F. Benton, Alfred Burger, Thomas I. Crowell. AF 33(038)-22947. October 1953. PB 121077. Order from OTS $3.50.

PHASE I. Literature Survey. The literature on oxidation and corrosion inhibitors for the various oils was completely surveyed for the period 1916 to date. There seem to be no pertinent references earlier than 1916. Each of the approximately 2500 references to research reports, patents, reviews, etc. was recorded and coded on a McBee Keysort punch card. Three punch card indices were devised covering 105 entries, each being designated by a four letter code word. A novel card needling box was designed and constructed to facilitate the use of the punch card file. A detailed report on the literature will be the subject of a separate memorandum report.

PHASE II. Evaluation of Additives and Mechanism Studies. With a specially constructed aluminum block thermostat, 60 likely-looking compounds of various types were tested as oxidation-corrosion inhibitors for di-(2-ethylhexyl) sebacate (Rohm and Haas, Plexol 201). Substances of the phenothiazine-type were the most effective at 400°F. These types were exhaustively studied and the effects of concentration, small changes of temperatures, metals, and substituents shown. Probably the most effective compounds were N-methyl- and N-ethyl-phenothiazine. At 400°F a concentration of about 1.6% is needed to hold the oxidation induction period for 72 hours in the presence of copper.

Systematic studies were started on the mechanisms of pyrolysis and oxidation of diesters, the chemistry of phenothiazine and the mechanism of its action, the rate of depletion of additives, and the role of metals in the oxidation phenomena. Each of these is of a complex nature and only tentative conclusions were reached.

PHASE III. Research and Development of New Materials. Efforts were directed toward improving syntheses of some of the substances tested in Phase II and preparing new compounds together with the study of properties of the more interesting compounds. Work was under the following headings: Purification of phenothiazine; preparation of p,p'-dihydroxydiphenylamine, p-benzyloxydiphenylamine, 3-benzyloxyphenothiazine, p-t-butoxydiphenylamine, 10-methylphenothiazine, 3-formyl-10-methylphenothiazine, 3-formyl-10-methylphenothiazine oxime, 3-(10-methylphenothiazinyl)methanol, β-[3-10-methylphenothiazinyl] acrylic acid, 1-nitro-2-[3-(10-methylphenothiazinyl]ethene, phenyl-beta-naphthylamine and 7-benzo(c)phenothiazine; nitration of 7-benzo(c) phenothiazine; preparation of N-acetyl-7-benzo(c) phenothiazine; nitration of N-acetyl-7-benzo(c) phenothiazine; Friedel-Crafts acetylation of N-acetyl-7-benzo(c) phenothiazine; preparation of x-acetyl-7-benzo(c)phenothiazine oxime; attempted rearrangement of the oxime of x-acetyl-7-benzo(c)phenothiazine; preparation of N-methyl-7-benzo (c)phenothiazine, N-methyl-7-benzo(c)phenothiazome-S-oxide, 7-benzo(c)phenothiazine-S-oxide and x-carboxy-7-benzo(c)phenothiazine.

Standard Oil Development Company. TR 53-68. SYNTHETIC LUBRICANTS. W. E. McTurk. AF 33(038)-14593. October 1953. PB 111565. Order from OTS $4.25.

This report is a compilation of all the information obtained under Contract No. AF 33(038)-14593, concerned with means of increasing the availability of synthetic lubricants for use at low and high temperatures.

The diesters of straight-chain dibasic acids lead the field of esters suitable as lubricants for use at both low and high temperatures, because of their desirable combinations of properties and potentially good availability. Adipic, azelaic, and sebacic acids are the most readily available dibasic acids suitable for ester lubricant production, while the petroleum derived Oxo alcohols appear to be the most available alcohols for this application. In addition, however, certain diesters of polypropylene glycols appear equivalent to dibasic acid esters in all the characteristics studied so far, and this type of ester therefore represents a promising source of synthetic oil. Mono-esters may be satisfactory lubricants where high temperatures are not encountered. The presence of small quantities of impurities are believed to cause variations in the oxidation and thermal stability of esters. The SAE E.P. Lubricants Tester is capable of measuring the load-carrying ability of synthetic lubricants, but further work is required to develop a completely reliable test for predicting full-scale gear performance.

The Research Institute of Temple University. TR 53-303. LOW-TEMPERATURE, HEAT AND OXIDATION STABLE MATERIALS AS POSSIBLE LUBRICANTS AND ELASTOMERS. Murray Hauptschein, Charles S. Stokes. AF 33(038)-10844. November 1953. PB 125043. Order from LC, Mi $3.60, Ph $9.30.

As part of the program of correlating properties with structural features of various types of fluorinated ester-type compounds, a good deal of research was carried out on devising methods for the syntheses of di-, tri-, tetra- and poly- esters.

There have been synthesized sizeable quantities of seven polyethylene glycol di-n-perfluorobutyrates and a polypropylene glycol di-n-perfluorobutyrate. In addition the nonfluorinated analogs of the polyethylene glycol di-n-perfluorobutyrates have been prepared for purposes of comparison.

The synthesis of 1,5-di-(2'-cyanoethoxy) pentane and the attempted conversion of this compound to 1,5-di-(2'carboxyethoxy) pentane is given.

Two unsuccessful attempts to prepare 1-iodo-1,1,2,2-tetrahydroperfluoropentane by the reaction of 1-iodoheptafluoropropane with ethylene are described.

The preparation of the triester, 2-2'-hydroxyethoxymethyl)-2,4-dimethyl pentanediol-1,5 triperfluorobutyrate and the attempted preparation of 2-nitro-2-hydroxymethyl propanediol-1,3 triperfluorobutyrate are reported.

The synthesis of two half-ester acids, namely, mono-n-1,1-dihydroperfluorobutyl perfluoroglutarate and mono-2-ethyl-n-hexyl perfluoroglutarate made possible the preparation, in good yields, of four pure tetraesters. These compounds are: (1) pentamethylene dithiol bis (mono-n-1,1-dihydroperfluorobutyl perfluoroglutarate); (2) pentamethylene glycol bis (mono-n-1,1-dihydroperfluorobutyl perfluoroglutarate); (3) trimethylene glycol bis (mono-n-1,1-dihydroperfluorobutyl perfluoroglutarate); (4) trimethylene glycol bis (mono-2-ethyl-n-hexyl perfluoroglutarate).

Due to the very great difficulty in the preparation of pure mono-esters of glycols of the types $C_3F_7COO(CH_2)_nOH$ and $C_3F_7COOCH_2CH_2OCH_2CH_2OH$, as well as in the preparation of pure half ester-acids such as $C_3F_7CH_2OOC(CH_2)_4COOH$, only impure tetraesters could be obtained on reacting these intermediates with various dicarboxylic acids and glycols, respectively.

The completely branched tetra ester, pentaerythritol monocaproate triperfluorobutyrate has been synthesized.

The preparation and properties of the polyester, poly-(diethylene glycol perfluoroglutarate) are reported.

Correlations on the viscosity and surface tension characteristics of the various series of ester-type materials have been made.

Southwest Research Institute. TR 53-337. POLYNUCLEAR AROMATIC COMPOUNDS FOR HIGH TEMPERATURE LUBRICANTS. Charles F. Raley, Jr. AF 33(616)-276. November 1953. PB 130242. Order from LC, Mi $3.30, Ph $7.80.

A literature survey covering the field of polynuclear aromatic compounds, with two or three nuclear rings, was made. All materials liquid at 35°C and stable up to 400°F were entered on file cards. From the data thus gathered, a list of seventeen compounds, considered to be representative of the common chemical structures, was compiled. These compounds were synthesized and evaluated as to thermal stability, viscosity at 100°F, 210°F, density, and boiling point. On the basis of this evaluation, the field of aryl phosphates was considered to have the most likely chance of providing compounds with the desired properties. Further research in this field has produced three liquid compounds with exceptional thermal stabilities, i.e., above 700°F; di-p-tolyl 1-naphthyl phosphate, di-m-tolyl 1-naphthyl phosphate, and tri-o-chlorophenyl phosphate. In the case of the first two compounds, all other screening tests, such as flash and fire point, autogenous ignition temperature and hydrolytic stability, gave excellent results. Other compounds which will be synthesized offer a reasonable chance of possessing even better properties.

University of Virginia. TR 53-293, Part II. HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS. PART 2 - EVALUATION OF ADDITIVES, SYNTHESES OF NEW COMPOUNDS, AND MECHANISM STUDIES. James W. Cole, Jr., Gordon P. Brown, Alfred Burger, Thomas I. Crowell. AF 33(038)-22947. December 1953. PB 121078. Order from OTS $4.50.

A total of approximately 200 compounds has been evaluated as oxidation-corrosion inhibitors in various ways in a total of 13 synthetic base oils. The base oils used include diesters, phosphonates, silicates and silicones.

Various methods of comparing effectiveness of additives are discussed. There is as yet no one satisfactory method for rating a particular compound in a specific oil.

Phenothiazine and some of its derivatives remain the best inhibitors, as a class, for the diesters at 204.4°C. Preliminary results suggest that phenothiazone-3 may be the best of this type. Two N-alkyl-alpha-naphthylamine derivatives, N-methyl- and N-ethyl-, also show good inhibitor properties in di (2-ethylhexyl) sebacate.

Many rubber antioxidants, particularly those containing sulfur, showed good antioxidant properties in the diester. However, these attacked copper in excess of that allowed in the specifications.

In mixed C_6-C_8 silicate and tetra (2-ethylhexyl) silicate, N-phenyl-alpha-naphthylamine continues to be one of the most effective oxidation inhibitors. Preliminary tests indicate that N-ethyl-alpha-naphthylamine, some

p-aminophenols and some p-phenylenediamines may be of equal activity.

DC Silicone 550 apparently needs no oxidation-corrosion inhibitors to meet the specifications at 204.4°C since it has an induction period in excess of 75 hours. Indeed, the change in measured properties of DC Silicone 550 is generally greater in the presence of those additives tested than it is in the absence of additives. This increase in apparent oxidation in the presence of additives might be assumed to be due to changes in the additives.

Mechanism studies have been conducted on the action of additives and the media under conditions of oxidation, pyrolysis and metal catalysis. The products of pyrolysis of di (2-ethylhexyl) sebacate in the range 260°-305°C over the first 10% of the reaction have been identified as 2-ethylhexene-1 and the mono-ester, 2-ethylhexyl hydrogen sebacate.

Studies have been made on the rate of disappearance of phenothiazine in di (2-ethylhexyl) sebacate in the presence and absence of copper, either as the metal or as a salt. The rate of disappearance of phenothiazine is slowed in the presence of copper but the length of induction period is shortened. An attempt is made to explain this phenomenon on the basis of accepted reaction mechanisms. It is also shown that the rate of disappearance of phenothiazine closely resembles first-order kinetics.

University of Virginia. TR 53-293, Part 3. HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS. PART 3 - THE THERMAL DECOMPOSITION OF DI-2-ETHYLEHEXYL-SEBACATE. Earl E. Sommers, Thomas I. Crowell. AF 33(038)-22947. December 1953. PB 121079. Order from OTS $1.25.

A brief survey of the literature is given describing the postulated mechanisms by which esters of the type in question decompose. The determination of the rate of thermal decomposition of di-(2-ethylhexyl) sebacate in the liquid phase in the temperature range of from 260° to 305°C is described. Conclusions to be drawn are that the decomposition proceeds only at a very slow rate at 204°C, yet it does occur; and that it is a simple reaction, i.e., not of the free radical type, nor is it affected by acids or a copper surface.

From the literature it is concluded that certain ester types which might be expected to be stable at these temperatures should be prepared and subjected to conditions analogous to, or more drastic than those described herein.

University of Virginia. TR 53-393, Part 4. HIGH-TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS. PART 4 - STUDIES IN THE COPPER-PHENOTHIAZINE Di-(2-ETHYLHEXYL) SEBACATE-SYSTEM IN THE NEIGHBORHOOD OF 200°C (400°F). James W. Cole, Jr., Lewis G. Cochran. AF 33(038)-22947. December 1953.

This is a detailed study at several temperatures in the neighborhood of 200°C of anomalous behavior encountered in the oxidation studies of diester lubricants in the presence of phenothiazine and copper and was designed to shed more light on the mechanisms of inhibitor action and metal catalysis. The behavior of phenothiazine in di-(2-ethylhexyl) sebacate under controlled oxidation conditions was followed by noting the change in the characteristic ultraviolet absorption spectra and by chemical methods. The rates of decrease of absorption of phenothiazine at its characteristic maximum, 2520 Å, indicated that phenothiazine disappeared less rapidly in presence of copper than when this substance was absent, even though the overall oxidation as measured by rate of oxygen absorption was more rapid. The effect of copper salts of the dibasic acids, sebacic and oxalic, was of the same order, but the detailed effect of concentration of the dissolved salts was complex. The results were interpreted in terms of possible reaction mechanisms. It appears that phenothiazine is rapidly converted to an intermediate oxidation state of finite life through energy-rich chains formed in the medium. The slower rate of disappearance in the presence of copper apparently arises from the action of the metal converting the energy-rich chain carriers relatively faster into free-radical chains and thereby reducing the concentration of the energy-rich units which act to convert phenothiazine to the intermediate oxidation state. The net effect in the presence of copper is that phenothiazine has less opportunity to become activated while at the same time the active form is dissipated more rapidly and inactive oxidation products form at a faster rate. A rate equation is derived for the disappearance of phenothiazine and the mechanisms proposed are related to the possibility of predicting antioxidants for higher temperatures.

TR 54-44.
INVESTIGATION OF "BENTONE-34" AS A GREASE THICKENER. Herbert Schwenker. RDO No. 613-11 (E-A). April 1954.

The project was initiated to develop greases from domestic, non-critical materials that will ensure the Air Force a supply of greases whose source will not be limited or cut off because of war or other emergencies.

"Bentone 34" was used as a thickening agent with various synthetic and petroleum oils in preparing greases. The greases were tested for their physical and chemical properties as possible aircraft lubricants. The test results indicated that it may be possible to formulate aircraft lubricating greases using "Bentone 34" as a thickener which will be equal in performance to the soap-type greases now in use.

Iowa State College. WADC TR 53-426 (Part 1). ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS. Henry Gilman, Robert K. Ingham, Richard D. Gorsich. AF 33(616)94. April 1954. PB 129468. Order from LC, Mi $6.30, Ph $19.80.

University of Virginia. WADC TR 53-353, Supp. 1. SURVEY OF THE LITERATURE ON ANTIOXIDANTS AND ANTICORROSION ADDITIVES FOR LUBRICANTS AT ELEVATED TEMPERATURES. James W. Cole, Jr., Alfred Burger and Arthur F. Benton. AF 33 (038)-22947. May 1954. ASTIA Document No. 34186. PB 121726. Order from OTS $10.00.

The literature was surveyed on oxidation and corrosion inhibitors in various media at elevated temperatures. Particular attention was given to phenothiazine types of additives and to lubricants of the synthetic-base type. Media, such as rubber, were included where the effects noted might have analogies in synthetic base lubricants. The literature revealed very few antioxidant or anticorrosion combinations for synthetic base lubricants at extreme temperatures. The survey covered the period 1916 to June 1952. There were very few significant literature references prior to 1916. The survey can be readily brought up-to-date when the 1952 subject index of Chemical Abstracts becomes available. Approximately 2500

abstracts of research reports, patents, reviews, articles, etc. were recorded on McBee Keysort Punch Cards of special design. Three punch card indices were devised covering approximately 100 entries, each entry being designated by a four-letter code word. A novel card-needling box was designed and constructed to facilitate use of the card file.

WADC TR 54-46.
AN INVESTIGATION OF LITHIUM 9/10 HYDROXY-STEARATE GREASES. Harvey C. Markle, Jr., Herbert Schwenker. August 1954. ASTIA Document No. 45579. PB 129527. Order from LC, Mi $2.70, Ph $4.80.

The melting points of lithium soaps, which were prepared from hydroxystearic acids, were determined. These soaps were combined with synthetic oils to prepare greases which, in turn, were analyzed to determine their physical and chemical properties. Except for water resistance, oil separation, and mechanical stability, the greases made from lithium 9 or 10 hydroxystearate were found to possess properties equal to those of lithium 12 hydroxystearate.

Western Gear Works. WADC TR 54-37.
DEVELOPMENT OF A GEAR AND SPLINE LUBRICANT TESTER. John Morris, Joseph Goldsworthy, John Scott, Charles Sauter. AF 33(616)-496. October 1954. ASTIA Document No. AD 52497. PB 132284. Order from LC, Mi $5.40, Ph $15.30.

Results of a compilation of lubricants, lubrication, and lubricant tester data include a selected bibliography with abstracts which has been arranged so that references are listed under the subject heading most applicable.

Design of a gear and spline lubricant tester which will accommodate, as test specimens, all the most popularly used types of gears; such as, spur, helical, worm, straight bevel, spiral bevel, and hypoid, as well as splines, is described. The lubricant tester will be capable of test gear speeds up to 30,000 RPM and tooth loads up to 6000 pounds per inch of face width.

The design of the tester was based on the analysis of information from sources listed in the bibliography, gear lubrication experience, and preliminary design studies of possible new simulation, as well as gear type testers.

It is concluded that a universal type, gear lubricant tester should prove advantageous in selecting lubricants for specific applications, as well as placing lubricants into a general classification of usefulness as gear lubricants.

The compilation of information, analysis, and final design of a gear and spline lubricant tester was conducted by Western Gear Works' Research Engineering Group.

WADC TR 54-157.
SYNTHETIC LUBRICANTS FOR AIRCRAFT. Herbert Schwenker, John A. King, James C. Mosteller. November 1954. ASTIA Document No. AD 63282. PB 128088. Order from LC, Mi $3.90, Ph $10.80.

As military aircraft fly at higher altitudes and speeds, the requirements for lubricants to afford greater resistance to thermal effects, wider liquid or usable temperature ranges, and longer life has and will continue to be an interesting research and development program. Petroleum oils as a source for greases, aircraft hydraulic fluids, engine oils and special purpose lubricants are rapidly being replaced by synthetic materials and will soon be only of historical interest.

Development of a -65° to 450°F grease for use in anti-friction bearings, electronic devices and other types of aircraft equipment is in progress. Emphasis is being placed on improving the wear characteristics of this grease and extending the low temperature limits. In addition, research and development in greases is being devoted to improvement of grease availability and the investigation of oils and thickening agents.

Synthetic lubricants for turbo-prop and turbo-jet applications have been formulated for use at temperatures of -65° to greater than 400°F. High gear loadings encountered in some engine and accessory applications have required extensive research and development of suitable anti-wear additives.

The presently available hydraulic fluids offer a maximum usable temperature range of approximately 250°F. Speed, miniaturisation of equipment, together with the necessity of operating hydraulic systems near heat producing bodies has increased this temperature range to greater than 400°F. Experimental fluids of the diester and the organosilicon classes have been developed and are being evaluated.

Stanford Research Institute. WADC TR 54-339.
DETERMINATION OF THE MECHANISM OF THE INCREASE OF VISCOSITY OF ORGANOSILICON COMPOUNDS AT HIGH TEMPERATURES. O. F. Senn. AF 33(616)-168. February 1955.

Oxidation of alkoxy-or aryloxysilanes is inevitably accompanied by hydrolysis. Oxidation rates were determined for tetraphenoxysilane, tetra(2-ethylhexoxy)silane, hexa(2-butoxy)disiloxane, and a series of isomeric tetrapentoxysilanes. The presence of 1020 steel, titanium, and copper during oxidation did not produce large changes in rate as compared to the rate for the test material alone.

The hydrolysis of tetraaryloxy- and alkoxysilanes apparently proceeds through a stepwise degradation. The rate of hydrolysis is affected largely by structure, being very rapid for tetraphenoxysilane, less rapid for tetra(2-ethylhexoxy)silane, and slow for branched tetrapentoxysilanes.

Pyrolysis is probably the least important factor in the degradation of the tetraaryloxy or alkoxysilanes. No significant degradation was observed with tetraphenoxysilane while tetra(2-ethylhexoxy)silane was degraded only slightly by thermal cracking of the 2-ethylhexyl group.

WADC TR 54-576 (Part 1). AD 63268.
EFFECT OF METALS ON LUBRICANTS (PART 1. DESIGN, DEVELOPMENT AND INSTRUMENTATION OF A HIGH TEMPERATURE BATH). Vernon A. Lauer. February 1955. ASTIA Document No. AD 63268. PB 131505. Order from OTS $0.50.

This report is the first of a series of reports concerning the effect of metals on the stability of lubricants at elevated temperatures. In order to accomplish this, equipment capable of attaining these temperatures was required.

This report describes the design, development and instrumentation of a high temperature bath capable of attaining and controlling temperatures up to 700°±1.0°F.

Southwest Research Institute. WADC TR 53-337 (Part 2).

POLYNUCLEAR AROMATIC COMPOUNDS FOR HIGH TEMPERATURE LUBRICANTS. Charles F. Raley, Jr. AF 33(616)-276. February 1955. ASTIA Document No. AD 61592. PB 136229. Order from LC, Mi $3.60, Ph $9.30.

The properties of triaryl esters of phosphoric acid were studied with regard to their suitability as high-temperature lubricants and related materials. It was found that naphthyl derivatives did not have sufficient thermal stability to compensate for the unwanted increase in viscosity and melting point. The 4-biphenylyl group possessed all the advantages of the naphthyl group with much better thermal stability. One particular compound prepared, bis(o-chlorophenyl) 4-biphenylyl phosphate had a thermal stability of over 510°C (950°F), by far the highest stability of any compound prepared under this contract. It was found that the chlorophenyl phosphates possessed outstanding thermal stability and seemed to be the most promising type of compound for further development. It was found possible to vary considerably the physical properties of a compound by incorporating three different groups. Finally, the pronounced volatilizing and viscosity-reducing effect of the trifluoromethylphenyl group was observed in several compounds which contained one or more of these groups. It was also found possible to compile a table of approximate thermal decomposition temperatures of numerous aromatic groups.

WADC TR 54-418.

PROCEDURE FOR DETERMINING VAPOR PRESSURES OF MATERIALS OF LOW VOLATILITY. O. M. Ballentine. February 1955. ASTIA Document No. AD 66390. PB 131489. Order from OTS $0.75.

An apparatus has been developed for determining absolute vapor pressures of both liquids and solids that exhibit low volatility characteristics. This method has the following advantages: obtaining vapor pressures up to 1000°F, requiring a minimum of operator's time (approximately one (1) hour), relative simplicity and high degree of accuracy in final results. The method employs Knudsen's equation, based on the kinetic theory of gases, in which the weight loss of material per unit time is proportional to the vapor pressure of the material.

Standard Oil Company (Indiana). WADC TR 53-83 (Part 2).

DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASES. Edward A. Swakon, Cecil G. Brannen. AF 33(038)-23687. March 1955.

In the work directed toward the development of an aircraft grease suitable for use over the temperature range of -65° to 450°F, emphasis has been placed on the development of thickeners for silicone fluid and on the evaluation of greases at high temperatures. Studies were made on silicone greases thickened with substituted ureas and other materials which might be expected to produce thermally stable greases. A new class of non-soap, organic thickening agents, called arylureas, has been developed. Greases made with arylureas are extremely heat stable, are easily made in conventional grease equipment, exhibit good rheological properties, and have out-performed all other greases in the bearing test at 450°F. The lowest temperature at which these greases meet the values given in the requirements is around -40°F. Twenty-six of thirty tests on eleven compositions made with DC-550 Silicone Fluid and arylureas ran between 350 and 1170 hours. On two greases designated MLG-9300 and MLG-9301, ten of fourteen tests ran well over 500 hours. They meet all of the other Air Force requirements except performance in the Navy gear-wear test. Failure in this test is caused by the inherently poor lubrication characteristics of silicone oil and attempts to improve the lubricity by the incorporation of additives have been unsuccessful.

Another arylurea grease made with DC-510 Silicone Fluid closely approached the Air Force requirements. This grease is suitable for use at -90°F. Three bearing tests at 450°F average 270 hours.

Bearing tests on greases comprised of DC-550 Silicone Fluid and the following miscellaneous thickeners-carbon black, copper phthalocyanine, calcium acetate, indigo, and N-benzoyl-p-amino-benzoic acid ran between 20 and 427 hours.

The utility of arylureas is not limited to silicone fluids. Arylurea greases with good rheological properties were made with the following types of fluids: petroleum oils, monoesters, diesters, fluorocarbons, phosphate esters, alkyl silicates and polyalkalene glycols. If high stability of a fluid could be combined with good lubricity, a truly multipurpose grease could be made.

The Pennsylvania State University. WADC TR 55-30 (Part 1).

FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. Merrell R. Fenske. AF 33(038)-18193. March 1955. PB 121508. Order from OTS $3.50.

This report describes the current status of development work on Specification MIL-L-7808 oils and turbo-prop lubricants. Fluids of the PRL 3161 and PRL 3313 types are covered in this discussion. A more complete review of the PRL 3161 type of lubricant is given in Wright Air Development Center TR 53-25 entitled "DEVELOPMENT WORK ON LUBRICANTS FOR AIRCRAFT TURBINE ENGINES".

The viscosity-temperature characteristics of synthetic lubricants are important. On the one hand, a low viscosity at low temperatures is desired to enable low temperature starting, while a high viscosity is desired at high temperatures to provide adequate lubrication, particularly to gears. Linear types of polymeric thickening agents with good shear stability are useful in preparing such lubricants.

A single composition can be formulated to meet both Specification MIL-L-7808 turbine engine lubricant and Specification MIL-L-6387 synthetic hydraulic fluid. Five samples of Specification MIL-L-7808 type fluids have been submitted to the Wright Air Development Center by potential commercial suppliers. Data presented in this report indicate that only one of the five commercial fluids meets the viscosity-temperature requirements of the specification.

Lubricants of the PRL 3313 type containing acidic organo-phosphorus compounds have been investigated. Approximately 15 organo-acid-phosphates and -phosphites have been studied in a PRL 3313 type gear lubricant composition. For the most part, these materials are essentially single chemical compounds. The organo-acid-phosphates, and -phosphites, appear to be considerably more effective than the tri-organo-phosphates and -phosphites in improving the wear and lubrication characteristics of synthetic lubricants. These materials have been evaluated in terms of wear and lubrication, oxidation and corrosion, hydrolysis stability, and their foaming tendencies after mild thin

film (250°F) oxidation and corrosion test.

Certain acid phosphites appear to be competitive with acid phosphates as lubrication additives and, in addition, show better corrosion characteristics and hydrolysis stability. The acid phosphates and phosphites appear to be effective as anti-wear lubrication additives in concentrations below 1.0 weight percent and in many cases, in concentrations of 0.1 weight percent. The triphosphates, on the other hand, are effective in concentrations of 3.0 to 5.0 weight percent in the extreme-pressure range of lubricity.

The results of a cooperative lubrication testing program with lubricants designated as PRL 3161, PRL 3312, and PRL 3313 show good general agreement. The relative rating of these fluids by this Laboratory is confirmed by the cooperative tests at other laboratories.

A comprehensive study of the oxidation and corrosion characteristics of synthetic turbine engine lubricants has been conducted. Emphasis has been placed on the bulk oil and thin film oxidation and corrosion characteristics at 347°F (175°C), the corrosion of lead and lead alloys; the storage stability, and the hydrolysis stability of these synthetic lubricants. Synthetic lubricants of the PRL 3313 and PRL 3161 types show good overall oxidation and corrosion stability at 347°F. These tests include the corrosion of the lead-indium coated bearing surfaces of a Pesco gear pump operating at a test temperature of 350°F. In general, it has been found that large differences exist between the corrosion of pure lead and some lead alloys, such as lead-indium. Indications are that small changes in the neutralization number of some PRL 3313 oils on storage are probably related to the catalytic hydrolysis of the ester base stock.

Foaming in certain of the used samples from turbo-prop engines has been noted. After some research, it has been possible to produce a similar foaming behavior in the laboratory by a 250°F thin film oxidation and corrosion test procedure. Foaming is not a property common to all ester types fluids containing organo-acid-phosphorous compounds as lubrication additives. It is possible to avoid foaming by proper selection of the additives.

Used samples of PRL 3161 and PRL 3313 fluids from turbo-prop engines have been evaluated in terms of viscosity stability, foaming characteristics, wear properties, and oxidation and corrosion stability. With the exception of the foaming properties of used PRL 3313 fluid, all of these lubricants show good service stability.

Analysis of a deposit from the combustion chamber of a turbo-prop engine has been made by this Laboratory. This deposit resulted from the leakage of PRL 3313 fluid into the combustion chamber through a faulty oil seal. There is no evidence that the polymeric thickener, oxidation inhibitor, or phosphate lubrication additives are concentrated in this deposit. The deposit does not differ from that expected from the synthetic ester base stock.

Viscosity-temperature characteristics and oxidation and corrosion stabilities have been evaluated for improved Specification MIL-L-6387 synthetic fluid compositions. The applicability of synthetic fluids and lubricants of Specification MIL-L-7808 and Specification MIL-L-6387 types to use as crankcase lubricants in aircraft piston engines and hydraulic fluids in certain Navy applications is discussed briefly.

A brief comparison is given of some of the various types of synthetic hydraulic fluids and lubricants now under development by the Services.

A trend toward decreased oxidation and corrosion stability since World War II in Specification MIL-O-5606 and Specification AN-O-9 fluids is indicated.

The status of development work on an ethyldibromobensene type of non-inflammable hydraulic fluid is reviewed. Laboratory evaluation of this fluid essentially has been completed. Toxicity studies and tests in a hydraulic "mock up" system are now being conducted by the Wright Air Development Center. Limited evaluations have been completed for a phosphate-base fluid and a silicone-diester-fluoro-carbon fluid.

The quantitative distribution of oxygen in oxidized esters has been measured. A satisfactory oxygen balance has been obtained with these techniques.

Studies have been made on the effect of metal catalysts on the oxidation and corrosion stability of certain phenothiazine-inhibited esters. The effectiveness of phenothiazine in esters and the effect of oxidation and corrosion conditions on the concentration of phenothiazine are reported. A technique is given for measuring quantitatively the concentration of phenothiazine in various esters. There is some indication that diphenyl amine or other materials containing aromatic ring structures may result from the oxidative deterioration of phenothiazine.

A survey has been made of the oxidation and corrosion characteristics at 500°F of several types of esters and other synthetic lubricants. The oxidation stability of di-2-ethylhexyl sebacate at 500°F has been studied in some detail. Little or no induction period has been noted in these tests at 500°F. The variables affecting oxidation rate appear to be quite critical in achieving reproducibility.

Some bulk oil oxidation and corrosion tests have been conducted at 400°F with conventional phenothiazine-inhibited ester type lubricants. The induction period at 400°F, appears to be about one-eighth the induction period at 347°F. This rate of change in induction period with temperature is in good agreement with similar data for mineral oil compositions inhibited with Paranox 441 and meeting Specification MIL-O-5606.

The thermal stability of various types of synthetic lubricants has been evaluated at 500°F. Thermal stability studies of Acryloid solutions have also been conducted. Studies have been completed on the effect of these 500°F temperatures on the oxidation, corrosion, and shear properties of Acryloid polymers. The effect of mechanical shear, oxidation, and corrosion on thermal stability has also been determined. These results indicate that certain Acryloid polymers are satisfactory for use in high temperature (500°F) lubricant compositions.

Lubricant studies on esters in the Shell four-ball wear and extreme-pressure lubricant tester are discussed. These studies include the effect of phosphoric acid and acid phosphates as anti-wear additives. The effectiveness of acid phosphates is compared with the tricresyl phosphate susceptibility of various esters. A study of the relative ease of lubrication of titanium and titanium alloys will be conducted in cooperation with Army Ordnance.

Current and future programs are outlined for tests using the Pesco 3000 psi gear pump, the Vickers 3000 psi piston pump, and the revised PRL high shear viscometer. The latter apparatus measures both permanent and temporary viscosity loss.

The current status of new specification tests suggested for inclusion in synthetic lubricant specifications such as Specification MIL-L-7808 and Specification MIL-L-7499 is discussed. These tests include (1) the thin film oxidation and corrosion test, (2) a wear test in the Shell four-ball wear and extreme-pressure lubricant testers, and (3) a vapor pressure and evaporation test.

This Laboratory will likely also carry out a study of thin film rust and corrosion preventives for Navy Ordnance.

A list is given of the low temperature viscosity standards, synthetic lubricants, and synthetic lubricant components that this Laboratory distributed during the past year.

The Pennsylvania State Univ.
WADC TR 55-30 (Part 2).
FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. Merrell R. Fenske, E. Erwin Klaus. AF 33(038)-18193. March 1955. PB 121509. Order from OTS $4.00.

This report describes work carried out on a continuing project to develop improved fluid and lubricant formulations for use by the Air Forces. During the period covered by this report, the emphasis has been placed on the development of synthetic turbo-jet and turbo-prop lubricants. The current program deals primarily with the effect of phosphorous-containing lubricity additives on an ester-base formulation with established over-all properties. The applicability of this type additive for use in mineral oil formulations has been examined.

Equipment has been designed and constructed for constant temperature use at temperatures in the range of 300° to 800°F. The oxidation behavior of various classes of synthetic fluids and hydrocarbons has been explored at temperatures of 300° to 500°F. Thermal stability of many of the fluids has been determined at temperatures up to 750°F.

Small quantities of dibasic acids have been shown to be effective metal deactivators for controlling metal corrosion in dibasic acid ester-base fluids and lubricants.

A survey of several brominated aromatics has been made to determine the applicability of these materials as base stock constituents for non-inflammable fluids.

The Pennsylvania State Univ.
WADC TR 55-30 (Part 3).
FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. E. Erwin Klaus, Merrell R. Fenske. AF 33(038)-18193. March 1955. PB 121510. Order from OTS $5.50.

This report describes work carried out on a continuing project to develop improved fluids, lubricants, and fuels for use by the Air Force. The current report deals primarily with the development of high temperature hydraulic fluids and jet engine lubricants and the study of dirtiness characteristics of jet fuel at elevated temperatures.

The hydraulic fluid development goal is a fluid suitable for use at 700°F for a limited time in a sealed hydraulic unit. The properties of high quality examples of the following chemical classes have been measured: silicones, silicate esters, diesters, pentaery-thritol esters, silicone-ester blends, mineral oils, synthetic hydrocarbons, polyglycol ethers, chlorinated aromatic hydrocarbons, and aryl phosphates. The properties measured include: viscosities from 0° to+700°F, thermal stabilities from 500° to 750°F, oxidation and corrosion stability at 347° and 500°F, and operation in a hydraulic pump (vane type) at 500° to 600°F.

The jet engine lubricant goal is a fluid suitable for use for extended periods at 500° to 600°F bulk oil temperature. The same general chemical classes enumerated for hydraulic fluids have been included in the engine oil studies. Particular emphasis in the jet engine oil studies has been placed on lubricity studies and severe oxidation and corrosion evaluations at 500°F.

Jet fuel dirtiness has been studied from two basic approaches. An attempt has been made to remove the dirty components from a fuel by physical separation techniques. Secondly, a laboratory method of reproducing high temperature dirtiness in jet fuels is being studied.

Coupled with the above programs, some miscellaneous investigations of hydraulic fluid and lubricant problems have been conducted at the request of the Services. In addition, viscosity standards, experimental fluid and lubricant formulations, and reports prepared by this Laboratory have been distributed at the request of industry and the Services.

University of Virginia. WADC TR 53-293 (Part 5).
HIGH-TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS: PART 5 - EVALUATION OF ADDITIVES, SYNTHESIS OF NEW COMPOUNDS, AND MECHANISM STUDIES. James W. Cole, Jr., Gordon P. Brown, A. Chandler Schmalz. AF 33(038)-22947. March 1955. PB 121081. Order from OTS $5.50.

The oxidation characteristics have been determined of various blends of selected additives with fluids of the diester, silicate, silicone and tri-(ortho-chlorophenyl) phosphate types in the absence and presence of aluminum, copper, magnesium, silver, cold-rolled steel and titanium. The temperature range was 200-250°C (400-482°F) with the majority of the runs at 240°C (465°F). A few runs with DC Silicone 550 were conducted at 600° and 700°F. Two experimental methods were employed. One was the previously described standard oxidation-corrosion method in which the fluids were heated under reflux with dry air bubbling through for specified periods of time. The other involves the same test cell, modified for removing small samples of the heated fluid at intervals. Many additives were compared with phenothiazine in di(2-ethylhexyl) sebacate and with phenyl-alpha-naphthylamine in mixed C_6-C_8 silicates. A number showed promise and recommendations are made for further study. At 400°F, the time needed to bring the oxidizing mixture to a definite break in the viscosity was roughly proportional to the molar equivalent concentrations. As the temperature was raised the effect of the additives became less pronounced until at 482°F the effect was only a slight decrease in the overall rate of oxidation. The behavior of the test metals was not always uniform but in general it was shown that magnesium was most active followed by copper and silver. A spectacular formation of Cu_2Se and Ag_2Se was observed with dilauryl selenide which was otherwise an effective antioxidant. Titanium, aluminum and cold-rolled steel were mostly without well defined effects.

A blend of disiloxane-silicone (MLO 8200 type) was examined to determine its oxidation pattern in the range 400-482°F. Oxidation promoted a decrease of about 30% in viscosity which was not dramatically affected by additives at the upper temperatures. After a short intermediate period of little change, the viscosity of the uninhibited fluid increased rapidly to about 50% of the original in 72 hours at 240°C. The presence of 2% dioctyl diphenylamine held the viscosity unchanged from the initial decrease for about 90 hours after which there was a steady increase in viscosity. Several additives prolonged the intermediate period of little viscosity change, but none was superior to diphenylamine. The silicones were only slowly oxidized at 465°F, but at 600°F the oxidation was rapid; in 24 hours the viscosity increased more than 100%. A few runs with tri-(o-chlorophenyl) phosphate at 240°C (465°F) indicated this oil in the absence of inhibitors to have the greatest oxidative stability of any fluid examined under this contract. However, in the presence of copper, oxidation was rapid with considerable reaction with the metal to form cuprous chloride. A mixture of the fluid containing phenothiazine and sebacic acid in the presence of copper oxidized even more rapidly.

Considerable attention was given to showing that the products of thermal and photo-oxidation of phenothiazine are identical and to relating their behavior to possible mechanisms of antioxidant activity. It is postulated that phenothiazine is active as an antioxidant, not so much in itself, as by being oxidized to 3-hydroxyphenothiazine which reduces hydroxyl free radicals to water. The antioxidant is thus converted to a semiquinone or other semi-stable free radical, which can then undergo further oxidation or be reduced back to the antioxidant. The postulated mechanism also affords an explanation of synergistic action and the fact that the same oxidation products are formed during the slow induction period as during the more rapid uninhibited oxidation.

The results of an extensive synthetic program on

phenothiazine derivatives are reported. Attention was given to synthesis of derivatives in the three, four and ten positions and to synthesis of derivatives of 7-benzo(c) phenothiazine. Where sufficient quantities were prepared they were evaluated as antioxidants. Several were highly effective especially the nitrogen substituted alkyl derivates.

WADC TR 54-417.
METHOD OF DETERMINING THERMAL STABILITY OF SYNTHETIC OILS. O. M. Ballentine. March 1955. ASTIA Document No. AD 73441. PB 128429. Order from LC, Mi $2.40, Ph $3.30.

An apparatus has been developed for determining thermal stability of synthetic oils. Thermal decomposition temperatures were determined by plotting vapor pressure over a wide temperature range as log P vs. $\frac{1}{T}$. The point at which the curve deviates from a straight line relationship will be the point at which thermal decomposition occurs. The following advantages are gained through this method of determining thermal decomposition points: the utilization of small size samples (2.0 to 3.0 grams), a wide temperature of operation (up to 1500°F), a high degree of accuracy in the final results, simplicity of operation (requiring one operator), and increased rapidity of operation.

Denver Research Institute. WADC TR 54-464.
DEVELOPMENT OF SCHEMATIC ANALYTICAL PROCEDURES FOR SYNTHETIC LUBRICANTS AND THEIR ADDITIVES. J. J. E. Schmidt, F. S. Bonomo. AF 33(616)2204. April 1955. PB 111856. Order from LC, Mi $9.90, Ph $34.80.

General properties and methods for the identification, determination, and separation of components of synthetic greases and synthetic lubricants are presented and discussed.

Included in these components are inorganic and organic gelling agents, soap and urea type thickeners, and such additives as antioxidants. A schematic analytical procedure for the separation of grease components is presented along with the application of paper chromatographic methods for the identification and separation of antioxidants in greases and synthetic lubricants.

Initial investigations for the separation and identification of dibasic acid esters in synthetic lubricants are presented.

WADC TR 54-580.
EVALUATION OF ORGANIC FLUORINE COMPOUNDS FOR USE IN MILITARY AIRCRAFT. Harold Rosenberg, J. C. Mosteller. April 1955. PB 111983. Order from OTS $0.75.

The evaluation of fluorine compounds for use in military aircraft is one phase of a general Air Force program which has been established for the purpose of obtaining materials of unusual properties and capable of advancing the design of new high-speed aircraft and guided missiles. The desirable properties of fluorine-containing organic compounds include wide liquid range, unusual chemical stability, good electrical conduction, desirable heat transfer characteristics and decreased flammability. Fluorine compounds have, accordingly, been studied for use as fire-extinguishing agents, acid-resistant coatings and greases, non-flammable hydraulic fluids, elastomers, electronic equipment and fungicides.

For extinguishing aircraft fires, particularly those involving rocket propellant mixtures, dibromodifluoromethane and bromotrifluormethane have been shown to be quite effective. Polymers of monochlorotrifluorethylene and tetrafluoroethylene have been employed successfully as seals, gaskets and in lubricants in aircraft reaction motors and nitric acid-refueling trailers. Various fluorocarbons, especially chlorofluoroheptenes, have been evaluated as "snuffer" and anti-wear additives for hydraulic fluids. Amongst the "fluorelastomeric" materials, the poly-1, 1-dihydroperfluoroalkyl acrylates, $[-CH_2CH(COOCH_2R)-]_n$, have shown the most promise from the standpoint of solvent resistance and low-temperature flexibility. Polyfluoroethylene derivatives have found use in electronic components of military aircraft. Recently, fluorodinitrobenzenes have been shown to be effective agents for protecting fabrics against fungi attack.

Iowa State College. WADC TR 53-426 (Part 2).
ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS. Henry Gilman, Richard D. Gorsich. AF 33 (616)94. April 1955. PB 111889. Order from OTS $3.50.

An extension has been made of the preliminary screening of organo-metallic and organo-metalloidal compounds for thermal stabilities in connection with applications involving high-temperature lubricants and related materials. On the basis of current studies it appears not desirable to give serious consideration to organo-metallic compounds of tin and of lead. Organosilicon compounds of various types are distinctly promising, and the studies now reported and in progress indicate some of the more promising radicals or groups that warrant incorporation in the organo-metallic and organo-metalloidal types. The effects of some of these groups are considered in the discussions which follow the experimental sections.

It is desirable to extend the organogermanium studies, for some of these may enjoy an advantage over corresponding organosilicon compounds in a property like higher thermal stability. Interesting properties have been observed with amines and with some organic phosphorous compounds (of the R_3PO type).

In the experimental part there is included a table which outlines the preliminary screening of one hundred and thirty-seven compounds.

Southwest Research Institute. WADC TR 55-90.
LITERATURE SURVEY OF LOW MOLECULAR WEIGHT POLYNUCLEAR AROMATIC COMPOUNDS. Charles F. Raley, Jr. AF 33(616)-276. May 1955. PB 121664. Order from OTS $4.75.

A literature search was carried out covering the field of low molecular weight polynuclear aromatic compounds with the object of determining the usefulness of such compounds as high-temperature lubricants. The highest literature boiling point, calculated atmospheric boiling point, melting point, and literature reference are given. Recommendations are made as to the compounds or types of compounds which appear promising as high-temperature lubricants.

Continental Oil Company. WADC TR 55-190.
DEVELOPMENT AND EVALUATION OF A GREASE FOR -100°F to +350°F. Ernest W. Nelson, C. Richard Bergen, Billy P. Scott, Warren W. Woods, Richard M. Tillman. AF 33(616)-2364. July 1955.

This report describes the history and present status of the development of a general purpose grease to be operational over a -100°F to +350°F temperature range. Experimental work has been conducted during the past 12 months by Continental Oil Company under AF 33(616)-2364 and is to be continued under a nine-month extension contract.

These studies have been limited to development of greases thickened by colloidal calcium carbonate coated

with calcium salts of aliphatic dicarboxylic acids. Using diester vehicles, it now appears probable that a balanced formulation can be made which will meet specifications for consistency, bleeding, work stability, dropping point, evaporation loss, low temperature torque, and water resistance. Some revision in composition for present greases will be required since incorporation of work stability and water resistance additives has been found to induce changes in consistency and bleeding which must be offset by slight changes in the thickener. In addition, an oxidation inhibitor must be incorporated prior to starting prolonged bearing tests at high temperatures.

Standard Oil Company (Indiana).
WADC TR 53-83 Part 3.
DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASES. Edward A. Swakon. AF 33(038)-23687. July 1955.

In the work directed toward the development of an aircraft grease suitable for use over the temperature range from -65 to 450°F and higher, emphasis was placed on the development and evaluation of arylurea greases made with the best available fluids. The chief criteria of laboratory evaluation were performance in the ABEC-NLGI Bearing Tester and the Navy Gear-Wear Tester. Sixty-seven bearing tests at 350°, 400°, 450°, and 500°F were run on thirty-two compositions. Studies were made of the effect on gear-wear tests of base fluid and thickener, of anti-wear additives in silicone greases, of diester-silicone and of Aroclor-silicone blends, and of composition of brass. Arylurea-silicone greases come closest to meeting the proposed temperature requirements, but have limited utility because of the inherently poor lubrication characteristics of silicone fluids. Six of seven arylureanon-silicone greases tested have out-performed a MIL-G-3278 grease in bearing performance at 350°F. The limiting component in the development of a grease with good lubricity for the temperature range from -65° to 450°F and above is a suitable oil.

Shell Development Company.
WADC TR 55-102 Part 1.
GREASE LUBRICATION OF HIGH SPEED ANTI-FRICTION BEARINGS. John B. Accinelli, Charles R. Greene. AF 33(616)-2443. July 1955. ASTIA Document No. AD 64341. PB 138656. Order from LC, Mi $3.90 Ph $10.80.

A study of the failure mechanisms of grease lubricated antifriction bearings operating in the DN range from 1.0×10^6 to 2.0×10^6 was the main object of this project. All test work was conducted at the Shell Development Company using a rig designed by this company. After necessary rig development work and bearing selection tests, preliminary work on the effect of various grease and operational variables on the performance of ball bearings operating up to DN values of 1.5×10^6 was initiated. The problem seems to be one of maintaining an effective lubricant film on the working surfaces of the bearing under the very strong stripping action of centrifugal force, high sliding velocity and very high rate of shear. The grease property apparently most critical to lubrication at high speeds is viscosity of the oil. Operation at high (300°F) temperatures also seems to aid lubrication.

Peninsular Chem Research, Inc.
WADC TR 55-187.
INVESTIGATION INTO THE USE OF HETEROCYCLIC COMPOUNDS AS LUBRICANT ADDITIVES. George B. Butler, O. Lee Gordon, Louis A. Haynes. AF 33(616)-2391. August 1955. ASTIA Document No. AD 93162. PB 132107. Order from LC, Mi $3.90, Ph $10.80.

A literature survey and synthesis program were carried out in order to prepare various nitrogen and sulfur, selenium, or oxygen-containing heterocycles for evaluation as antioxidant, anti-wear and extreme pressure additives for lubricants, hydraulic fluids and greases. In addition, a number of organoselenium compounds of the selenide and diselenide types were synthesized, primarily for evaluation as high-temperature oxidation inhibitors. Solubilities of the prepared compounds in various referenced fluids were determined.

WADC TN 55-372.
A SURVEY OF THE LITERATURE ON THE LUBRICATION OF TITANIUM. Robert Benzing, 1/Lt., USAF, Ray E. Bryant, 1/Lt., USAF. August 1955.

A survey of the literature on the lubrication of titanium was made. The work studied is broken down into three main groups: Frictional and Lubricating Properties, Surface Coatings and Treatments, and Machining Practices. A brief discussion summarizing the literature is given which outlines the present status of the work with suggestions for future practices.

Lehigh Univ. WADC TR 55-240.
BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP LUBRICATING GREASES. G. J. Young, J. J. Chessick, David Chu, A. Ulincy, E. Borger, A. C. Zettlemoyer. AF 33(616)-2440. December 1955. ASTIA Document No. AD 84149. PB 129318. Order from LC, Mi $3.00, Ph $6.30.

Pertinent physical properties of several oils and surface characteristics of various types of thickening agents have been determined in order to index the nature and extent of the oil-thickener interface in non-soap grease systems. These properties of the oils and thickening agents are correlated with the behavior of grease systems formulated from them. In addition, modifications of the oil-thickener interfacial region by traces of water and by polar organic additives have been studied. Ramifications of the alteration of this interfacial zone on gel structure and stability of grease systems are discussed.

WADC TN 55-554.
INVESTIGATION OF LITHIUM DIHYDROXY AND POLYHYDROXY STEARATES AS GREASE THICKENERS. Robert J. Benzing, 1/Lt., USAF. January 1956.

A dihydroxy and a polyhydroxy stearic acid supplied by the Baker Castor Oil Company were evaluated as potential raw materials in the reproduction of thickening agents for lithium soap greases. The greases made with these soaps are highly thixotropic, but the greases made from the dihydroxy stearic acid are better in this respect than the greases made with the polyhydroxy stearic acid.

Shell Development Company.
WADC TR 55-102 Part 2.
GREASE LUBRICATION OF HIGH SPEED ANTI-FRICTION BEARINGS. John B. Accinelli, Charles R. Greene. AF 33(616)-2443. February 1956. ASTIA Document No. AD 80397. PB 133015. Order from LC, Mi $3.60, Ph $9.30.

The work reported is a continuation of the study of grease lubrication of ball bearings operating at very high DN values (1.0×10^6 to 2.0×10^6). The work was performed at the Shell Development Company on two high speed rigs designed and built for the Air Force by this company. These rigs, utilizing 20 mm ball bearings, required considerable development work in order to obtain satisfactory

operation at the desired DN values. Before work on greases was initiated each rig was calibrated with oil-air mist lubrication by running continuously for 100 hours at 1.2×10^6 DN in duplicate tests with two oils, a mineral oil of SAE grade 1010 (MIL-O-6081) and a diester synthetic oil (MIL-L-6085A). The rigs are now ready for the grease studies. In the course of these calibration runs it was found that slight dynamic unbalance of the rotating components has a very deleterious effect on bearing operation, and that for successful operation, in the very high range of DN, micro balancing of the rotor is required. The very high centrifugal forces encountered in very high speed operation seem to cause continuous stripping of the lubricant film, and this action requires that lubricant be continually applied to the bearing at a fairly high rate of flow in order to obtain prolonged operation. This factor may limit the applicability of greases as lubricants for very high speed ball bearing application.

The laboratory investigation of grease properties in a force field obtained by centrifugation has been continued. It has been shown that bulk greases will not adhere to metal surfaces subjected to the unimpeded stripping action of centrifugal forces comparable to those encountered in some areas of the operating high speed bearing. Oil loss from grease structures, through the shearing and crushing action of centrifugal force, has been shown to vary with grease type and composition. The results obtained have permitted some qualitative conclusions on the way the grease properties studied may affect bearing operation.

Peninsular Chem Research, Inc.
WADC TR 55-187 Part 2.
INVESTIGATION INTO THE USE OF HETEROCYCLIC COMPOUNDS AS LUBRICANT ADDITIVES. George B. Butler, O. Lee Gordon, Louis A. Haynes. AF 33(616)-2391. February 1956. ASTIA Document No. AD 93138. PB 131223. Order from OTS $0.50.

A literature survey and synthesis program were carried out in order to prepare various nitrogen and sulfur-selenium-, or oxygen-containing heterocycles for evaluation as antioxidant, anti-wear and extreme pressure additives for lubricants, hydraulic fluids and greases. In addition, a number of organoselenium compounds of the selenide and diselenide types were synthesized, primarily for evaluation as high-temperature oxidation inhibitors. Solubilities of the prepared compounds in various referenced fluids were determined.

WADC TR 55-449 Part 2.
MICRO LUBRICANT TEST METHODS Part 2 Swelling of Synthetic Rubber Color of Lubricating Oil and Petrolatum Hydrolytic Stability. John B. Christian, Harry M. Schiefer, 1/Lt., USAF. March 1956. PB 121386. Order from OTS $0.75.

This report introduces micro methods for the determination of the swelling of synthetic rubbers in aircraft greases and hydraulic oils, and the determination of color of lubricating oil and petroleum through the use of the A.S.T.M. Union Colorimeter, and the determination of hydrolytic stability of aircraft oils.

These methods were arrived at through the comparison of modified small scale tests with the full scale tests.

Continental Oil Company. WADC TR 55-190 Part 2.
DEVELOPMENT AND EVALUATION OF A GREASE FOR - 100 to+ 350°F. Ernest W. Nelson, Warren W. Woods, C. Richard Bergen, William P. Scott, Richard M. Tillman. AF 33(616)-2364. April 1956.

The thickener for this wide temperature range grease development has been limited to a colloidal calcium carbonate coated with the calcium salts of aliphatic dicarboxylic acids. The aliphatic diesters were studied as possible base fluids. Although promising in many respects, their use is precluded because of high evaporation rates and lack of a sufficiently active oxidation inhibitor at 350°F. Greases prepared with certain hexa alkoxy disiloxanes appear promising. Formulations using the base oil hexa (2-ethyl hexoxy) disiloxane very nearly meet or surpass target requirements, the lone exception being high temperature performance. Here a maximum of 350 hours has already been achieved. Formulation changes designed to improve performance of such greases have been developed, but testing has been hampered because of the short supply of the base fluid.

University of Virginia. WADC TR 53-293 Pt 6.
HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS. Part 6. Evaluation of Additives Mechanism and Radiochemical Studies. James W. Cole, Jr., Donald R. Campbell. AF 33(038)-22947. April 1956.

The object of this work is the evaluation of promising additives and the determination of their mechanism of action as anti-oxidants in non-petroleum base hydraulic and lubricating fluids at temperatures in the range 400° to 700°F. This report describes the effects of structure and concentration of the additives and the effects of test specimens of copper, silver, aluminum, titanium, magnesium, and steel in diester, silicate, disiloxane-silicone, silicone, and phosphate base fluids. Mechanisms of oxidation and anti-oxidant activity have been postulated with radioactive sulphur being used as a tracer in some of these quantitative studies.

Phenothiazine and its derivatives showed some effectiveness as anti-oxidants in a representative diester sebacate fluid over the temperature range 400° to 500°F. Several other additives, including aryl amines and selenium derivatives, appear promising in the higher temperature range. Magnesium, copper and iron were most severely attacked with titanium, aluminum and silver generally being unaffected.

No additive system appears to meet specifications for use in mixed C_6-C_8 silicates, blended disiloxane-silicone, and DC silicone 550 although some had anti-oxidant activity.

Additives showing promise for use in a G. E. silicone in the range 500° to 700°F are p,p'-dioctyldiphenylamine, phenyl-alpha-naphthylamine and N,N'-di-2-naphthyl-p-phenylenediamine.

Mechanism studies were performed in a special apparatus using small amounts of fluid under controlled conditions of flow, temperature and pressure. Radiochemical studies of S^{35} phenothiazine in di-(2-ethylhexyl) sebecate show the rate of formation of insoluble matter and indicate the relative contribution of the fluid and additive in forming sludges and in attacking metal surfaces.

WADC TR-55-449 Pt 1.
MICRO LUBRICANT TEST METHODS VISCOSITY-NEUTRALIZATION NUMBER. John B. Christian, Arthur L. Miller, Vernon A. Lauer, Harry M. Schiefer, 1/Lt. April 1956. PB 121355. Order from OTS $0.50.

This study was initiated to develop miniaturized tests for petroleum, petroleum products, and related materials. Procedures for determining neutralization number, and viscosity are described and their correlation with macro tests provided.

The small scale test techniques discussed herein are considered as satisfactory as their full scale counterpart for their intended purpose.

WADC TR 55-475.
FOAMING CHARACTERISTICS OF AIRCRAFT OILS. George Baum. May 1956. PB 121445. Order from OTS $0.50.

The foaming characteristics of several lubricating oils were studied in connection with power plant and propeller installation difficulties ascribed to oil foaming. Wide variations in quantity and collapse time of foam formed during aeration were observed in laboratory tests. Chemical additives of silicone and sorbitan type proved effective as heave petroleum oil defoamers. No really effective defoamers for light oils were noted. A laboratory test which gives a sharper delineation between a group of light oils than the present ASTM test has been studied and may with sufficient refinement provide a better index of foaming tendency of oils than the present test.

WADC TR 55-449 Pt 3.
MICRO LUBRICANT TEST METHODS Part 3. Corrosion and Oxidation-Separation of Oil From Lubricating Greases--Corrosiveness of Greases and Oils. John B. Christian. May 1956. PB 121443. Order from OTS $0.75.

This report contains the following test methods: Corrosion and Oxidation, Separation of Oil from Lubricating Greases, and Corrosiveness of Greases and Oils. These test methods are designed for the micro analysis of petroleum, petroleum products, and related materials. They were arrived at through the modification of existing test procedures which require greater quantities of test samples.

Southwest Research Institute. WADC TR 53-337 Pt 3.
POLYNUCLEAR AROMATIC COMPOUNDS FOR HIGH TEMPERATURE LUBRICANTS. Charles F. Raley, Jr. AF 33(616)-276. May 1956. PB 132124. Order from LC, Mi $3.60, Ph $9.30.

The program of investigation of the class of aryl phosphate esters was continued. These materials possessed the most promising high-temperature properties of those examined. The aryl groups found to be the most thermally stable were phenyl, fluoro- and chlorophenyl, m-trifluoro-methylphenyl, 4-biphenylyl, dimethylphenyl, 5-indanyl and naphthyl. The simple phosphate structure was found to be quite stable. Pyrophosphates had the drawbacks of mediocre thermal and hydrolytic stability, although apparently possessing good oxidative stability.

Compositions prepared by reacting two or three different phenols with the proper amount of $POCl_3$ were also investigated. These compositions exhibited markedly lessened tendency to crystallization. Many of the compositions possessed properties almost identical to pure compounds having equivalent phenol proportions, and had the advantage of greater ease of preparation.

A total of nine fluids were prepared which possessed a liquid range of at least -20 to 800°F. Many of the compounds and compositions prepared, including others not meeting this liquid range, are considered to have potential use value.

Several generalizations were drawn from the results of the over-all program. It was found that thermal and oxidative stability do not necessarily go together. Strongly polar substituents on the aromatic ring adversely affect hydrolytic stability. Two or more polar groups on a monocyclic ring adversely affect the thermal stability; one polar group on a bicyclic ring adversely affects the thermal stability. A marked effect on such physical properties as boiling point, melting point, viscosity, etc., is observed, depending on the nature of the substituent groups. The more compact molecules generally have the greatest tendency to crystallize, with boiling point increasing with the molecular weight.

WADC TR 56-175.
STARTING AND RUNNING LOW TEMPERATURE TORQUES. Bernard Rubin. May 1956. PB 121440. Order from OTS $0.75.

Low temperature (-65°F) starting and running torque data for greases are reported in size 104, 204, and 306 bearings using a procedure under investigation by the ASTM Technical Committee G on Lubricating Grease. The investigation with the 204 bearing was a part of the cooperative effort with other laboratories within the ASTM group working towards the standardization of a low temperature torque procedure. Volume of grease in the bearing could not be directly correlated with starting and running torque.

Recommendations are made for improvement of test procedure for specification purposes.

Denver Research Institute. WADC TR 54-464 Pt 2.
DEVELOPMENT OF SCHEMATIC ANALYTICAL PROCEDURES FOR SYNTHETIC LUBRICANTS AND THEIR ADDITIVES. Josef J. E. Schmidt, John R. Hobaugh, Francis S. Bonomo. AF 33(616)-2204. June 1956.

General properties and methods for the identification and determination of components of synthetic lubricants and base-oils and their additives are presented and discussed.

Included in these compounds are dibasic acid esters, silicate esters, and silicone oils, and such additives as antioxidants. Analytical procedures employing column and paper partition chromatographic techniques are presented for the identification and separation of different groups of components, such as dibasic acid esters from silicate esters and silicone oils as well as different dibasic esters from each other, along with methods for the paper chromatographic identification and separation of antioxidants in synthetic lubricants.

WADC TR 56-177.
POLYARYLUREA GREASES. D. T. Kjerland, 2/Lt. July 1956. ASTIA Document No. AD 97138. PB 121531.

Arylurea compounds prepared from aromatic isocyanates or aromatic diisocyanates and aromatic amine or combination of aromatic amines have in the last three years been shown to be good grease thickening agents having high temperature (500°F) stability. In this report, preliminary data are presented on polyarylurea thickening agents prepared from aromatic diisocyanates and aromatic diamines.

Initial data show that polyarylureas have much better thickening ability than the simple arylureas with equivalent heat stability.

Petroleum Refining Laboratory, Pennsylvania State University. WADC TR 56-254.
MINERAL OILS AS HIGH TEMPERATURE FLUIDS AND LUBRICANTS. E. Erwin Klaus, Merrell R. Fenske. AF 33(616)-2851. August 1956. ASTIA Document No. AD 97184.

In a survey of fluids and lubricants for use at 400° to 700°F., certain types of mineral oils were found to be among the most promising materials studied. While synthetic lubricants have better performance

characteristics than most mineral oils and hydrocarbons in the moderate temperature range (200° to 400°F), they do not possess this margin of superiority in the high temperature range (400° to 700°F). Certain synthetics have better viscosity-volatility and viscosity-temperature characteristics than hydrocarbons.

Some mineral oils and diesters of the Spec. MIL-L-7808 type have good lubricity properties at high temperatures. Using a hydraulic pump and a laboratory wear tester, it has been found that there are lubricity additives that are still effective at 500° to 700°F in mineral oils and diesters.

Mineral oils and certain types of hydrocarbons have better thermal stability and oxygen tolerance than many synthetics at 600° to 700°F. The concept of oxidation stability, or inertness toward oxygen, is not normally applicable at these temperatures. Oxidation rate and oxygen tolerance are better criteria for judging oxidative deterioration at 500° to 700°F.

It appears that some additives and additional refining techniques (such as hydrogenation) will be beneficial in improving the oxidation behavior of mineral oils and certain other hydrocarbons at moderate and high temperatures.

The thermal and oxidative components of high temperature coking on metal surfaces are demonstrated in a controlled atmosphere panel coker. It may be necessary to control the atmosphere in engine parts requiring lubrication at 500° to 700°F because of the possibility of spontaneous ignition of the lubricant or of fragments from its decomposition.

In a lubrication system where the oil is used for a limited time and then discarded (perhaps to the burners) it is necessary to know the effect of contaminating the fresh oil with oxidized or deteriorated oil. Data have been obtained indicating that this effect need not be serious with esters and mineral oils at 500°F.

Iowa State College. WADC TR 53-426 Pt 3. ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS. Henry Gilman, Richard D. Gorsich. AF 33(616)-94. August 1956. ASTIA Document No. AD 97236.

The purpose of this investigation is to synthesize and evaluate organometallic and organometalloidal compounds as base stock materials for high-temperature lubricants and hydraulic fluids. Preliminary screening has been extended and has shown that some of the more promising types are cyclic in nature where silicon or germanium is part of the heterocycle. In addition, the biphenyl group, the phenoxyaryl groups, the long-chained alkyl groups and the haloaryl groups are very promising.

New syntheses have been developed for a number of these types, and these are described in the experimental section. There is also included, in the experimental part, a table which outlines the preliminary screening of forty-one compounds.

Lehigh University. WADC TR 55-240 Pt II. BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP LUBRICATING GREASES. John J. Chessick, Albert C. Zettlemoyer. AF 33(616)-2440. September 1956. ASTIA Document No. AD 97282.

Physical properties of several oils and surface characteristics of a variety of thickening agents were measured in order to index the nature and extent of the vehicle-thickener interface in non-soap grease dispersions. These properties of the oils and solids were correlated with the behavior of grease systems formulated from them. Modification of the interfacial region by water and polar organic additives was also investigated.

The combined results of these studies have led to some interesting conclusions regarding the mechanism of flocculation with consequent gel formation of polar and nonpolar solids dispersed in grease vehicles. The relatively low area polar solids used in this work require the presence of small amounts of water in the grease system for flocculation to occur in nonpolar vehicles. These polar solids, at least in the quantities used in this work, would not be effective thickeners in pure liquids or in liquids which dissolve or are miscible with large quantities of water. On the other hand, the nonpolar thickeners when dispersed in a vehicle do not need water to flocculate into a gel structure. These nonpolar materials were shown to be effective thickeners in both polar and nonpolar liquids. A proposed mechanism to explain the flocculation of these solids is based on the reduction in free surface energy brought about by the flocculation.

In addition, the mechanism of additive action has been explained qualitatively in terms of the mechanism of flocculation of these solids in the simple grease systems.

A variety of experimental approaches were used to gain a better understanding of grease structure. These included electrokinetic and conductance measurements and low angle X-ray scattering. X-ray scattering techniques have proved to be the most rewarding; indeed, marked diffraction maxima have been found corresponding to spacings of the order of 100 to 2000 A° units for copper phthalocyanine thickened greases.

Standard Oil Company (Indiana). WADC TR 53-83 Pt 4. DEVEOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASES. Edward A. Swakon. AF 33(038)-23687. September 1956. ASTIA Document No. AD 97313.

In the work directed toward the development of an aircraft grease suitable for use over the temperature range from -65° to 450°F and higher, emphasis was placed on the development and evaluation of arylurea greases made with the best available fluids and the development of thickeners for use as high as 700°F. The chief criteria of laboratory evaluation were performance in the ABEC-NLGI Bearing Tester, the Navy Gear-Wear Tester, low-temperature torque test, apparent viscosity and linear screw-and-nut actuator.

The grease selected to replace MLG-9305 consists of DC 550 Silicone Fluid thickened with para-tolyl and para-chlorophenylurea derivatives of bitolylenediisocyanate. It was designated MLG-9349. All the thickener ingredients are commercially available and the manufacturing procedure was greatly simplified by this choice of ingredients. The corresponding grease made with DC XF 258 Silicone Fluid was designated MLG-9360 and comes closest to meeting the proposed requirements for a grease for the temperature range from -65° to 450°F. Arylurea grease of Hercoflex 600 (polyester) is the best choice for a grease for use from -65° to 350°F with good lubricity; bearing performance at 350°F averaged about eight times longer than that obtained with MIL-G-3278 greases. Pteridine derivatives were the most promising compound uncovered during this period for thickeners for use in greases in excess of 450°F and as high as 700°F. An anti-oxidant, MLO-55-535, was at least as good as phenothiazine or dilauryl selenide in ester greases. An anti-wear agent, MLO-55-499, in silicone greases was effective in reducing wear in the Navy gear-wear test, but not enough to pass the requirements under the ten-pound-load conditions. The fluid continues to be the limiting component in the development of a grease with good lubricity for the temperature range from -65° to 450°F and above.

WADC TR 53-466 Part II.
EVALUATION OF DRY FILM LUBRICANTS.
Bernard Rubin. September 1956. ASTIA Document No. AD 97318.

A large number of commercial films were evaluated by the previously reported Falex Test procedure and wide variation in wear endurance life noted. The nature of failure pattern and effect of load are noted for some of these films. Certain films showed very good life in the repeatable 30 minute test procedure. Titanium disulfide in a resin binder had poor fraction and endurance properties. Tung-Lube, a proprietary friction reducer, showed some promise and should be evaluated further.

Preliminary data at elevated temperatures indicate that high temperature static soak (to 500°F) and subsequent test at room ambient temperature is much less severe on endurance life than evaluation at 225°F. Endurance life of films on line contact specimens (block and cylinder) is shown to vary inversely with speed and load. However, at light loads sliding velocities of 300 to 400 feet per minute for significantly useful periods of time under unidirectional motion have been observed for a representative dry friction reducing film.

Petroleum Refining Laboratory, Pennsylvania State University. WADC TR 56-224.
HIGH TEMPERATURE LUBRICANT STUDIES. E. Erwin Klaus, Merrell R. Fenske. AF 33(616)-2851. September 1956. ASTIA Document No. AD 97291.

Basic information on the behavior of hydraulic fluids and lubricants is needed at temperatures above 400°F if successful operation of engines and hydraulic systems is to be achieved at high temperatures. It is necessary first to develop reliable test procedures and techniques covering the measurement of viscosity, lubricity, corrosion, and oxidation and thermal stability at temperatures of 500° to 700°F. Several fluids were evaluated in this manner. These fluids are representative of the different chemical classes, such as silicones, silicates, hydrocarbons and mineral oils, diesters, and halogenated aromatic hydrocarbons.

It was found that the property differences between the various types of fluids at 500° to 700°F were much narrower than exist in the 200° to 350°F range. Data are presented showing the inadequacy of extrapolating 200° to 350°F data as a means of predicting performance at 500° to 700°F. The behavior of fluids and lubricants in the 200° to 350°F range has been quite well defined in recent years for the materials used in this study. However, there is a paucity of information at 500° to 700°F. This paper attempts to remedy somewhat this situation.

Stanford Research Institute. WADC TR 54-339.
DETERMINATION OF THE MECHANISM OF THE INCREASE OF VISCOSITY OF ORGANOSILICON COMPOUNDS AT HIGH TEMPERATURES. Edward E. Ryskiewicz. AF 33(616)-168. October 1956. ASTIA Document No. AD 110426. PB 121717.

A study of the oxidation of the tetrapentoxysilanes at 120°C (248°F) has revealed an apparent correlation between oxidation rate and chemical structure.

Antioxidants could not be studied effectively at 120°C (248°F) because of the prolonged protection afforded by all the compounds studied. Ionol in a 0.1% concentration protected tetra (2-pentoxy)silane against oxidation at 120°C (248°F) for 408 hours.

Hydrolysis of the tetrapentoxysilanes was shown to be markedly affected by products which could form during oxidation. An apparent relation between chemical structure and rate of hydrolysis has been found for the tetrapentoxysilane isomers.

Petroleum Refining Laboratory, Pennsylvania State University. WADC TR 55-30 Part 4.
FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. E. Erwin Klaus, Merrell R. Fenske. AF 33(616)-2851. October 1956. ASTIA Document No. AD 110688.

This report describes work carried out on a continuing project directed toward the development of improved hydraulic fluids, jet engine lubricants, and jet fuels for the Air Force. Primary emphasis for the period covered by this report has been placed on the study of the high temperature aspects of these fluids developments.

Hydraulic fluid developments have been focused on fluids suitable for use at 500° to 700°F for limited times in sealed hydraulic systems. Tests have been developed for a basic study of viscosity, thermal stability, oxidation stability, and lubricity in this desired temperature range. The high temperature properties of 10 chemical classes of fluids have been surveyed. Additional efforts have been concentrated on the study of chlorine-containing silicones, hydrocarbons, esters, and chlorinated aromatic hydrocarbons for application to these high temperature hydraulic systems.

Jet engine lubricants are needed for operation at 500° and 600°F bulk oil temperatures and up to 700°F hot spot temperatures. In addition to the data obtained in the hydraulic fluid study, emphasis has been placed on more severe lubricity, oxidation, and corrosion requirements. Again emphasis has been placed on the same four types of compositions. Lubricity, oxidation, and corrosion studies over the range of 500° to 700°F are presented. Attention has been focused on the best utilization in a lubricant system of the properties obtainable in this temperature range.

Techniques for the measurement of jet fuel dirtiness have been studied. Methods of improving jet fuel dirtiness by additives and other treatments have been investigated.

Some additional miscellaneous duties in the form of fluid formulations, distribution of standard fluid samples and Laboratory reports have been performed at the request of the Wright Air Development Center.

Western Gear Corporation. WADC TR 56-262.
OPERATING INSTRUCTIONS FOR GEAR AND SPLINE LUBRICANT TESTER. Charles W. Sauter. AF 33(616)-2967. October 1956. ASTIA Document No. AD 110422.

A universal gear lubricant tester has been constructed to evaluate developmental high temperature gear lubricants under simulated flight conditions. This report gives the operating instructions for such a tester operating at speeds up to 30,000 revolutions per minute and temperatures up to 800°F.

Shell Development Company. WADC TR 56-370.
RESEARCH IN HIGH TEMPERATURE BEARING LUBRICATION IN THE ABSENCE OF LIQUID LUBRICANTS. Charles H. Bailey, Stanley S. Sorem. AF 33(616)-2999. October 1956. ASTIA Document No. AD 110411.

The applicability of the "protective atmosphere" system to the operation of high temperature alloy rolling element bearings at temperatures from 600°F to 800°F is being investigated. This system involves surrounding the operating bearing with a reducing atmosphere consisting of a mixture of organic vapor and a volatile extreme pressure additive in air. The reducing environment prevents the formation of destructive abrasive metal oxides. Reactions of the extreme pressure additive and the organic vapor with or on the metal surfaces results in the formation of films which provide the necessary "lubrication" for high temperature operation.

Results of this investigation to date show that the

"protective atmosphere" system is applicable to bearings constructed of ferrous alloys. Bearings with tool steel balls and races and steel cages appear best suited. The desired films cannot be formed on metals of low oxidation potential such as bronze or silver nor on the oxidation resistant alloys such as the stainless steels. The contribution of the organic portion of the atmosphere to the protective film formation has only recently been recognized. Exploitation of this discovery may lead to further improvements in "protective atmosphere" composition.

Best results thus far have been obtained with a "protective atmosphere" consisting of a mixture of air and JP-4 fuel vapor in a weight ratio of 8 to 1. The JP-4 used contained about 0.2% sulphur. In this atmosphere a size 206 all ferrous metal bearing has been operated 100 hours at 10,000 rpm and 700°F without appreciable wear or other indications of bearing deterioration. The limits of the "protective atmosphere" system with respect to bearing temperature, speed and load are yet to be determined.

WADC TR 55-449 Part 4.
MICRO LUBRICANT TEST METHODS - Part 4: Evaporation Loss of Lubricating Greases and Oils, Viscosity of Lubricants at -65°F, and Foaming Characteristics of Crankcase and Aircraft Engine Lubricating Oils. John B. Christian. November 1956. ASTIA Document No. AD 110587. PB 121849.

This report describes test methods for the determination of evaporation loss of lubricating greases and oils, viscosity of lubricants at -65°F, and the foaming characteristics of crankcase and aircraft engine lubricating oils. All procedures were arrived at through the comparison of the micro test results with results obtained through the use of existing test procedures.

WADC TR 56-229.
STUDY OF RUST-INHIBITING COMPOUNDS IN SYNTHETIC GREASES BY THE HUMIDITY CABINET METHOD. H. Schwenker, H. D. C. Hill, R. J. Benzing. November 1956. ASTIA Document No. AD 110597. PB 121873.

A series of rust-inhibitors were investigated for their effectiveness in a Lithium 12 hydroxystearate-di-2(ethyl-hexyl) sebacate grease and in a Bentone 34-dipropylene-glycol-dipelargonate grease by means of the Humidity Cabinet Test. Mechanical stability tests were made on the soap greases and electronmicrographs taken of several of the test greases. Sulfonates and amines were the most effective inhibitors in the soap-diester greases. The amount of inhibitor used was also found to be critical with regard to the effectiveness of certain additives. Mechanical stability of the soap-diester type greases was not affected by the various rust-inhibitors. some effect on the unworked penetrations of the soap-diester greases when different inhibitors were used was noted. Electromicrographs of the soap-diester greases revealed a difference in the dispersion and growth of the soap fibers when in the presence of certain inhibitors. However, no significant effect on the consistency of these greases was noted.

The Bentone 34-dipropylene-glycol-dipelargonate greases were not particularly susceptible to rust-inhibitors of any type. The best results obtained were with sulfonates and dibutyl ammonium oleate. However, these inhibitors provided only borderline protection at most. No effect on the mechanical stability of these greases by the use of rust-inhibitors was noted.

WADC TR 56-430 Part I.
THE EFFECTS OF NUCLEAR RADIATION ON MILITARY SPECIFICATION GREASES. William L. R. Rice, 1/Lt. December 1956. ASTIA Document No. AD 110644. PB 121914.

A portion of the Air Force effort toward the development of nuclear radiation resistant lubricants is devoted to an evaluation of the effects of gamma radiation on available specification and non-specification greases. Data are presented on the effects of gamma radiation on forty-seven greases. Many of the greases tested appear to be satisfactory for use after exposure to about 1×10^8 roentgens, the screening dosage used for these studies.

WADC TR 56-475.
EVALUATION TESTS OF ARYLUREA-SILICONE GREASES IN AIRCRAFT EQUIPMENT. Herbert Schwenker. December 1956. ASTIA Document No. AD 110634. PB 121915.

Several types of arylurea-silicone greases have been evaluated in various types of test equipment. Some of the tests were conducted with actual aircraft components, while other tests consisted of mock-ups of various aircraft systems.

Arylurea-silicone greases have good performance in the following high temperature applications: airframe oscillatory bearings, electric motor bearings, low speed roller bearings, instrument bearings.

Arylurea-silicone greases did not function satisfactorily in screw jack actuator applications and in high speed ball bearings.

The type of application, equipment design, materials used in construction, and the surrounding environment all were found to be significant factors influencing the success or failure of arylurea-silicone greases to function as lubricants.

WADC TR 56-11.
THE PREPARATION AND PROPERTIES OF SOME FLUORINE-CONTAINING DIESTERS. Joseph F. O'Brien, Capt., Robert Filler, Jack V. Fenner, 1/Lt., George Rappaport. December 1956. ASTIA Document No. AD 110674. PB 121910.

The preparation and properties of six different types of fluorine-containing diesters for possible use as high temperature lubricants and hydraulic fluids are described. Both dicarboxylic acid diesters and glycol esters were prepared from fluorine-containing mono- and dicarboxylic acids, alcohols and glycols. Diesters derived from fluorine-containing acids and hydrocarbon alcohols or glycols were readily prepared by direct esterification. Diesters derived from fluorine-containing alcohols or glycols and hydrocarbon acids or fluorine-containing acids were prepared from the acid chloride and alcohol or glycol. The fluorine-containing diesters most resistant to hydrolysis were derived from the fluorine-containing alcohols or glycols and the hydrocarbon acids. The boiling point, refractive index, and surface tension of the fluorine-containing diesters were lower than those of their hydrocarbon analogs whereas the density and absolute viscosity were higher. The infra-red spectra of the diesters were recorded and the effect of fluorine atoms on the ester carbonyl frequency is described.

WADC TR 56-1.
ALASKAN TEST SITE OIL EXPOSURE PROGRAM. Robert J. Bensing. January 1957. ASTIA Document No. AD 110702. PB 121846.

A program was undertaken to determine the effect of prolonged outdoor unheated shed storage on the pour

points of several specification oils of mineral oil and diester base stock origin. Permanent increases in pour point following prolonged cyclic low temperature storage had previously been reported for automotive and industrial oils. Qualitative daily visual observation and monthly pour point data on ten mineral oils and four diester oils covering two years of observation in Fairbanks, Alaska, are reported.

Petroleum oils, in general, showed increases in pour points after storage while the synthetic base oils had stable pour points. The conclusion drawn was that with the increase of use of synthetic oils, no problem should be experienced in low temperature use after storage.

Shell Development Company.
WADC TR 55-102 Part III.
GREASE LUBRICATION OF HIGH SPEED ANTI-FRICTION BEARINGS. John B. Accinelli. AF 33(616)-2443. January 1957. ASTIA Document No. AD 110701. PB 139000. Order from LC, Mi $3.00, Ph $6.30.

The report summarizes the final work done on grease lubricated anti-friction bearings at speeds of 1.0×10^6 DN and higher. The work was performed at the Shell Development Company on two 20 mm ultra high speed ball bearing rigs (Rig No. AF-2443-1 and -2) designed and built by this company for the Air Force. Work with grease at 1.0 to 1.2×10^6 DN suggests that the interplay of mechanical factors such as rotor dynamic unbalance, bearing type, size and precision, load alignment, bearing-to-housing fits, etc., will affect performance more than changes in grease properties. Grease lubrication at these ultra high speeds appears to be affected considerably more by mechanical factors than is oil-air mist lubrication, indicating that grease lubrication must be inherently more marginal. One test at 1.0×10^6 DN with a grease meeting MIL-G-3278 ran well for 109 hours, thus demonstrating that under the proper conditions satisfactory long term lubrication with grease is possible.

With all greases, operation of the rig at 30,000 rpm was significantly better than operation at 50,000 rpm. A grease containing silicone oil was poor at both speeds.

Univ. of Virginia. WADC TR 53-293 Part 7.
HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS - Part VII: Evaluation of Antioxidants in Synthetic Fluids. James W. Cole, Jr. AF 33(616)-3234. January 1957. ASTIA Document No. AD 110728. PB 121990. Order from OTS $2.25.

The object of this work is the evaluation of selected additives as antioxidants and oxidation inhibitors for non-petroleum base lubricating and hydraulic fluids in the range 400° to 700°F. The emphasis in this report is on the further examination of selected amines in methyl phenyl silicones, chlorophenyl silicones, and a tetrakis-n-dodecyl silane in the presence of aluminum, silver, copper, stainless steel and titanium. Acridines, dipyridyl amines and aryl phenylene-diamines showed considerable promise as inhibitors of increases in viscosity arising from oxidative attacks. Variations in the relative effectiveness of other amines examined do not allow the establishment of definite relationships between structure and antioxidant activity at the upper temperatures.

Some studies with selected additives in a pentaerythritol ester and in bis-(1-methyl cyclohexylmethyl) sebacate in the range 400° - 500°F showed the same general oxidative phenomena as previously described for bis-(di-2-ethylhexyl) sebacate. Experiences are described with five chlorophenyl phosphate fluids in the range 600°-700°F. This type has considerable inherent stability to oxidation in the absence of additives and metals. No additive was found which inhibited oxidation in the presence of metals.

WADC TR 56-375.
WEAR STUDIES WITH TITANIUM. Robert J. Benzing, Arthur N. Damask. January 1957. ASTIA Document No. AD 110585. PB 121885.

A study of the wear of titanium using typical oils was made on the Falex Wear Tester and the Shell Four-Ball Wear Tester. The purpose of this study was to provide data for comparison with other studies dealing in friction coefficients and wear of specimens in non-standard lubricant testers. A mineral oil, diester, silicate ester, silicone, and halogenated hydrocarbon were the oils studied. C-130-AM, Ti-150A and 3Al-5Cr alloys were used and both cyanided and uncyanided surfaces are included in the program.

WADC TR 55-449 Part 5.
MICRO LUBRICANT TEST METHODS - Part 5: High Temperature Viscosity. John B. Christian. February 1957. ASTIA Document No. AD 118048. PB 121995. Order from OTS $0.50.

This report introduces a small scale method of analysis for the determination of viscosity at high temperatures (550°-700°F). Its application is especially important in instances where only minute quantities of sample are available. The small scale replica of the modified Ostwald Viscometer employing 1.5 millimeters of sample was used in the test method described.

A bath solution composed of the nitrates of lithium, potassium, and sodium has been substituted for the usual oil bath and has proved to be satisfactory.

WADC TR 55-385.
HIGH TEMPERATURE EVALUATION OF ANTIOXIDANTS IN DIESTER BASE FLUIDS. Howard D. C. Hill. March 1957. ASTIA Document No. AD 110664. PB 121991.

This study was initiated to evaluate various organo-selenium, organo-sulfur, and organo-nitrogen additives as oxidation inhibitors in diester base fluids. Samples of these materials were submitted to oven storage at 350°F for a period of eight (8) days after which evaporation losses, change in viscosity, and sludge content was determined. A temperature of 350°F was selected as representing the upper temperature limit of a -100° to 350°F grease requirement for which the additives were evaluated.

The results of the tests discussed herein indicate that, of the three groups of compounds evaluated, the selenium-containing compounds provided the best oxidation inhibitors from the standpoint of low evaporation losses and low viscosity change.

WADC TR 56-275.
RECLAMATION OF SYNTHETIC BASE OILS. Richard W. Sneed, H. M. Schiefer, 1/Lt. March 1957. ASTIA Document No. AD 118110. PB 131181.

The feasibility of the reclamation of synthetic base oils, specifically of the diester type, has been investigated. Various filtration, percolation, and distillation techniques have been employed in an attempt to clean used oils to such an extent as to make them reusable, either as an aircraft gas turbine engine lubricant or as a plasticizer grade ester. It has been determined that hydraulic fluid used for flushing alternator drives can be filtered and used again instead of being discarded as is the present practice in depots. Preliminary studies of reclamation procedures for used engine oils indicate that full scale reclamation is possible. However, considerable work would have to be done to ascertain the efficiency and economy of the reclamation operation and also the problems involved with full scale operation of engine with reclaimed oil.

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Shell Development Company.
WADC TR 56-370 Part II.
RESEARCH IN HIGH TEMPERATURE BEARING LUBRICATION IN THE ABSENCE OF LIQUID LUBRICANTS. Charles H. Bailey, Stanley S. Sorem. AF 33(616)-2999. March 1957. ASTIA Document No. AD 118121.

The "protective atmosphere" system for operation of high temperature alloy rolling element bearings at temperatures from 600°F to 800°F is being investigated. This system involves surrounding the operating bearing with a special reducing atmosphere. This consists of a "rich" mixture of organic vapor and air with small additions of volatile film forming compounds. The reducing environment prevents the formation of destructive abrasive metal oxides. Reactions of the additive and organic vapor with or on the metal surfaces results in the formation of films which provide the necessary "lubrication" for high temperature operation.

Phase I work on mechanisms and atmosphere compositions was continued using size 206 bearings operating under light load at 10,000 rpm. The upper temperature limit of a specific atmosphere composition was found to be 800°F. Possible reasons for the limit are given and means of increasing it are discussed. A group of oxygen and nitrogen containing compounds naturally occurring in JP-4 (approximately 1%w) were found to beneficially affect the nature of the lubricating films formed, thus leading to superior "protective atmosphere" operation. Several representative compounds from this group have been tested individually and of these m-toluidene was outstandingly good.

Phase II work, outlining the load, speed, and life characteristics of bearings operated in a "protective atmosphere" was started using size 209 bearings operating at various speeds and loads. A bearing operated at 700° F under 2/3's of catalog load rating successfully completed 33 hours. Wear was appreciable during this test but compromises in both the mechanical installation and atmosphere composition were necessary in order to work with available bearings. These may have affected the wear results.

WADC TR 57-113.
DETERMINATION OF THE SHEAR STABILITY OF VARIOUS AIRCRAFT LUBRICATING GREASES BY THE HIGH TEMPERATURE SHELL ROLL TESTER. Donald T. Kjerland, 2/Lt. April 1957. ASTIA Document No. AD 118246. PB 131161.

The shear stability of various high temperature lubricating greases was determined at temperatures up to 600°F by use of a Shell Roll Tester which was adapted for high temperature testing. The greases evaluated were of both soap and non-soap types and contained the following types of base fluids: silicone, pentaerythritol ester, diester, substituted m-terphenyl and biphenyl, and petroleum oil.

Bargraphs plotting time in the Shell Roll Tester at a given temperature and rotational speed versus the resultant grease consistency were prepared for each of the lubricating greases evaluated. Although the main emphasis in this work was on the determination of the shear stability of a given grease at various temperatures, some testing was done in an effort to determine what effect, if any, rotational speed had on the change in grease consistency in this test.

The High Temperature Shell Roll Tester has been shown to be an effective method to determine the shear stability of a lubricating grease at high temperatures.

Continental Oil Company
WADC TR 55-190 Part III.
DEVELOPMENT AND EVALUATION OF A GREASE FOR -100°F TO+350°F. Ernest W. Nelson, Warren W. Woods, William P. Scott. AF 33(616)-2364. APRIL 1957. ASTIA Document No. AD 118236. PB 131078.

In the work directed toward the development of an aircraft grease suitable for use over a wide temperature range, emphasis has been placed on the utilization of certain hexa alkoxy disiloxane fluids as base oils. The thickener was limited to colloidal calcium carbonate coated through chemical interaction with the calcium salt of 3-methyl glutaric acid. Formulations using the base oil hexa (2-ethyl butoxy) disiloxane show promise of utility over a -100°F to+325°F temperature range. Formulations using the base oil hexa (2-ethyl hexoxy) disiloxane show promise of utility over -100°F to+350°F. This fluid was not available in large quantities until the last quarter of the contract period. Time has permitted the preparation and evaluation of only a limited number of hexa (2-ethyl hexoxy) disiloxane greases. These greases perform in the range of 450 to 600 hours at 350°F in the high speed testers, providing pure oxidation inhibitor (p, p'-dioctyl-diphenylamine) is used. The pure grade is not commercially available at this time. The use of commercial grade inhibitor limits performance to around 150 hours at 350°F.

Standard Oil Company (Indiana).
WADC TR 53-83 Part V.
DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASE THICKENERS. John C. Goossens. AF 33(038)-23687. April 1957. ASTIA Document No. AD 118219. PB 131092.

In this work directed toward the development of new grease thickeners operable at temperatures above 450°F and as high as 700°F, a variety of materials have been tested. Those thickeners which produced greases in DC 550 Silicone Fluid in less than 30% concentration and withstood preliminary screening tests were further tested in the ABEC-NLGI Bearing Tester.

The general types of materials tested as high temperature thickeners were organo-metal compounds, polymers and polar organic compounds. Selection of materials for test was based on thermal stability, ease of preparation and ability to prepare the substance in small particle size. Of the many materials tested, those which have shown the greatest promise are substituted pteridines, urea derivatives and a polymer of melamine with dimethylsulfoxide.

As they presented themselves, a few additives for improving the lubricity and load carrying capacity of silicone fluids were tested in the Navy Gear Wear Test and the Shell Four Ball Wear Test. None of the tested additives were notably effective. Thus a number of promising high temperature grease thickeners are available while the fluid continues to be the major limiting factor in the development of a lubricating grease for use as high as 700°F.

Denver Research Institute.
WADC TR 54-464 Part 3.
DEVELOPMENT OF SCHEMATIC ANALYTICAL PROCEDURES FOR SYNTHETIC LUBRICANTS AND THEIR ADDITIVES. Francis S. Bonomo. AF 33(616)-3336. April 1957. ASTIA Document No. AD 118215. PB 131063.

Methods for the separation, identification, and determination of certain components of synthetic greases and synthetic lubricants are presented and discussed. Included in these components are such base-oils as dibasic acid esters, silicate esters, disiloxanes, and silicone oils, and selenide and phenol type antioxidants.

Analytical chromatographic procedures employing column partition, paper partition, and adsorption techniques for the separation and identification of different groups of components are presented and discussed. Column partition chromatography is shown to be a useful means for isolating high molecular weight dibasic acid esters from silicate esters, disiloxanes, and silicone oils. Adsorption chromatography is employed for the separation and concentration of antioxidants from base-oils, for the removal of impurities from lubricants, and for qualitative comparisons of lubricants of known compositions.

Qualitative and quantitative procedures for the detection and determination of selenium-containing antioxidants are presented. Attempts to detect phenol-type antioxidants by volatilization were unsuccessful.

Preparation of the laboratory manual, an outline of the analytical methods in the manual, and a list of ASTM methods included in whole or in part in the manual are discussed briefly. The manual itself, resulting from two and one-half years' research for WADC under Contracts AF 33(616)-2204 and AF 33(616)-3336, is Part IV of this report.

Coordinating Research Council, Inc.
WADC TR 57-36 Part I.
AIRFRAME LUBRICANTS. Airframe Lubricants Group. AF 33(616)-2888. May 1957. ASTIA Document No. AD 118277. PB 131203.

The annual report on Airframe Lubricants is a status report that reviews the activities of the various projects undertaken to assist the Air Force in conducting research on the problems of mutual adaptation of airframe lubricants and equipment, with particular emphasis on the work done from June 1954 to December 1956. This report, covering seven projects in all (High Temperature Testing of Greases in Antifriction Bearings, Fretting Corrosion, Rust-Preventive Properties of Greases, Plain Bearings, Screw Thread and Gear Mechanisms, Bonded Solid-Film Lubricating Coatings, and Methods for Cleaning Bearings Prior to Use in Laboratory Tests), describes the scope and objective of each project, the work of the Laboratory and Service Evaluation Panels for each, the conclusions reached, and the plans under consideration for future work along the same lines.

WADC TR 57-299.
NUCLEAR RADIATION RESISTANT LUBRICANTS. William L. R. Rice, 1/Lt. May 1957. ASTIA Document No. AD 118329. PB 131159.

This report presents a general summary of the state of the art of nuclear radiation resistant lubricant development. Data are presented on the effects of gamma radiation on currently available fluids and lubricants, showing the limitations of each. Development of advanced materials is discussed, with a general outline presented of the approach taken by the major support contractors.

It is estimated that on the basis of information to date, conventional ester type lubricants should prove operable to at least 1×10^7 roentgens of gamma radiation at temperatures from -65° to 350°F. Improved ester types, of the aromatic ester or complex ester variety, with proper additives, are expected to give suitable performance up to about 1×10^8 roentgens, operating in the temperature range -40° to 400°F or higher. Selected mineral oils, usable from about 0° to 400°F, may possibly be reliable to about 2 to 3×10^8 roentgens. The aromatic ethers, which are expected to be stable at temperatures well above 400°F (laboratory tests show thermal decomposition temperatures of 700° to 800°F), have not been evaluated for radiation resistance, but based on similar types of aromatic compounds they should be stable in the range 10^9 to 10^{10} roentgens. Much further work is required to verify this estimate.

A summary is presented of the future plans and major problem areas remaining in the fluid and lubricant development programs.

Iowa State College. WADC TR 53-426 Part IV.
ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS. Henry Gilman, Richard D. Gorsich. AF 33(616)-3510. May 1957. ASTIA Document No. AD 118307. PB 131176.

This project is concerned primarily with the syntheses of new compounds to be examined as potential hydraulic fluids and lubricants. The syntheses and preliminary screening of organo-metallic and organo-metalloidal compounds has been extended. New cyclic compounds continue to be promising. It has also been found that the meta-biphenylyl group can be added to those groups which impart desirable physical characteristics. The methyldi-benzyl-silylmethyl group is also of significant promise.

In the experimental part, there is included a table which outlines the preliminary screening of thirty-six compounds.

Lehigh Univ. WADC TR 55-240 Part III.
BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP LUBRICATING GREASES. John J. Chessick, Albert C. Zettlemoyer. AF 33(616)-2440. June 1957. ASTIA Document No. AD 130808. PB 131233.

This project is aimed at the determination of the fundamental factors in the formation of non-soap greases, and the relation of these factors to their thermal, chemical and mechanical stability. The vehicle-thickener interface was early recognized as the seat of important properties in non-soap greases. In order to index the nature and extent of this interface, first the physical properties of several oils and the surface characteristics of a variety of thickening agents were measured. These properties of the oils and solids were correlated with the behavior of grease systems formulated with them. Modification of the interfacial region by water and its effect on grease building by inducing flocculation was then investigated.

The influence of concentration and surface area of hydrophobic thickeners on the consistencies of various greases was determined specifically. Above 200 $m^2/g.$, grease building varies directly with surface area, but falls off sharply below this level. To give greases of a specified consistency, the amounts of thickeners required varies as the square of their surface areas. It appears also that the effect of area is similar for all thickeners.

Grease consistency was found to be directly proportional to the amount of water adsorbed at the oil-thickener interface for hydrophilic thickeners. As might be expected, the hydrophilic thickeners adsorb relatively large amounts of water and yield much larger changes than greases built with hydrophobic thickeners. The amount of water in a grease varies considerably with temperature, amount of working and the relative humidity.

While water enhances the gel forming ability of hydrophilic solids in nonpolar vehicles, somewhat surprisingly, the opposite effect is found for these solids dispersed in polar liquids miscible with water. The thickening effect of hydrophobic thickeners is not nearly so sensitive to the polarity of the vehicle, since very little water is adsorbed to develop flocculation.

These and related data were used to develop a mechanism for the flocculation of hydrophobic and hydrophilic solids in grease vehicles to form non-soap gels. Of great importance was the finding that in gelling pure liquids, the

particle size of the thickener is more important than the polarity of the surface. However, in the presence of trace amounts of water, commonly present under practical conditions, grease consistency is strongly dependent on the polar nature of the solid surface. For thickeners dispersed in oils containing both water and additives the polarity of the surface determines whether the water or additive will be preferentially adsorbed.

Further studies of the influence of heptyl additives, their benzene analogues, as well as more complex additives were carried out. In addition, rheological measurements of a number of thickener solids in several grease vehicles were made. The preparation of arylurea thickeners was also recently begun. These studies have not yet proceeded to a point where a quantitative discussion can be offered.

Low angle X-ray scattering techniques have been used to study grease structure. Marked diffraction maxima have been found corresponding to spacings of the order of 100 to 2000 Å units for copper phthaloyanine thickened grease.

Southwest Research Institute. WADC TR 57-93. PROCEEDINGS OF AIR FORCE-NAVY-INDUSTRY CONFERENCE ON AIRCRAFT LUBRICANTS. Mrs. Opal M. Fielder. AF 33(616)-3820. June 1957. ASTIA Document No. AD 130815.

The Annual Air Force-Navy-Industry Conference on Aircraft Lubricants was held at the Hilton Hotel, San Antonio, Texas, on November 5, 6, and 7, 1956. Approximately 300 representatives from the Department of the Air Force, the Department of the Navy, other government agencies, and the industry, attended. A total of 36 papers were read: 20 on gas turbine lubricants, 7 on greases, 5 on hydraulic fluids, and 4 on solid film lubricants.

Denver Research Institute. WADC TR 54-464 Part IV. DEVELOPMENT OF SCHEMATIC ANALYTICAL PROCEDURES FOR SYNTHETIC LUBRICANTS AND THEIR ADDITIVES. Part IV: Laboratory Manual for the Analysis of Synthetic Lubricants, Greases and Their Additives. Francis S. Bonomo, Joseph J. E. Schmidt. AF 33(616)-3336. July 1957. ASTIA Document No. AD 130922. PB 131414. Order from OTS $6.00.

Laboratory methods and techniques for the analysis of certain synthetic lubricants, greases, and their additives are described in detail, including drawings and photographs of equipment when necessary. As stipulated by WADC, none of the methods includes optical or elaborate instrumental techniques of analysis. Most of the methods employ simple wet chemical manipulations, or such techniques as adsorption or partition paper or column chromatography. The individual methods are numbered and patterned after Federal Test Method Standard No. 791.

The manual is divided into five major sections:

(1) Preliminary qualitative examination of the lubricant or grease, including solubility, elemental analysis, identification of grease type, and behavior of the lubricant (or base-oil from a grease) upon adsorption on a column of silica gel.

(2) Qualitative detection and quantitative determination of antioxidants.

(3) Qualitative detection and quantitative determination of corrosion preventive compounds.

(4) Separation and quantitative determination of gelling agents and thickeners.

(5) Separation, identification, and determination of base-oils from lubricants and greases.

In a number of instances parts of existing ASTM methods have been incorporated into the analytical procedures, where applicable, to avoid the use of parallel methods to the same goal.

Shell Development Company. WADC TR 57-177. ENGINE OIL DEVELOPMENT. C. Lynn Mahoney, William W. Kerlin, Emmett R. Barnum, Karl J. Sax. AF 33(616)-3182. July 1957. ASTIA Document No. AD 130925.

Inhibited lubricants, made from ester or aliphatic-hydrocarbon base oils, are highly sensitive to radiation damage even in absence of oxygen. The effect on the base oil itself, measured by such properties as viscosity change or formation of acidic products, is generally relatively small at radiation doses less than 10^8 reps. The antioxidant, however, acts as a radical scavenger. While protecting the base oil against radiation-induced change, it is selectively inactivated. Loss of the antioxidant activity leaves the lubricant unprotected against attack by oxygen, which leads to formation of corrosive materials and deposits. Degradation products formed during irradiation of ester oils also greatly reduce the oxidation stability of inhibited lubricants. For example, a di-2-ethylhexyl sebacate blend, containing 0.5% phenothiazine as an inhibitor, had all antioxidant activity destroyed during irradiation to 5×10^7 reps (γ-rays, ambient temperature, nitrogen atmosphere). With a MIL-L-7808C diester lubricant, containing 1% phenothiazine and 5% tricresyl phosphate, formation of strong phosphoric acid derivatives accelerated destruction of the inhibitor. Complete loss of antioxidant activity occurred in this lubricant after irradiation to 10^7 reps. The strong phosphoric acid derivatives increased decomposition of the ester, leading to very high deposit levels in panel coke tests. The high panel coke deposits found with other irradiated ester lubricants were also caused by the presence of tricresyl phosphate.

Auto-oxidation proceeds through free radical chain reactions. Therefore, oxidation reactions would be accelerated during irradiation. The stability of inhibited oils, in oxidation-corrosion tests conducted in a γ-ray source, decreased rapidly as the radiation dose rate was increased. At 400°F, a dose rate of 1.5×10^4 reps/hr. shortened the stable life of a wide variety of antioxidant-oil combinations to approximately 70% of that obtained in absence of radiation. When the dose rate was increased to 7.2×10^5 reps/hr. only 25% of the original stability was retained. In oxidation tests at 347°F and dose rates of 7.2×10^5 reps/hr., only 12% of the unirradiated stability was found for various types of antioxidant-oil blends. The stable lives of all inhibited lubricants tested, in irradiated oxidation-corrosion tests at both 347°F and 400°F, appeared to be approaching zero at an estimated dose rate of 2.5×10^6 reps/hr. The total γ-ray dose absorbed during the stable life of the various antioxidant-oil blends depended on the relative stability of the blend, the temperature of the test and the radiation dose rate, but in no case was it greater than 2×10^7 reps.

No protection against antioxidant radiation damage was found through use of radical scavengers or energy transfer agents (various iodine-containing compounds or aromatic materials). Base oil damage could be reduced through use of such materials but the reactivity of antioxidants towards radicals was considerably greater than compounds added as protectors.

The dialkyl selenides show some promise for use with ester or hydrocarbon base oils at temperatures and radiation levels somewhat above those attainable with the more usual antioxidants. On an equal weight basis, the dialkyl selenides are slightly less effective than phenothiazine in imparting oxidation stability or in protecting ester base oils from radiation damage. However, they can be

used in much higher concentrations without greatly affecting the deposit-forming tendency of the lubricant. The selenides must be kept from contact with copper and silver to avoid corrosion and loss of activity.

For use at bulk oil temperatures above 400°F or at total radiation doses of 10^8 reps or higher, inherently-stable, aromatic-containing materials will probably be needed. Preliminary work with specifically designed compounds has been directed towards determining the advantages and disadvantages of the various types of substituents and linkages that can be used with aromatic compounds. Tertiary butyl and carbomethoxy groups are promising substituents for use with benzene derivatives.

Silyl, ether, and ester groups can be used to link aromatic nuclei without large reduction in oxidation or radiation resistance. Preliminary examination of some of these synthetic compounds shows that their useful life, determined in oxidation tests at 400°F, would be at least 10-20 times longer than the most stable of the MIL-L-7808C ester lubricants. Additional aromatic derivatives are being prepared and examined in various high temperature screening tests.

WADC TR 57-283.
EVALUATION OF ANTI-WEAR AND EXTREME PRESSURE CHARACTERISTICS OF NEW HETEROCYCLIC COMPOUNDS. Robert J. Benzing. July 1957. ASTIA Document No. AD 130902. PB 131377.

A series of nineteen compounds were studied as possible anti-wear additives or extreme pressure additives. The materials were evaluated in Di(2-ethylhexyl) sebacate in the Shell Four-Ball Wear Tester and Shell Extreme Pressure Tester.

The basic types studied were compounds of heterocyclic structure containing one or more of the following heteroatoms: nitrogen, oxygen, selenium and sulfur. Some gave promise as good wear and extreme pressure additives.

Pennsylvania State Univ.
WADC TR 55-30 Part V.
FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. E. Erwin Klaus, Merrell R. Fenske. AF 33(616)2851. July 1957. ASTIA Document No. AD 130907. PB 131405.

This report describes work carried out on a continuing project directed toward the development of improved hydraulic fluids and jet engine lubricants for use in the high temperature range of 400° to 700°F. With few exceptions these studies are equally applicable to hydraulic fluids and jet engine lubricants.

Efforts have been concentrated on the study of mineral oils, hydrocarbons, and improved stability esters for application to high temperature systems. Super-refining of mineral oils and hydrocarbons by hydrogenation, acid extraction, and silica gel percolation has been shown to improve materially the overall quality of these materials as hydraulic fluids and lubricants. Selection of the molecular configuration of esters has been shown to affect their thermal stability and hence their overall utility as high temperature fluids.

A number of new and improved test techniques have been developed or applied to the high temperature evaluation of hydraulic fluids and jet engine lubricants. These tests include: (1) high temperature density measurement, (2) the controlled atmosphere panel coker test, (3) a modified thermal stability test including catalyst metals, (4) a high temperature PRL thin film oxidation test, and (5) a single pass high temperature lube rig.

A number of large scale blends designed for use in bench, mock-up, and actual service equipment (jet engines and high temperature hydraulic systems) have been formulated for evaluation by WADC and industry. Used samples of fluids from actual tests in jet engines and high temperature hydraulic systems have been evaluated. Results of these evaluations offer some insight into the actual behavior of fluids in service and the degree of realism in various laboratory tests in policing fluid quality necessary for adequate service behavior.

Some additional miscellaneous duties have been performed at the request of the Wright Air Development Center. These duties include: (1) distribution of Laboratory reports. (2) fluid formulation, (3) distribution of standard fluid samples, (4) preparation of WADC Technical Reports and papers on specific topics of interest, and (5) evaluation of specific fluid properties.

WADC TR 57-166.
HIGH TEMPERATURE WEAR EVALUATION TECHNIQUES AND DATA. Robert J. Benzing. July 1957. ASTIA Document No. AD 130953. PB 131410.

A general description is given of the modifications required to increase the operating temperature potential of the following four lubricant testers: Shell Four-Ball Wear Tester used for evaluating the lubricity of fluids; Shell Roll Tester used to examine the effects of mechanical shearing on grease consistency; bearing endurance grease tester for evaluation of the high speed and high temperature performance of greases; and hydraulic fluid pump stand for determination of dynamic effects of circulation on shear stability of fluids. The British IAE gear machine, recently acquired for evaluation of the anti-scuffing properties of lubricants, is described.

Lubricity data obtained with the four-ball tester are reported for Specification MIL-O-5606 hydraulic fluid and MIL-L-7808 turbine engine oil. The wear scar data for a wide variety of experimental fluids are compared to the wear patterns for the two specification materials.

WADC TR 57-199.
EVALUATION OF AN ARYLUREA-PENTAERYTHRITOL ESTER GREASE IN WHEEL BEARINGS OF INTERNAL TYPE BRAKE WHEEL ASSEMBLIES. D. T. Kjerland, 2/Lt. September 1957. ASTIA Document No. AD 131050.

The use of Specification MIL-L-3545 greases in wheel bearings of internal type brake assemblies has proven unsatisfactory. High temperatures encountered in wheel bearings as the result of heat dissipation from the brakes during aircraft take-off and landing operations have caused MIL-L-3545 greases to run out of the wheel bearings. An arylurea-pentaerythritol grease (MLG-9373) developed by Standard Oil Company (Indiana) under Contract AF 33(038)-23687 was subsequently evaluated in wheel bearings of laboratory test rigs of internal type brake assemblies and in wheel bearings of aircraft using the internal type brake assembly. Complete physical, chemical, and mechanical laboratory tests were performed on MLG-9373.

MLG-9373 grease gave satisfactory performance in all the wheel bearings of the various internal type brake assemblies tested from -65°F to temperatures in excess of 350°F while under heavy loads.

Specification MIL-L-25760 (USAF) has been written covering this type of grease. In the near future, greases qualified under Specification MIL-L-25760 (USAF) will be required as wheel bearing lubricants for internal type brake assemblies and other wheel bearing applications requiring a lubricant having a wider temperature range than Specification MIL-L-3545 greases.

Esso Research and Engineering Company.
WADC TR 57-255.
NUCLEAR RADIATION RESISTANT TURBINE ENGINE LUBRICANTS. Alfred H. Matuszak. AF 33(616)-3181. September 1957. ASTIA Document No. AD 131065.

This report covers the results of work aimed at developing radiation resistant turbine engine lubricants. The work has been divided into two parts: (1) Phase I covering finished lubricants and (2) Phase II covering potential lubricating oil base stocks, all screened at dosages up to 10^8 roentgens.

In the Phase I program, twenty-four oils were screened. These included finished or slightly modified mineral-based aviation oils, ester-based synthetic lubricants and a polyalkylene glycol mono ether lubricant. At 10^7 roentgens, no appreciable changes occurred in lubricant properties. At 10^8 roentgens, certain modifications of the mineral oils evaluated showed good retention of original physical and chemical properties. With uncompounded oils (Esso Aviation Oil 65 and 100) borderline performance was obtained in the MIL-L-7808 foam test and in the 347°F oxidation-corrosion stability test. These deficiencies can be partially overcome through the incorporation of additives. For improved oxidation-corrosion stability, phenyl-alpha-naphthylamine is beneficial. However, silicones are not very effective in maintaining foam suppression after irradiation. Additional work will be required to improve the foaming tendencies of mineral oils. This work will be undertaken using Barosa 56 as the base oil because of its slightly better test performance characteristics.

WS-3019, an MIL-L-25336 type synthetic lubricant, showed somewhat greater stability toward irradiation than any of the other synthetic materials evaluated. It retained most of its original properties and generally met the MIL-L-25336 requirements deviating only slightly in the 347°F oxidation-corrosion stability test. It exhibited, however, an undesirable flash point degradation (from 450 to 355°F after 10^8r) which can be attributed to a small amount of irradiation-produced contaminants.

A third oil, LO-2062, an MIL-L-7808 type adipate oil was indicated to have some promise providing its copper corrosion tendencies after irradiation can be corrected. The addition of about 1% tricresyl phosphate appears to offer a possible fix for improved stability up to gamma dosages of 10^8 roentgens. The effect of tricresyl phosphate in reducing copper corrosion at higher dosages will be investigated.

In the Phase II program twenty-three base stocks of varying chemical structures were screened at 10^7 and /or 10^8 roentgens. As in the case of Phase I lubricants the Phase II lubricant base stocks were evaluated against Military specification tests, notably MIL-L-7808 and MIL-L-9236. The most attractive materials evaluated were the inhibited mineral oil base stocks of which Barosa 56, a paraffinic base, is representative. Evaluation of this material at higher gamma dosages (5×10^8 and 10^9 roentgens) is contemplated. A sample of this base stock has been sent to WADC for more extensive laboratory and engine evaluations.

In the ester field, di-tridecyl carbonate appeared to be the most resistant to 10^8 roentgens. It exhibited good retention of physical properties but showed moderately high panel coking deposits. Additional work is planned with other carbonates at higher screening dosages. Higher flash diesters, such as di-tridecyl adipate, azelate and sebacate also showed promise of approaching a reasonable resistance to 10^8 roentgens. Their tendency to corrode copper and magnesium will require some additional attention. In this connection, the ameliorating effect of tricresyl phosphate will be investigated.

In the future program this initial effort to develop a radiation resistant lubricant will be expanded to include the evaluation of other synthetic and petroleum type finished oils and base stocks. Particular stress will be placed on higher flash base stocks, many of which are available commercially. Others may have to be synthesized. Any promising materials coming out of this screening program will be tested at higher gamma dosages. The most attractive base stocks will then be blended into finished oil formulations and finally evaluated further as radiation resistant engine lubricants.

Clevite Research Center.
WADC TR 57-61 Part I.
LUBRICATION OF TITANIUM. Richard O. Lee, Nicholas Fatica. AF 33(616)-3350. December 1957. ASTIA Document No. AD 142187. PB 131650.

The susceptibility of titanium to galling and seizure prevent it from replacing many steel parts involving sliding contacts in spite of the favorable strength-weight ratio of titanium. Particularly in aircraft, where one pound of steel replaced may result in gains of up to five pounds in pay load, the lubrication of moving parts involving titanium is an urgent problem. Past efforts to solve this problem have uncovered special lubricants and various surface treatments that have shown promise.

This investigation sought to explain the role of the oxide film on titanium, to obtain some information about the frictional properties of modified titanium coatings, and to make a direct comparison of the wear resistance of the best surface treatment in the presence of various lubricants using equipment generally acceptable for wear tests under conditions of boundary lubrication.

Studies on the stick-slip machine have shown that the oxide layer on titanium is easily penetrated even at moderate loads while the friction coefficient remains practically constant through this transition.

Other results on this machine show that moisture normally present in air is an important factor in determining the frictional properties of titanium. Although its effects depend on both the treatment of the metal and the lubricant used, generally speaking the moisture acts as a lubricant.

Temperature was found to be an important factor in the frictional properties evaluated on the stick-slip machine but its effect cannot be predicted even qualitatively without taking into account the surface treatment, the lubricant and the test condition.

Tests on the stick-slip apparatus also showed Halocarbon 11-14 to be generally effective as a lubricant on both untreated and treated titanium surfaces.

Studies on the Shell Four-Ball Wear Tester also showed Halocarbon 11-14 to be the most generally effective lubricant on both treated and untreated surfaces. There were no significant differences among the wear properties of untreated metals (Ti 75A, Ti 6Al 4V, Ti C-130 AM) using Halocarbon 11-14. Nitriding improved the wear properties and generally lowered the friction coefficient in the presence of practically all of the lubricants tested.

Carburizing improved the wear and frictional properties of Ti 75A in most of the lubricants tested. Carburizing Ti 6Al 4V lowers the friction coefficient but seems to be without effect on the wear resistance.

Polypropylene glycol 1025 and Halocarbon 11-14 have practically identical frictional and wear properties for any of the nitrided metals. The same may be said for carburized Ti 75A. However Halocarbon 11-14 is definitely superior to polypropylene glycol 1025 for carburized Ti 6 Al 4V.

In general, it may be said that all the other lubricants used were inferior to Halocarbon 11-14 and polypropylene glycol 1025 in performance with any metal surface. Where it was possible to compare the performance of the individual members of the inferior group (Nujol+1% fatty

acid addition, Carbowax, di-iso-octyl sebacate, Versilube F-50, Silicone (Dow F-60), Dow #41 grease, and silicate ester (MLO 8200), the results showed that they were practically equivalent.

Bronze sliding against titanium is an effective answer to the frictional and wear properties of Ti 75A for any lubricant tested. However, the use of this material partially defeats the objectives for using titanium in the first place.

The best wear values listed for any titanium specimen in the best lubricant are still about twice as large as those found for steel.

WADC TR 55-449 Pt VI.
MICRO LUBRICANT TEST METHODS. Part VI. Cloud Point, Pour Point, and Cloud Intensity. John B. Christian. December 1957. ASTIA Document No. AD 142210. PB 131648.

Miniaturized methods have been developed for the determination of cloud point, pour point, and cloud intensity. The methods described herein require a 5-milliliter sample for the three determinations.

Iowa State College. WADC TR 53-426 Pt. V.
ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS. Henry Gilman, Richard D. Gorsich, Bernard J. Gaj. AF 33(616)-3510. December 1957. ASTIA Document No. AD 142211.

Several novel synthetic procedures have been developed in connection with studies concerned with the preparation of organometallic and organometalloidel compounds to be examined as potential hydraulic fluids and lubricants. The promising cyclic compounds have been extended by making available cyclic-Si-M types. Procedures have been developed for the synthesis of non-cyclic unsymmetrical R3SiM compounds. One of the new developments involving tetrahydrofuran as a solvent in organo-silicon chemistry is the stepwise introduction of a wide variety of R groups by reactions between the Si-H group and some RM compounds. This provides the opportunity of synthesizing R4Si compounds, where all R groups may be unlike, and where there is a maximum promise of obtaining liquids.

The experimental part contains a table which describes the preliminary screening of twenty-eight compounds.

The security classification of this report is unclassified.

WADC TR 54-576 Pt II.
EFFECT OF METALS ON LUBRICANTS. Part II. Corrosion and Oxidation Stability at 400° Fahrenheit. John B. Christian. February 1958. ASTIA Document No. AD 150977. PB 131710.

This report presents data which deals with the general effects which silicates and siloxanes have on various metals; and the effects which these metals have on the fluids under severe conditions.

The corrosion and oxidation stability of a diester blend, a siloxane, and a silicate in the presence of various metal specimens is discussed.

University of Virginia. WADC TR 53-293 Pt VIII.
HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS. Part VIII. Evaluation of Antioxidants in Synthetic Fluids. James W. Cole, Jr. AF 33(616)-3234. February 1958. ASTIA Document No. AD 150994. PB 131975. Order from OTS $2.00.

This is a continuation of a laboratory program on the evaluation of the oxidation patterns of synthetic fluids in the presence and absence of inhibitors and metals in the temperature range 400°-700°F. Two methyl chlorophenyl silicones, F-50 and F-60 were examined at 500°F and 600°F. N,N'-di-2-naphthyl-p-phenylenediamine in 0.1 - 0.2% was the most interesting additive. Work with a mineral oil, MLO 57-30, did not reveal additives of outstanding activity. Substances containing sulfur and selenium showed promise, but some attack on silver and copper. The experience with four tetra-substituted silanes showed that these substances did not have outstanding response to additives. A series of runs with a pentaerythritol ester, MLO 55-584, indicate that ring substituted aryl amines have considerable antioxidant activity over the range 400°-500°F. Some additional data for bis-(2-ethylhexyl) sebacate are included to compare the promising amines with the phenothiazine type. The former retain the inhibition of activity over a wider temperature range. Some attention was given to determining the nature of the components in an oxidized diester which contributes to the acidity. It appears that some improvement may be achieved in a partial oxidized fluid by washing with hydrocarbon solvents. The limitations of a laboratory test procedure are discussed, especially with respect to the evaluation of the effects of the test metals, aluminum, silver, copper, titanium and several steels.

Midwest Research Institute. WADC TR 57-455.
HIGH TEMPERATURE SOLID DRY FILM LUBRICANTS. Melvin T. Lavik. AF 33(616)-3684. February 1958. ASTIA Document No. AD 150982. PB 131986. Order from OTS $0.75.

This report covers the development and evaluation of a dry lubrication test machine and the testing of several dry film lubricants. In order to meet the very high temperature and pressure requirements currently placed on lubricants in the aircraft industry, it was necessary to devise a suitable test instrument. The first part of this program was spent in designing, constructing and calibrating such a device capable of testing dry film lubricants at pressures as high as 50,000 psi and at temperatures up to 800°F. Several minor revisions have been made during the program leading up to the machine as reported here.

Several commercial dry film lubricants and also some materials prepared at the Institute were investigated for both friction coefficient and wear life at the various temperatures and pressures available with the test machine. In general, the materials worked with, while effecting reasonable lubrication, did not exhibit the wear life which is desired. The lubricant properties of several materials are discussed in this report, with the discussion serving mainly to point out areas in which further information is needed to realize the full benefits available from these materials.

Standard Oil Company. Indiana. WADC TR 53-83 Pt VI.
DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASE THICKENERS. John C. Goossens. AF 33(038)-23687. March 1958. ASTIA Document No. AD 151053.

This work was directed toward the development of new grease thickeners operable at temperatures above 450°F and as high as 700°F. During this period a variety of substances were prepared and tested as thickeners for silicone and ester fluids. Those which proved to be effective thickeners in less than 40% concentration were given preliminary screening tests and the better samples were run in the ABEC-NLGI bearing tester.

The general types of materials tested as high temperature thickeners were polar organic compounds, polymers, and organo-metal compounds. Selection of materials for test was based on thermal stability, ease of preparation, and ability to prepare the substance in small particle size. Of the many materials tested, those which have shown the greatest promise are, in decreasing order of their effectiveness, a new substituted pteridine, derivatives of hydrazine with bitolylene diisocyanate, phthaloyl melamine and some derivatives of cyanuric acid. However, at present none of these appears as good a high temperature thickener as the arylureas developed previously.

In addition to the usual 450°F high-speed bearing tests, 600°F bearing tests were conducted on silicone greases thickened with the two better thickeners, arylurea and a pteridine derivative. The tests were run both in air and under a slow stream of nitrogen. Both greases ran 85 to 120 hours under nitrogen. In air the arylurea had better oxidative stability running 40 to 60 hours in contrast with approximately 20 hours for the pteridine thickened grease.

Lehigh University. WADC TR 55-240 Pt IV. BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP LUBRICATING GREASES. John J. Chessick, Albert C. Zettlemoyer, James P. Wightman. AF 33(616)-3999. April 1958. ASTIA Document No. AD 151120.

Rheological measurements of dispersions of grease thickeners in pure vehicles or in vehicles containing water, additives or both these components have been carried out. Previous studies were made with the systems Aerosil--Plexol 201 and Santocel C2-paraffin oil. These types of measurements have been extended to include both polar and nonpolar solids dispersed in grease vehicles. The solids studied now include Copper Phthalocyanine, G. S. Hydrophobic Silica, HiSil, Aerosil, Santocel and Permagel. It was demonstrated that surface hydrophilicity and the presence of trace water far overshadows the influence of particle diameter and shape in the formation of gel structure by solid thickeners.

Further studies of the behavior of commercial additives have been made by measuring the change in consistency of a grease with increased concentration of additive. Because of the very complex nature of additive behavior a fundamental program has been developed to learn more of this phenomenon. This study has begun and includes measurements of additive adsorption from solution by selected solids as well as heat of immersional wetting measurements of solids in additive solutions at various concentrations. Studies thus far indicate that the heat of solution of additive in the grease vehicle and the heat of wetting of the solid thickener by pure additive relative to the vehicle appear to govern adsorption from solution. With Aerosil, a weakly polar solid, the heats of solution of the additives studied appears to predominate, and only those additives which dislike the vehicle (high endothermic heats of solution) adsorb and fluidize Aerosil-built greases. With rutile-built greases, however, the surface polarity of the thickener and consequent heat of adsorption is sufficiently high to influence adsorption of additives ineffective in Aerosil-built greases.

The program for the preparation of arylurea thickened greases has progressed satisfactorily. Several variables have been found to influence the particle diameter of the thickener solid formed. These include type of reactant, method of addition of reactants and the temperature of the reaction. Trace or larger quantities of water drastically increase particle size of arylurea thickeners prepared in solvents from which they can be isolated. The effect on greases prepared "in situ" is believed to be similar. This point is being checked.

California Research Corporation. WADC TR 56-646 Pt II.
EFFECTS OF RADIATION ON AIRCRAFT LUBRICANTS AND FUELS. R. O. Bolt, J. G. Carroll. AF 33(616)-3184. April 1958. ASTIA Document No. AD 151176.

The objectives of the work reported included the development of radiation resistant lubricants and a survey of the radiation stability of jet fuels.

Aromatic base materials were needed in the lubricant work, and most of these had to be synthesized. Exploratory synthesis evolved alkaryl ethers, alkaryl esters, alkylbenzenes, alkyl diphenyl ethers, and diarylalkanes. Chemical structure was correlated with physical properties and also with oxidation, thermal, and radiation stability. Although esters were inferior, each class had members with good radiation stability. Over-all, the alkyl diphenyl ethers offer the most promise for lubricant development. Polymers were also synthesized. These were used in an alkyl diphenyl ether to improve viscosity index and to increase viscosity. Compared to conventional thickeners, alkylated poly(α-methyl-styrenes)(APAMS) and poly(alkylphenoxyethyl methacrylates) (APEMS) show promise for use in future radiation resistant lubricants.

The grease research produced CALRESEARCH 159, a material of enhanced radiation stability. It is a selenide-inhibited alkylbiphenyl gelled with an aromatic salt. Promise of improved products was shown in work on new oils, e. g., tris(phenoxyphenyl)dodecylsilane; on new gelling agents, e.g., sodium N-p-tolylterephthalamate of mixtures of aromatic salts; and on new additives, e.g., N,N'-di-2-naphthyl-p-phenyl-enediamine.

Many formulations of various hydraulic fluids were evaluated before and after irradiation. The alkyl diphenyl ethers looked best in radiation stability and in thermal stability at 700°F. CALRESEARCH 216 evolved from the formulation work. It is an alkyl diphenyl ether inhibited with a selenide and thickened with a polybutene. Chemical inhibitors improved oxidation stability both before and after gamma irradiation. Aromatic hydrocarbon additives in MLO 8200 fluid reduced viscosity change and gassing caused by irradiation. This fluid was shown to be usable to about 10^{10} ergs/g C of gamma radiation. Original and irradiated aromatic base fluids operated satisfactorily in aircraft piston pumps at 275°F. Isothermal bulk modulus was found to decrease markedly in a gas-liquid system such as would prevail under irradiation.

The alkyl aromatics were found best as base materials for gas turbine oils. Esters, mineral oils, and polyglycols tested were deficient in radiation or thermal stability. CALRESEARCH 230 was developed in the work; it is a selenide-inhibited alkyl diphenyl ether containing a petroleum bright stock and other additives. The alkyl diphenyl ethers showed good oxidation stability before and after irradiation. Their tendency towards high coking and low lubricity was improved by additives. Irradiation lowered coking in these bases and also in alkylbiphenyls. In all base stocks, foaming increased after irradiation, with or without silicone being present. Coking was reduced by blending with high boiling materials, e.g., bright stocks. Certain oil soluble dyes showed synergism with the beneficial dialkyl selenides in oxidation tests.

The thermal stability of nine jet fuels (five JP-4's, three JP-5's, and an RP-1) was studied in the CFR coker. Original stocks and samples irradiated for three different levels were tested. Low level irradiation (0.8×10^{10} ergs/g C) impaired thermal stability. Higher level radiation (8×10^{10} ergs/g C) improved thermal stability. An equation was developed by which it was possible to predict increase in viscosity with irradiation at any dosage once a single dosage point was known.

WADC TR 54-576 Pt III.
EFFECT OF METALS ON LUBRICANTS. John B. Christian. May 1958. ASTIA Document No. 155518. PB 151446. Order from OTS $1.00.

This report shows the effects which an ester and a disiloxane have on various metals, and the effects which those metals simultaneously have on the fluids at elevated temperature (550°F). The disiloxane proved to be compatible with all of the metals tested, while the ester showed signs of being generally incompatible with all of the metals under the test conditions.

Clevite Research Center. WADC TR 57-61 Pt II.
LUBRICATION OF TITANIUM. Nicholas Fatica. AF 33(616)-3350. May 1958. ASTIA Document No. AD 155564. PB 131981. Order from OTS $2.25.

This investigation sought to obtain some information about the frictional properties of modified titanium coatings and to make a comparison of the wear resistance of the best surface treatments in the presence of various lubricants using the Shell Four-Ball Wear Tester and the Falex Tester.

It has been found that systems showing high wear rates in one tester will also show high wear rates in the other. If wear is expressed as volume or weight loss (W), it may generally be correlated with load (L) and time (t) by an expression of the form $W = KLT$ for either tester. The constant K includes the characteristics of the machine, the nature of the specimens and the nature of the lubricant. It is concluded that the essential nature of the action taking place in either tester is identical and that therefore any lack of correlation must be attributed to operation in widely different pressure and temperature ranges.

The correlation of wear rates with the frictional characteristics of the different systems was attempted in both testers with moderate success.

The reproducibility of the results for various systems is too poor to permit differentiating between the different titanium base alloys used in any particular treatment. As for the various surface treatments investigated, it appears that it is possible to obtain equivalent wear resistance in many cases using electroless nickel plates, cyanided, nitrided, carburized, oxidized, or carburized iron-plated titanium. Of these, oxidized and electroless nickel plated titanium fail at high loads. The use of conventional lubricants for any but electroless nickel and carburized iron-plated titanium is unreliable. Halogenated materials appear to be the best lubricants for the other treatments. It has been shown that the higher the halogen content of the lubricant, the better its lubricating properties.

Resin-bonded coatings fail at higher test temperatures.

WADC TR 57-299 Pt II.
NUCLEAR RADIATION RESISTANT LUBRICANTS. William L. R. Rice, 1/Lt., USAF. May 1958. ASTIA Document No. 155591.

This report presents a summary of current information on the state of the art of nuclear radiation resistant lubricant development. Data are presented on the effects of gamma radiation on a number of commercial fluids and lubricants, with a discussion of potential problem areas. Advanced materials for extremely high temperatures and radiation exposures are discussed, and the present status of preferred lubricants is outlined.

Of the new base materials for use in extreme conditions of temperature and radiation, the polyphenyl ethers show the most promise. Relatively low melting compounds can be prepared that undergo minor change in 500°F oxidation-corrosion tests and form little coke at temperatures as high as 800°F. Stability during in-source oxidation-corrosion tests has been shown to be good. The major drawback to use of the polyphenyl ethers is their low temperature limitation. At present, the majority of these materials melt at temperatures in the range 100°F to over 200°F.

Western Gear Corporation. WADC TR 57-773.
EVALUATION OF HIGH TEMPERATURE GEAR AND SPLINE LUBRICANTS. Donald J. Fessett, Charles W. Sauter. AF 33(616)-3539. June 1958. ASTIA Document No. AD 155670.

The load carrying ability of three lubricants, Dow Corning XF-258, General Electric Versilube F-50, and MIL-L-7808, was determining on spur and helical gears. Tests were conducted on 2.000 inch gears at speeds from 2500 rpm to 15,000 rpm and inlet lubricant temperatures from 165° to 700°F.

This report includes the results of these tests displayed in graph and table form, a discussion of these results, and conclusions. Also included in this report are test results previously obtained from WADC Universal Gear and Spline Lubricant Tester and the operating procedure to be used on the evaluation of gear and spline lubricants at high temperatures.

Pennsylvania State University. WADC TR 55-30 Pt VI.
FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. E. Erwin Klaus, Merrell R. Fenske. AF 33(616)-2851. June 1958. ASTIA Document No. 155569.

This report describes work carried out on a continuing project directed toward the development of improved hydraulic fluids and jet engine lubricants for use in the high temperature range of 400° to 700°F bulk oil temperatures. With few exceptions these studies are equally applicable to hydraulic fluid and jet engine oil development.

Extensive laboratory evaluation studies are presented for a series of mineral oils and hydrocarbons which have been super-refined by a pilot plant hydrogenation over nickel catalyst. Advantages of hydrogenated and exhaustively acid-refined mineral oils and hydrocarbons over conventionally refined mineral oils are demonstrated for application as base stocks for hydraulic fluids and lubricants. Completed hydraulic fluid formulations containing antifoam, antioxidant, and lubricity additives and jet engine oil formulations containing, in addition to these three additives, Acryloid and paraffinic resin dispersants are described. A silane, several esters, Spec. MIL-L-7808 fluids, and an improved lubricity silicone hydraulic fluid are evaluated critically in high temperature tests.

Properties of used samples of several jet engine oils evaluated in the J-57 jet engine are presented. These samples indicate more fluid deterioration than is predicted by Spec. MIL-L-7808 type stability tests. Several more severe oxidative tests, including the Petroleum Refining Laboratory thin film oxidation test and the isothermal and non-isothermal successive oxidation tests are described. These tests are designed to simulate more nearly the severity encountered in the J-57 engine test.

The effects of several variables, including fluid type, fluid molecular weight, additives, fluid flow rate, and air flow rate, on the high temperature single-pass lube rig are illustrated. Procedures for the analysis of exhaust gas from the single-pass lube rig have been established to place oxidation encountered in this test on a quantitative basis.

Bulk oil oxidation tests of high temperature fluids are shown for test temperatures of 347°, 500°, and 700°F.

The effect of fluid type and mineral oil class, degree of refining, and molecular weight are discussed for the 700°F tests. An evaluation of the character of the volatile products of the 700°F oxidation tests is presented.

Wear and lubrication studies in the four-ball testers are used to illustrate the additive effects of silicones, silicates, thiocarbamates, and diisopropyl acid phosphite in ester and mineral oil fluid types.

Panel coker data in the Model C and controlled atmosphere units are compared and summarized for the effects of fluid type, degree of refining, molecular weight, and additives.

The construction of an evaporation unit of the type used in the Federal Test Method Standard No. 791 is described.

Some additional miscellaneous duties have been performed at the request of the Wright Air Development Center. These duties include: (1) distribution of Laboratory reports, (2) formulation of fluids, (3) distribution of standard fluid samples and, (4) evaluation of specific fluid properties.

Coordinating Research Council, Inc. WADC TR 57-36 Pt II.

AIRFRAME LUBRICANTS - Part II. Development of a Laboratory Technique for Determining Rust-Preventive Properties of Lubricating Greases. Airframe Lubricants Group. AF 33(616)-2888. July 1958. ASTIA Document No. 155726. PB 151184. Order from OTS $1.50.

This final report on rust-preventive properties of lubricating greases describes the work of CRC laboratory and service evaluation panels in their objective to develop test techniques for predicting the performance in actual service of airframe and accessory lubricants. A description is included of the three test techniques developed and the reasons for ultimately recommending one of them, the Research Technique for Determining Rust-Preventive Properties of Lubricating Greases in the Presence of Free Water (CRC Designation L-41-957).

Esso Research and Engineering Company. WADC TR 57-255 Pt II.

NUCLEAR RADIATION RESISTANT TURBINE ENGINE LUBRICANTS. Alfred H. Matuszak. AF 33(616)-3181. July 1958. ASTIA Document No. AD 155736.

Work performed under Contract No. AF 33(616)-3181 on the development of radiation resistant turbine engine lubricants for possible use in aircraft nuclear power plants is described. This is a continuation of work reported in WADC TR 57-255 and completes the screening and evaluation of over one hundred mineral and synthetic materials consisting of finished lubricants (Phase I) and inhibited lubricant base stocks (Phase II). These materials were irradiated at the MTR and at Hanford under static conditions using a screening dosage of about 10^{10} ergs/gm C (10^{8} roentgens) and a dose rate of about 3×10^{7} ergs/gm C/hr. Extended irradiations were done at 5 and 10×10^{10} ergs/gm C on the more promising lubricants.

In the Phase I work finished mineral oils generally showed greater retention of original lubricant properties at 10^{10} ergs/gm C than did the finished synthetic oils. Of the mineral oils investigated a Paraffinic Grade 1065 Aviation Oil meeting MIL-L-6082B requirements but inhibited with phenyl-alpha-naphthylamine was the most promising. Foaming was a problem at 10^{10} ergs but not at higher dosages. Lubricant degradation in general was more severe, however, at the higher levels. The most serious deficiencies resulting from the irradiation of the Grade 1065 oil over the range 10^{10} to 10^{11} ergs/gm C were its viscosity increase and flash point decrease. Metal corrosion was negligible. Overall, the Grade 1065 Aviation Oil is indicated to possess reasonably good resistance to radiation under static conditions to dosages between 10^{10} and 5×10^{10} ergs/gm C, and if viscosity and flash point are not limiting, to possibly 10^{11} ergs/gm C. Although the changes noted are not considered excessive except for viscosity increase and flash point, dynamic in-source testing will be necessary to establish the usefulness of this oil as a nuclear powered aircraft lubricant.

In the Phase II mineral base stock work wherein 19 base stocks or blends were screened during the second year an inhibited paraffinic base stock looked the most attractive. It paralleled very closely the performance of the Paraffinic Grade 1065 Aviation Oil. Hydrogenation of this as well as a more viscous paraffinic bright stock did not effect improvements in stability toward radiation and oxidation. The addition of stable aromatic base stocks to the paraffinic base resulted in several expected improvements. As in the case of the Grade 1065 Oil, dynamic testing will be required to evaluate this oil further. Other stable base stocks were Kenflex B and Philrich 5. These are viscous aromatic hydrocarbon oils which may be useful as blend components with other attractive oils.

In the more recent Phase I work on "finished" synthetic oils thirteen blends newly formulated from attractive base stocks and a MIL-L-25336 approved oil were screened as possible Phase II type oils. Three oils showed promise. Two of these were di-octyl sebacate based oils containing Plasticizer HM (an aromatic polyester) as a stabilizer against radiation. One contained TCP. These showed moderately good radiation and oxidation stability at the 10^{10} ergs/gm C. Radiation at higher dosages unfortunately was not carried out. This will be necessary to determine their value as more stable radiation resistant lubricants. The third oil, an all aromatic blend containing Cabflex HS-10 and Kenflex B, likewise showed good radiation and oxidation stability. It is comparable with the paraffinic mineral oils in many respects. It shows less viscosity change but tends to be slightly corrosive as a result of the greater acidities developed only during higher radiation dosages.

In the Phase II base stock evaluation program aromatic esters were generally more stable than aliphatic diesters, complex esters, glycols or polyalkylene glycol monoethers. Of the thirty synthetic base stocks screened in the more recent work Cabflex HS-10 (an alkyl-aryl phthalate), isodecyl naphthalate and Plasticizer HM were the most stable. Although these materials showed less viscosity increase than the mineral oils they exhibited somewhat greater changes in flash point, acidity and corrosivity. The changes noted after irradiations up to 5×10^{10} ergs/gm C would not be considered prohibitive however as far as lubricant performance is concerned. Some question would arise as to their usefulness at 10×10^{10} ergs/gm C, however.

It is recommended the oils mentioned above be further evaluated in dynamic in-source tests to more clearly define their usefulness as radiation stable lubricants.

E. F. Houghton & Company. WADC TR 58-288 Pt I.

DEVELOPMENT OF HIGH TEMPERATURE, HEAVY LOAD-CARRYING GREASES. Robert K. Smith, William Eismann, Jr. AF 33(616)-5156. August 1958. ASTIA Document No. AD 155855.

The object of this study has been to develop greases which are capable of lubricating heavily loaded (50,000 to 75,000 psi.) - rod ends, pulleys, anti-friction bearings, sliding surfaces of hinge components, gear boxes, and

plain bearings subjected to oscillatory motion. The temperature range over which these greases are to operate is from -65°F to 425°F. Greases were prepared from both conventional and recently developed silicone and ester oil base fluids thickened by Bentone 38 or aryl substituted urea materials. Various blends of these base fluids have been formulated into greases to obtain optimum lubricity, thermal stability and good low temperature characteristics.

A number of silicone oils were heat treated with a wide range of metal salts in an effort to improve one or more of the above properties. Greases prepared from the treated fluids did not show any significant improvement over those greases made with the untreated base fluids.

The incorporation of commercially available extreme pressure additives resulted in excessive corrosion at high temperatures. In order to meet the heavy load-carrying requirements, a new class of extreme pressure additives has been developed based on pentachlorobenzene thiol and pentachloromercapto acetic acid and their respective derivatives.

Several of these derivatives in 3 to 6% concentration tripled the load-carrying capacity of silicone and silicone-ester base fluid greases as shown by Mean Hertz Load values as high as 98.

Shell Development Company.
WADC TR 57-177 Part II.
ENGINE OIL DEVELOPMENT. C. Lynn Mahoney, Emmett R. Barnum, W. S. Saari, William W. Kerlin, Karl J. Sax, P. H. Williams. AF 33(616)-3182. August 1958. ASTIA Document No. AD 155862.

Conventional ester and petroleum hydrocarbon based lubricants, depending on inhibitors for resistance to oxidation, are very sensitive to radiation damage and lack sufficient stability for high temperature use. Selected aromatic compounds, designed for stability in absence of inhibitors, appear to be capable of withstanding considerably higher temperatures and radiation levels than present oils. Aromatic esters, carbonates, silanes and polyphenyl compounds have been investigated but polyphenyl ether derivatives have shown the greatest over-all promise.

The unsubstituted polyphenyl ethers are very stable materials. They undergo little change in oxidation-corrosion tests at 500°F and form only small amounts of coke in panel coke tests at 900°F. Their initial thermal decomposition temperatures are over 800°F. Physical property changes are moderate at radiation levels of 10^9 rad. Their wear characteristics are very much better than most other classes of highly aromatic compounds.

Alkyl substituents generally lower the melting points of polyphenyl ethers but only carefully selected groups, such as the -cumyl- and tertbutyl-, can be used without seriously affecting the stability. These alkyl-substituted polyphenyl ethers are not as stable as the corresponding unsubstituted ethers but undergo little change in 475°F oxidation-corrosion tests, form little coke at 800°F and have initial thermal decomposition temperatures of 700 to 770°F. Physical property changes in these alkyl substituted polyphenyl ethers are also moderate at radiation levels of 10^9 rad but radiation greater than 2.5×10^8 rad seriously lowers their resistance to oxidation.

Several methods of lowering the melting points of polyphenyl ethers are being investigated at the present time. Initial results with isomerization procedures have been very promising. Several stable liquid alkyl substituted ethers have been prepared. Additional work with alkyl derivatives is planned and some effort is being made to lower the melting points of unsubstituted ethers through use of isomerization reactions.

Shell Development Company
WADC TR 56-370 Part III.
RESEARCH IN HIGH TEMPERATURE BEARING LUBRICATION IN THE ABSENCE OF LIQUID LUBRICANTS. R. A. Coit, S. S. Sorem, R. L. Armstrong, C. A. Converse. AF 33(616)-2999. August 1958. ASTIA Document No. AD 155856.

A method of operating rolling contact bearings at high temperatures without using conventional liquid lubricants has been evolved. This method employs a "protective atmosphere" around the bearing. The atmosphere has two functions, the prevention of the formation of abrasive oxides at the bearing surfaces and the formation, by reaction with the bearing surfaces, of antiscuffing films to prevent catastrophic wear at sliding contacts. Factors involved in the selection of atmosphere compositions and bearing materials for use with this method of operation are discussed. Test operations which have demonstrated the performance of this system at various temperatures from 600 to 1000°F are described.

WADC TR 58-15.
THE EFFECT OF STRUCTURE OF POLYHYDRIC ALCOHOL ESTERS ON THEIR SUITABILITY AS HIGH TEMPERATURE LUBRICANTS. Harold W. Adams, William L. R. Rice. September 1958. ASTIA Document No. AD 155870.

The research and development effort discussed in this report concerns the feasibility of using esters of the neopentyl type as lubricants up to 400° to 500°F.

The absence of the beta hydrogen on the alcohol moiety has, in previous investigations, resulted in superior thermal stability of the esters of such alcohols. This increase in thermal stability appears to be in the range of approximately 100°F. This is accomplished by forcing thermal degradation to proceed through free radical formation.

Although these neopentyl type esters have improved thermal stability over conventional diester blends, they do not necessarily posses superior oxidation stability. The use of neopentyl type ester fluids in an oxidizing environment at 400° or 500°F will definitely depend upon the development of an efficient oxidation inhibitor.

The over-all assessment of the chemical and physical properties of the neopentyl type ester fluids shows considerable promise for high temperature application.

Midwest Research Institute. WADC TR 58-297. DEVELOPMENT OF EVALUATION TECHNIQUES FOR DETERMINATION OF THE LUBRICITY AND STABILITY OF NEW HIGH-TEMPERATURE LUBRICANTS AND HYDRAULIC FLUIDS. Vernice Hopkins, Andrew D. St. John. AF 33(616)-5202. October 1958. ASTIA Document No. AD 203382.

A Shell four-ball wear tester has been modified for operation up to 800°F. A lubricant circulating system was included in the modification, and comparable noncirculating and circulating lubricant evaluation programs were completed. Silicone fluids and silanes neat and with anti-wear additives, a mineral oil, pentaerthritol ester and disiloxane fluids and lubricants were evaluated at 167°F, 400°F, and 700°F, 10 kg. and 40 kg. loads, and 600 rpm and 1,200 rpm spindle speeds. Wear scar, frictional torque, and wear rate data were recorded. The lubricants have been rated relative to each other by applying the results of a statistical analysis performed on the wear scar data.

A partially completed analysis of the energy dissipation and heat transfer in a roller bearing is reported. The analysis will ultimately provide scaling relations to be used in the design of a bearing temperature stabilisation tester. The major simplifying assumptions and results may be listed as follows:

The lubricant is assumed to have constant viscosity

and density. Also, the lubricant flow through the rolling-slipping contacts is calculated without consideration of the local body distortions. The friction force which occurs as the result of direct contact between bearing elements is described with a constant coefficient. The extent of the physical contact is dependent on the load carried directly (rather than hydrodynamically) and the statistical description of the surfaces.

Equations of motion for the bearing assembly are formulated for pure hydrodynamic lubrication and for a combination of hydrodynamic and direct contact situations. The equations are solved for the pure hydrodynamic case using the assumption of constant roller and cage speeds. The assumption of constant roller spin is found inadequate for those cases in which sizeable loads are carried by direct contact and no proper solutions for this case are reported.

In preparation for heat transfer calculations, the lubricant streamlines are obtained in a generalized form as are the lines of constant dissipation. The heat transfer problem is introduced.

A survey of machine components which required extreme pressure lubrication was performed. The functional characteristic of these components were compared to the test elements of extreme pressure lubrication testers. As a result of this comparison and the limited operational temperature range of existing testers a new extreme pressure lubricant tester was designed for operation up to 700°F. This new tester possesses more flexibility than existing testers. The test results should correlate better with performance data as the conditions of testing may be selected nearer to the operating conditions of a machine component.

An evaluation facility has been designed in which accessory components may be mounted and used as test rigs for the practical evaluation of lubricants and hydraulic fluids. A variety of types and sizes of components may be tested over a temperature range of -100°F to +800°F. This facility consists of a 5 ft. by 5 ft. by 5 ft. environmental chamber, 50 h.p., 12,000 rpm variable speed, mechanical drive, and AC and DC power supplies for various components.

Gulf Research & Development Company.
WADC TR 58-350.
DEVELOPMENT OF GREASES FOR HIGH SPEED BALL AND ROLLER BEARINGS. Paul R. McCarthy, Guy C. Blewett, Joseph J. McGrath. AF 33(616)-5020. October 1958. ASTIA Document No. AD 203388.

Performance life of ten greases approved under Specifications MIL-L-7711, MIL-G-3278 or MIL-L-3545 was determined in a modified Pope Spindle Tester at 20,000 rpm. and at the temperatures shown below:

TYPE OF GREASE	TEST TEMPERATURES, °F
MIL-L-7711	250 and 350
MIL-G-3278A	250 and 350
MIL-L-3545	300 and 350

The general compositions of the specification greases evaluated are as follows:

TYPE OF GREASE	GENERAL COMPOSITION
MIL-L-7711	Fluid Component:
MLG 57-233(a)	(a) Polyalkylene glycol
MLG 57-234(b)	(b) Petroleum Oil
	Thickener:
	(a) Lithium soap
	(b) Sodium-Calcium soaps
MIL-G-3278	Fluid Component:
MLG 57-235(a)	(a) Diester
MLG 57-236(b)	(b, c, and d)
MLG 57-237(c)	Diester-Petroleum Oil
MLG 57-238(d)	Thickener:
	(a, b, c and d)
	Lithium soap
MIL-L 3545	Fluid Component:
MLG 57-239(a)	(a, b, c and d)
MLG 57-240(b)	Petroleum Oil
MLG 57-241(c)	Thickener:
MLG 57-242(d)	(a) Lithium soap
	(b, c, and d) Sodium soap

Consistency, dropping point, oil separation, evaporation, shear stability and oxidation stability tests were run on the same greases in an attempt to correlate properties of the greases determined by such tests with performance life in bearings.

Both commercially obtainable as well as new experimentally prepared fluids and thickeners were evaluated as components for improved high temperature greases. Of the fluids, the silicones showed the best thermal stability; 5,5-diphenylhydantoin proved to be the best thickener. Experimental greases containing combinations of silicones and 5,5-diphenylhydantoin, in general, gave the longest performance life at 400°F and 20,000 rpm. All experimental greases, however, failed to consistently meet the target requirement of 500 hours minimum life under the foregoing conditions. The longest single run (891 hours) at 400°F and 20,000 rpm was obtained with an arylurea grease "RLG 216-54".

A high speed bearing test ring, believed to be capable of operating under the following conditions, was designed:

Speed: DN value of 0.9×10^6 using ball bearings of 20 and 25 mm. bore
Temperature: Ambient to 600°F.
Radial Load: Up to 75% of the rated capacity of the bearings in the test section at the maximum speed of 45,000 rpm.

Midwest Research Institute. WADC TR 57-455 Pt II.
HIGH TEMPERATURE SOLID DRY FILM LUBRICANTS. Melvin T. Lavik. AF 33(616)-3684. October 1958. ASTIA Document No. AD 203121. PB 151423. Order from OTS $2.25.

Solid dry film lubricants appear to be the answer for many high temperature lubrication problems. However, the wear-life of these films is dependent upon a large number of factors which complicate the choice of a particular lubricant film and its proper application. This report describes three phases of work directed toward the development of high temperature solid dry film lubricants.

(1) Parametric study of wear-life factors: The fractional factorial design of experiments (total of five) is used to evaluate the effect of ten factors upon the wear-life of several solid film lubricants. The factors studied are environmental temperature, bearing load, substrate hardness, film thickness, relative linear surface speed, lubricant material, film binder, lubricant-to-binder ratio, type of motion, and geometry of rub shoe. In the range of values investigated, the temperature (400-550°F) and the load (10,000 to 65,000 psi) had the greatest effect upon the wear-life of the lubricant films. Although the results are limited, a procedure is developed for evaluating a lubricant (for a particular application) with a minimum number of tests.

(2) Nuclear radiation effects: Four commercial solid dry film lubricants were submitted to gamma and neutron irradiation. Three of the films were resin-bonded mixtures of graphite and molybdenum disulfide. The other film was a mixture of lead oxide and graphite bonded with a glass-like material. Only the gamma irradiated specimens have been tested. The wear-life of one lubricant film is possibly affected by gamma irradiation; the others

showed no significant effect.

(3) Search for high temperature lubricants: A group of 16 inorganic materials have been selected for further investigation as possible high temperature (800°F) lubricants. This selection was made on the basis of a literature survey and the present knowledge of lubrication mechanisms. These materials are simple compounds and are mostly sulfides, oxides and chlorides of metals occurring in periods 3, 4, or 6 of the periodic table.

Southern Research Institute. WADC TR 58-283. MEASUREMENT OF VISCOSITIES OF GAS-TURBINE LUBRICANTS AT ELEVATED TEMPERATURES AND PRESSURES. Jack S. Barr. AF 33(616)-3900. October 1958. ASTIA Document No. AD 203642.

A high-pressure viscometer to measure the viscosities of some aircraft lubricating oils at elevated temperatures and pressures was constructed. The equipment was calibrated, but viscosities of the various oils will be determined on a subsequent contract.

A maximum pressure of 188,000 psi was obtained during a test of the equipment. The apparatus was designed for tests at temperatures from ambient to 400°F.

WADC TR 54-576 Pt IV. THE EFFECT OF METALS ON LUBRICANTS. John B. Christian. December 1958. ASTIA Document No. AD 155716.

The qualitative effects which several aircraft fluids and lubricants have on the surface of various metals at room temperature and at 400°F for periods up to 528 hours are reported.

Sixteen different metals were cut into one-inch square specimens and stored with aircraft fluids and lubricants in capped bottles under the above conditions. Each was observed at the end of 48 and 96 hours, and every 72 hours thereafter until the end of 528 hours.

Qualitatively, all metals, with the exception of lead, and fluids were compatible at room temperature up to 528 hours, but at 400°F compatibility generally ceased at 384 hours.

Heyden Newport Chemical Corporation. WADC TN 58-248. INVESTIGATION OF NEOPENTYL POLYOL DERIVATIVES AS BASE FLUIDS FOR HIGH TEMPERATURE TURBINE ENGINE OILS. Adolph J. Deinet. AF 18 (600)-1667. December 1958. ASTIA Document No. AD 206669.

Forty-four esters, ether esters, and polyesters based on hexa, tetra, tri, and di methylol neopentyl alcohols were prepared. Of these, twenty were submitted to Wright Air Development Center for bench screening. This report tabulates data obtained on these compounds.

WADC TR 58-499. THE EFFECTS OF NUCLEAR RADIATION ON SOLID FILM LUBRICANTS. William L. R. Rice, William L. Cox, 1/Lt. January 1959. ASTIA Document No. AD 207795.

The effect of nuclear radiation on twelve commercial solid film lubricants was determined. The lubricants were typically phenolic or epoxy resin bonded, incorporating graphite or molybdenum disulfide. Gamma exposures covered the range 8.71×10^9 through 2.61×10^{11} ergs per gram carbon. Neutron exposures were over the range 1×10^{15} to 3×10^{16} nvt fast.

Data were obtained on the wear life, corrosion resistance, fluid resistance, and thermal stability. It was observed that radiation, in certain instances, improved wear life rather than decreasing it. Also, those solid films that possessed good corrosion and fluid resistance and good thermal stability prior to irradiation generally were not seriously changed in these properties. Films of poor initial properties were greatly degraded by the radiation.

It was concluded that certain of the films should be usable up to the maximum dosages used, but that careful selection would be necessary.

WADC TR 59-99. DEVELOPMENT OF GREASES AND SEALS FOR 275°F PNEUMATIC SYSTEMS. A. A. LePera, John Q. Griffith. May 1959. ASTIA Document No. AD 214425.

Evaluation methods, including functional tests, were devised to determine grease and elastomer compatibility in a pneumatic system operating at -65 to 275°F and 0 to 3000 psi pressure range.

O-rings fabricated from Buna-N and Butyl rubber compounds lubricated by a commercially available silicone grease, operated successfully under the environments desired.

The compatibility of these materials at temperatures in the 165° to 275°F range relieves design limitations that had hitherto been encountered in operational pneumatic systems.

E. F. Houghton & Company. WADC TR 58-288 Pt II. DEVELOPMENT OF HIGH TEMPERATURE HEAVY LOAD-CARRYING GREASES. Robert K. Smith, William Eismann, Jr. AF 33(616)-5156. May 1959. ASTIA Document No. AD 214615.

The purpose of this study was to develop high temperature greases capable of lubricating heavily loaded surfaces moving against each other in sliding, oscillatory, rolling and rotational motion. Examples of such surfaces are found in actuators, gimbal rings, rocket devices, control mechanisms, hinge pins and rod end bearings.

The development of an extreme pressure grease with a -65°F to 425°F operating capability was completed. Two arylurea thickened greases have successfully completed all the parameter screening tests. One grease contains a methyl phenyl silicone base fluid while the other has a blended base fluid of a methyl phenyl silicone with a high molecular weight ester oil. Pentachlorophenyl mercapto acetic acid is the extreme pressure additive utilized in both greases. Functional testing is now being conducted in heavily loaded bearings and actuators.

Work was initiated to obtain extreme pressure grease systems capable of operating in the temperature ranges of -40 to 500°F and 0 to 600°F. Presently there is no oil commercially available which will operate successfully for prolonged periods at temperatures above 450°F. There are, however, several experimental fluids which show promise for high temperature use in the near future. Various silicone, ester and mineral type oils with relatively low volatility and high flash points were employed to evaluate the thickening ability of the experimental high temperature materials. The following types of materials were investigated for use as high temperature grease thickeners-graphite, urethanes, imidazolidines and imides of both aromatic and aliphatic dibasic acids. One type of thickener developed - the aluminum complex of pyromellitimides - has melting points above 900°F and shows excellent thickening properties.

Western Gear Corporation. WADC TR 57-773, Pt II.

EVALUATION OF HIGH TEMPERATURE GEAR AND SPLINE LUBRICANTS. Donald J. Fessett. AF 33(616) 5631. May 1959. ASTIA Document No. AD 214614.

The load carrying abilities of the following seven lubricants were determined on 16-pitch 2,000 in. pitch diameter spur gears:

Octadecyl tri (decyl) silane
MLO 57-426 -- Pentaerythritol tetracaproate
MLO 58-431 -- 10% silicone, 90% pentaerythritol tetracaproate
MLO 7379 -- Super refined bright stock
MLO 7380 -- Super refined paraffinic neutral
MLO 7381 -- Super refined paraffinic neutral with 0.2% alkyl acid phosphite
MLO 7383 -- Super refined naphthenic mineral oil

Tests were conducted at speeds up to 20,000 rpm and lubricant temperatures to 600°F. Octadecyl tri(decyl) silane lubricant exhibited the best load carrying ability of 2500 lb/in. of face width at 10,000 rpm and 600°F. MLO 58-431 and MLO 7379 ranked second and MLO 7381 third in overall mechanical performance. MLO 7383 scored at a higher load at 600°F than at 400°F. The load carrying ability of MLO 57-426 decreased the greatest with increasing temperature from 400 to 600°F.

Modifications made on the Universal Gear and Spline Tester included a more efficient test lubricant heating system, a temperature controller and recorder, an electronic speed indicator and an optical system. Operating procedure of the modified tester is included in the appendix.

Coordinating Research Council, Inc. WADC TR 57-36, Part IV.

AIRFRAME LUBRICANTS, PART IV Status Report. AF 33(616)-5684. June 1959. ASTIA Document No. AD 225900.

This report reviews the activities of the various projects undertaken to assist the Air Force in conducting research on the problem of mutual adaptation of airframe lubricants and equipment, with particular emphasis on the work carried out during the period from April 1958 to December 1958. This report, describing work on five phases of activity (high-temperature testing of greases in anti-friction bearings; fretting corrosion; sliding motion under show, highly-loaded, high temperature conditions; bonded solid film lubricant coatings; and aircraft powerplant bearings and their lubrication), discusses the scope and objective of each, the work of the panels, the conclusions reached, and plans for future work.

Midwest Research Institute. WADC TR 58-297, Part II.

DEVELOPMENT OF EVALUATION TECHNIQUES FOR DETERMINATION OF THE LUBRICITY AND STABILITY OF NEW HIGH-TEMPERATURE LUBRICANTS AND HYDRAULIC FLUIDS. Vernice Hopkins, Andrew D. St. John. AF 33(616)-5202. June 1959.

A series of torque temperature tests were performed on the modified Shell Four-Ball wear tester by increasing the temperature about 10°F/min while the other conditions were kept constant. A sharp increase in frictional torque was experienced for most of the lubricants at temperatures ranging from 400 to 700°F. Conventional wear tests were made on a silicone (MLO 56-839) an ester (MLO 57-427), and an ether (MLO 58-432) at temperatures of 400, 500, 600 and 800°F at loads of 4, 10, and 40 kg., and spindle speeds of 600 and 1,200 rpm. Wear scar, frictional torque, and wear rate data were recorded. The lubricants were rated relative to each other by applying the results of a statistical analysis performed on the wear scar data. These three lubricants and the six lubricants evaluated in the earlier initial phase of the program are compared to each other on a relative basis.

An analytical study is being performed in the development of scaling equations which are to be used as the basis of design for a bearing temperature stabilization tester. This analysis is nearly completed. The general approach is presented together with results on the bearing position, energy dissipation mechanisms and distribution of dissipation. The heat transfer analysis is described and a tentative numerical procedure for the solution of the heat transfer equations is presented in detail.

An extreme pressure lubricant tester has been constructed which is capable of running tests at temperatures up to 700°F. The construction of this new tester was based on the design described in WADC TR 58-297.

Construction of the environmental chamber system recommended in the initial phase of the program was started by purchasing a 5 ft. x 5 ft. x 5 ft., -100°F to /800°F environmental chamber. Midwest Research Institute erected a building at their Deramus Field Station which is to be used as a lubricant evaluation laboratory. This laboratory will house the environmental chamber system, the WADC high temperature hydraulic fluid test stand, and other small lubricant testers. Both the environmental chamber and the WADC high temperature hydraulic fluid test stand were installed in the new lubricant evaluation laboratory.

Gulf Research & Development Co. WADC TR 58-350, Part II.

DEVELOPMENT OF GREASES FOR HIGH SPEED BALL AND ROLLER BEARINGS. Paul R. McCarthy, Guy C. Blewett, Joseph J. McGrath. AF 33(616)-5020. June 1959. ASTIA Document No. AD 220014.

Work was continued towards the development of greases capable of lubricating, for a minimum period of 500 hours, antifriction bearings operating at 20,000 rpm and 400°F. Approximately 60 materials were synthesized or obtained from other sources for trial as thickeners, antioxidants or fluids for greases. Of the experimental greases tested at 20,000 rpm and 400°F, those containing as thickeners spiro (hydantoin-5,1'-indan) and 5,5-diphenylhydantoin performed satisfactorily for the longest periods (304 to 690 hours). In performance tests at 10,000 rpm and 400°F, greases containing as thickeners 4,4'-biantipyrine, 4,4'-biphenyldicarboxylic acid, disodium tetrachlorophthalate, disodium tetraphenyl-phthalate, and benzoguanamine showed good promise.

A test program covering the evaluation of two types of bearings were initiated. This was done in an attempt to improve the repeatability of life performance tests.

All drafting work on the 45,000 rpm tester was completed. Construction of the tester is in progress. Orders were placed for accessory equipment required for operation of the tester.

Construction of a modified spindle, intended for a study of the effects of bearing fits on performance life, is in progress.

Petroleum Refining Laboratory. WADC TR 55-30, Part VII.

FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. E. Erwin Klaus, Merrell R. Fenske. AF 33(616)-5460. June 1959.

This report describes work carried out on a

continuing program to characterize the behavior of various base stocks and fluid formulations for application as hydraulic fluids and/or jet engine lubricants in the temperature range of 350°F. Esters, mineral oils, and hydrocarbons are evaluated for use as base stocks. The use of selected structures is shown to result in improvement in thermal stability and/or low temperature properties for esters and polyolefins. For mineral oils and hydrocarbons, the emphasis is on the achievement of improved additive response by super-refining, optimum viscosity-volatility properties by close cut fractionation, and improved viscosity-low temperature relationships by deep dewaxing. Oxidation tests of bulk oil, thin film, deposition, and successive (contamination) types are critically compared and contrasted for a series of super-refined and conventional mineral oils containing various additive packages. The additive package ranges from a single additive to an integrated package designed to control oxidation, corrosion, dirtiness, foaming, lubricity, low temperature fluidity, and viscosity level. Four low molecular weight, shear stable polymeric thickeners have been evaluated and compared with the conventional polymeric thickeners. Two of these materials provide multifunctionality as dispersants and pour depressants as well as thickeners. Used jet engine oils (silicones, mineral oils, and esters), from J-57 engines run at 300° and 350°F oil-in have been evaluated for changes in physical and overall stability properties. Quantitative analysis of the used samples for oxygenated groupings provides an estimate of the extent of oxidative deterioration. The degree of oxidation of the used engine oils is compared with the severity of various types of specification and laboratory tests. A modification of an experimental high temperature hydraulic fluid which would offer rust protection is proposed for evaluation as a missile hydraulic fluid. Fuel dilution has been explored as a method to improve low temperature fluidity of mineral oils.

Midwest Research Institute. WADC TR 59-166.
HEAT CAPACITY DETERMINATION OF MINERAL AND SYNTHETIC ENGINE OILS, LUBRICANTS, FUELS AND HYDRAULIC FLUIDS IN THE TEMPERATURE RANGE 70° - 500°F. T. M. Medved, C. E. Hensen, C. C. Bolze, J. W. Barger. AF 33(616)-5269. June 1959. ASTIA Document No. AD 225705. PB 161030. Order from OTS $1.75.

The heat capacities of 33 mineral and synthetic engine oils, lubricants, fuels, and hydraulic fluids were measured over a temperature range of 70° to 500°F.

Calorimeter constants were obtained by internal standardization foregoing the use of a standard liquid.

The calculated errors in the final results were 3 to 5 per cent.

A comparison of latent heats of vaporization of phenyl ether samples is included.

Midwest Research Institute. WADC TR 57-455, Part III.
HIGH TEMPERATURE SOLID DRY FILM LUBRICANTS. Melvin T. Lavik. AF 33(616)-3684. June 1959.

Considerable progress has been made in the research and analysis leading to the development of a high temperature dry film lubricant. The chemical stability and coefficient of friction have been determined for a selected group of materials. Changes in weight, color and physical appearance were noted as the selected materials are heated to 1000°F in air and a special pellet friction machine was used to determine their coefficients of friction at these temperatures while under light loads.

Ceramic materials, stable at high temperatures, have been considered as possible dry film bonding agents. Silicon dioxide films have been prepared with and without solid lubricants from a hydrolyzed tetraethyl orthosilicate solution. Similar films were prepared using boric oxide. Ceramic materials used appear to be compatible with some lubricants, yielding low coefficients of friction and reasonable wear-lives.

Samples of five commercial lubricants have been irradiated at levels up to 10^9 roentgens (gamma) and 3×10^{16} NVT (neutron). Wear-life tests on the irradiated samples, although not conclusive, indicate that the radiation has little permanent effect on the lubricative properties of the films.

Midwest Research Institute. WADC TR 59-48.
MODIFICATION OF SHELL FOUR-BALL WEAR TESTER FOR 800°F OPERATION AND DEVELOPMENT OF A LUBRICANT EVALUATION TECHNIQUE. Vernice Hopkins. AF 33(616)-5202. June 1959. ASTIA Document No. AD 215447. PB 145451. Order from LC, Mi $3.90, Ph $10.80.

The modifications made to adapt the Shell four-ball wear tester for 800°F operation have been outlined. Detailed discussions of the design changes, the frictional torque measuring setup, lubricant circulating system, and the application of additional controls and instrumentation are presented. The evaluation of the performance of the modified wear tester is described. Operating test procedures for a conventional test and the alterations required for lubricants solid at room temperature and for lubricants circulating through the ballpot assembly are included. Testing experiences are discussed, especially for high temperature tests. A method for evaluating lubricants is outlined.

Stanford Research Institute. WADC TR 58-335, Part II.
SYNTHESIS AND EVALUATION OF HIGH TEMPERATURE ANTI-OXIDANTS FOR SYNTHETIC HYDRAULIC FLUIDS AND LUBRICANTS. Robert M. Silverstein. AF 33(616)-5276. June 1959.

During the period covered by the current report, candidate antioxidants have been screened in the following lubricating fluids: F-50 silicone fluid, silane fluid MLO 57-628, naphthenic mineral oil MLO 57-573, and pentaerythritol tetracaproate MLO 57-426.

A number of additives were found to be effective in retarding high temperature oxidative degradation of F-50 silicone fluid. Condensed aromatic ring structure containing three or more rings were effective as a class. The additives of choice were determined on criteria of effectiveness, solubility and safety. These additives were sufficiently soluble in the fluid at low temperatures to warrant their consideration for practical use. The mechanism of inhibition of oxidative degradation was studied.

None of the additives evaluated was effective in any of the other fluids.

Clevite Research Center. WADC TR 57-61, Part III.
LUBRICATION OF TITANIUM. Nicholas Fatica. AF 33(616)-3350. July 1959. ASTIA Document No. AD 227178.

The adaptation of the crossed-cylinder specimen concept in a bench tester removed most of the problems associated with specimen preparation in the investigation of various titanium-lubricant systems. It is shown that the crossed-cylinder test results correlate closely with the Shell-Four-Ball test results.

The crossed-cylinder tester was used to evaluate the wear resistance of cyanided and electroless-nickel

plated specimens of six titanium alloys. The results show that electroless-nickel plated titanium, using conventional lubricants, is superior to the best cyanided titanium systems studied. Significant differences are shown between the wear resistance of the six nickel plated alloys in the same lubricant, and the same may be said for the various cyanided titanium alloys.

The best lubricants to use with cyanided titanium alloys are the same as for the untreated alloys; Halocarbon 11-14 is generally superior to polypropylene glycol 1025, while the lubricants normally used for steel and electroless nickel are definitely inferior of these two.

The comparison of wear rates for two metals having widely different moduli of elasticity has been considered, and a method of handling the problem proposed. On the basis, it is shown that the wear rate of the best electroless nickel-plated titanium alloy is equivalent to 52100 steel.

Carburized iron-plated titanium was also investigated to a limited extent and found to give results approaching 52100 steel.

University of Michigan. WADC TR 59-185.
THERMAL CONDUCTIVITY OF LUBRICATING OILS AND HYDRAULIC FLUIDS. D. W. McCready. AF 33(616)-5745. July 1959.

An all-metal concentric cylinder type of thermal conductivity cell was used to measure the thermal conductivity of twelve natural and synthetic base lubricating fluids.

Thermal conductivity values in the temperature range of from 70 to 500°F are reported for fluids considered stable to the higher temperature. The maximum temperatures for other fluids were limited by their instabilities under test conditions. Since each fluid has individual characteristics, no correlation of conductivity values appears possible. Values are considered precise and for possible correlation can be compared to those of a fluid chosen as a "standard reference".

Midwest Research Institute. WADC TR 59-203.
HIGH TEMPERATURE METALLO-ORGANIC AND INORGANIC BASE FLUIDS. L. W. Breed, F. V. Morriss. AF 33(616)-5718. September 1959. ASTIA Document No. AD 229676.

A literature retrieval program was initiated to aid in guiding research on high temperature lubricants. A group of about 22,250 IBM punch-cards has been prepared, each card representing a different inorganic or metallo-organic compound. For each compound one card was prepared which included the literature source, the empirical formula, formula weight, structural codes, and the boiling point, melting point, liquid range, and thermal stability, provided one or more of the last four items were known. These data were obtained from an extensive search of the chemical literature and Government research reports. Preliminary analysis of the data is described as well as a discussion of the limitations of data correlation programs with particular emphasis on their application in this program.

WADC TR 59-191, Part I.
RESEARCH AND DEVELOPMENT ON HIGH TEMPERATURE ADDITIVES FOR LUBRICANTS AND HYDRAULIC FLUIDS. Kathleen Brown, Kenneth L. McHugh, Emery N. Wescott, J. O. Smith. September 1959.

An extensive literature system consisting of 11,861 abstracts (about 900 pages) and a set of IBM cards describing each abstract has been compiled on additives for lubricants and hydraulic fluids. The necessary explanatory material for the use of the system is described.

An experimental program to synthesize and evaluate extreme pressure agents, anti-wear materials and viscosity index improvers for esters, silanne and specially refined mineral oil base stocks for use in the 400 to 700°F temperature range has been started. The experimental program which was exploratory in nature, has shown the triaryl phosphine sulfides and the phosphorus pentasulfide treated polyisobutylenes to be potentially useful additive materials. The tri-aryl- and tripridylphosphine sulfides have given good results in the Shell 4-Ball machines as anti-wear and extreme pressure agents. The phosphorus pentasulfide treated high molecular weight (10,000) poly-isobutylenes have anti-wear properties. The phosphorus pentasulfide treated polyisobutylenes also show viscosity index improving properties in the mineral oil. The majority of these additives act as anti-oxidants in the oxidation corrosion test. The triarylphosphine sulfide appear to be corrosive to copper and are not so soluble in the base stocks as would be desired. The polyisobutylene-phosphorus pentasulfide reaction products are quite soluble in the silane and hydrocarbon, but only slightly soluble in the ester.

WADC TR 59-244.
PROCEEDINGS OF THE AIR FORCE-NAVY-INDUSTRY LUBRICANTS CONFERENCE. Robert J. Benzing. October 1959.

This report is a compilation of papers presented at the Air Force- Navy - Industry Lubricants Conference held 17-19 February 1959, at the Biltmore Hotel, Dayton, Ohio. The conference was attended by over 400 representatives of industry and the Department of Defense and other Governmental agencies. Thirty-four papers were presented providing a review of requirements for fluids and lubricants and of contract, industrial, and internal research and development in greases, dry friction-reducing films, engine oils, hydraulic fluids and missile component lubrication.

Massachusetts Institute of Technology. WADC TR 59-603.
FRICTION AND WEAR AT ELEVATED TEMPERATURES. Ernest Rabinowicz. AF 33(616)-5963. January 1960. PB 161719. Order from OTS $0.75.

A new high-temperature friction apparatus has been constructed which allows sliding experiments to be carried out at temperatures to 2000°F, in controlled atmospheres, and at speeds varying over a wide range. Tests have been run on this machine an on an older machine. These and other results are discussed theoretically in terms of the surface energy--hardness ratio and of the wear coefficient. From the experimental results, it appears that the main influence of temperature on the friction and wear results in through changes of hardness, of surface energy, and of tensile strength.

WADC TR 59-764.
LUBRICATING BEHAVIOR OF LIQUID METALS. Patrick H. McDonald. March 1960. PB 161888. Order from OTS $2.00.

This report describes the investigation of the lubricating behavior of liquid metals in two categories: hydrodynamic and boundary lubrication. The basic theory of the hydrodynamic study is that of the Reynolds equation. This theory has been extended, after the manner of Christopherson, to a new state of development in this report. An experimental device has been designed and constructed for confirming this theory as it relates to liquid

metals. The complex and elaborate aspects of this apparatus are described in detail in the report. The study of a variety of liquid metals with this apparatus is anticipated. A microscopic theory of the boundary lubrication region has been employed. This theory has been applied to a cylinder-flat combination, and the lubricating behavior has been seen to depend upon the state of contact stress for this configuration. An apparatus for these investigations is also described in the report.

Lehigh University. WADC TR 55-240, Part VI. BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP LUBRICATING GREASES. John J. Chessick, Albert C. Zettlemoyer, James P. Wightman, Lovella Raub. AF 33(616)-3999. April 1960.

A systemization of the most probable states for solids dispersed in organic media has been made. Specific attention has been paid to the development of more rigorous definition where ambiguity existed previously. Flocculation is discussed in terms of (primary) van der Waals type attractive forces and secondary and tertiary attractive forces. These latter two result from the influence of flocculating agents. Reduced deflocculation occurs due to steric hindrance, adsorbed lyosphere, entropic repulsion or double layer effects.

An investigation designed to determine the causes of structural instability of non-soap greases during use, and the application of the knowledge obtained to the production of superior greases was initiated. Various techniques such as ion incorporation or resin treatment of thickener solids were employed to increase the mechanical and temperature stability of greases containing various inorganic thickeners. Good success was achieved here, apparently. Grease with excellent stability properties can be prepared with surface modified thickeners. Surface modification was accomplished by deposition of phenol-formaldehyde type resins onto the surfaces of the solids in question followed by curing at elevated temperatures to a stable, coherent, though not necessarily, a mono-molecular film.

Miscellaneous work included the construction of a high temperature Shell Roll Tester and limited studies of the influence of additives on grease structure.

WADC TR 59-633, Part I. HIGH TEMPERATURE INSTRUMENT OIL. Albert A. Schwartz, S. Frank Murray, Harry R. Broadley, Jr., Robert S. Norman. April 1960.

An investigation was undertaken with the objective of developing a high temperature instrument lubricant for application in the temperature range of -65 to 400°F. This project is being directed not toward the synthesis of new high temperature lubricant materials (fluids, additives, etc.) but rather toward the utilization of existing materials for the formulation of a satisfactory lubricant.

A screening procedure was developed for the evaluation of the materials chosen for investigation on the basis of preliminary studies and surveys of high temperature fluids and additives. Sliding and rolling wear tests, coast-down time studies, and thin film static oxidation tests combined with coast-down time tests were used to select materials for further investigation. Studies to determine the effect of the properties of the test materials on their behavior in bearings were also carried out.

Full scale performance tests in instrument bearings are under way. Major emphasis in these tests is being placed on the silicone fluids because of their desirable physical properties and superior performance in the screening tests. Results to date indicate that lack of lubricating effectiveness may be the main obstacle in the path of the successful application of these fluids. Work is currently under way to find ways and means of remedying this deficiency. In this connection, lubricity improving additives and blends of silicones with other types of fluids are being investigated.

A simulated gyro gimbal assembly has been constructed. This assembly is designed to evaluate lubricated bearing surfaces undergoing small oscillatory motions at relatively slow speeds. This is similar to the motion experienced by the gimbal bearings in an operational gyro except that this system is designed to perform at 400°F.

High speed gyro motors have been completely redesigned to operate successfully at 400°F. This required development of a high temperature insulation system, high temperature adhesives and the use of stainless steel for bearings and shafts.

WADC TR 57-299, Part III. NUCLEAR RADIATION RESISTANT LUBRICANTS. William L. R. Rice, David A. Kirk. April 1960.

This report is intended to present a summary of the state-of-the-art of extreme temperature, nuclear radiation resistant fluid and lubricant development. It presents data on the effects of gamma radiation on a number of commercial fluids and lubricants, with a discussion of potential problem areas. Selection of improved materials of greatly increased thermal and nuclear radiation stability is discussed and the present status of preferred lubricants is outlined.

It is estimated that on a basis of information to date, the following conclusions can be made:

(1) That up to the dosages (2.61×10^{11} ergs/gm carbon gamma and 3×10^{16} nvt fast neutrons) investigated ambient temperature wear life of commercial solid film lubricants did not show significant changes on irradiation.

(2) Data on the silicone base greases show that the large changes which do occur in dropping point, bomb oxidation, bomb copper corrosion, and oil separation had little to no effect on the bearing performance tests. Preliminary in-source bearing tests on an arylurea silicone blend have performed satisfactorily for an average of 500 hours at 450°F and at a dose rate of 4.5×10^{7} ergs/gm carbon hour.

(3) The most promising candidate high temperature radiation resistant engine oils are the polyphenyl ethers. Laboratory tests indicate that they should be usable to bulk oil temperatures as high as 600°F in the presence of integrated nuclear dosages of 1×10^{11} ergs/gm carbon.

(4) In-source testing of a hydraulic loop showed that failure of Viton A dynamic seals could be expected at 3.0×10^{9} ergs/gm carbon gamma dosage and at 275°F.

(5) Aircraft instrument oils (MIL-L-6085) oxidation-corrosion stability was severely diminished at gamma dosages as low as 8.71×10^{8} ergs/gm carbon.

WADC TR 58-287, Part II. STORAGE STABILITY OF FLUIDS AND LUBRICANTS, PART II. Room Temperature Storage of Aircraft Greases. John B. Christian. April 1960. PB 161808. Order from OTS $0.50.

Eleven greases qualified under Military Specifications MIL-G-3278, MIL-L-3545, MIL-L-4343, MIL-G-7421, and MIL-L-7711, and supplied by various manufacturers were placed in storage at room temperature for two years. The greases were examined periodically to determine if storage had affected any of their properties.

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At the termination of two years of storage, it was discovered large changes in consistency had occurred in three out of four Specification MIL-L-7711 greases. The properties of these greases were no longer within specification limitations. All other greases maintained minimum requirements.

WADC TR 59-736, Part II.
LOAD SUPPORT AND ANTI-SCUFFING PROPERTIES OF LUBRICANTS PART II. WADC Universal Gear and Spline Lubricant Tester. Leslie R. Drane, Jr. May 1960. PB 171055. Order from OTS $0.75.

The design and operating characteristics of a gear and spline lubricant tester are described. Results of studies to determine the effects of lubricant flow rate, gear speed, and inert atmosphere on gear scuff load are presented.

Lubricant load carrying ability decreased sharply in the 5000 to 10,000 rpm range, but leveled off in the 15,000 to 20,000 rpm range. A slight increase in scuff load with increasing speed was noted in some cases.

The use of an inert atmosphere caused a decrease in scuff load rating with a mineral oil, a sebacate, and a silicone-ester blend. Two silicone fluids showed no significant change in scuff load when tested in an inert atmosphere.

Gulf Research and Development Company.
WADC TR 58-350, Part III.
DEVELOPMENT OF GREASES FOR HIGH SPEED BALL AND ROLLER BEARINGS. Paul R. McCarthy, Guy C. Blewett, John F. Hedenburg. AF 33(616)-5020. June 1960.

During this phase of the contract, approximately forty fluids and ninety experimental thickeners were evaluated as components for greases intended for operation at 20,000 rpm and 400°F.

The most promising of the fluids were certain of the silicones, m-bis (m-phenoxyphenoxy) benzene and a silphenylene. Since the m-bis (m-phenoxyphenoxy) benzene and silphenylene were in short supply, very limited work was done with these fluids.

Of the thickeners used primarily in conjunction with DC-550, certain lots of spiro (hydantoin-5, 1'-indan) and 1, 1'-methylenebis (5, 5-dimethyl-hydantoin) produced experimental greases which operated satisfactorily for a period of 500 hours at 400°F and 20,000 rpm. Greases prepared with other lots of the same materials failed in much shorter periods. Arylureas and combinations of arylureas and Benzidine Yellow as thickeners may provide greases showing even better performance under the above conditions. More extensive evaluation is needed to substantiate this.

Statistical analyses to determine (a) correlation between screening tests and performance of greases in antifriction bearings and (b) precision limits on performance life tests at 20,000 rpm and 400°F were made. Results on the analyses indicated no correlation for (a) and very poor precision for (b).

Construction of two 45,000 rpm extreme high speed testers was completed and one of these testers put into operation. The second tester will be in operation by February 10, 1960. Operation of the tester and accessory control equipment is very good. Sixteen trial runs on two greases showed very short life (0.25 to 10 hours) for the greases and bearings at speeds above 30,000 rpm.

Pennsylvania State University. WADC TR 55-30, Part VIII.
FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS. E. Erwin Klaus, Merrell R. Fenske, Elmer J. Tewksbury. AF 33(616)-5460. June 1960. ASTIA Document No. AD 242699.

This report describes work carried out on a continuing program to characterize the capabilities of various base stocks and fluid formulations for application as hydraulic fluids and/or jet engine lubricants under a wide variety of conditions. An antiwear or chemical polishing mechanism similar to that suggested for tricresyl phosphate is shown for silicon-containing fluids with ferrous bearing surfaces. Variables designed to improve yield, efficiency, and fluid properties are discussed for low temperature solvent dewaxing. In many cases, the viscosity-temperature characteristics of the oil and wax fractions are both better than those of the original oil prior to separation. Formulation, distribution, and hardware testing of several mineral oil fluids for use over the range of -65° to 700°F are noted. Results obtained in a 550°F vickers pump test program are discussed. The basic trends in thermal degradation with mineral oils have been studied in a pressure cylinder. Quantitative evaluation of the gaseous and liquid reaction products suggest different mechanisms for the gas and liquid phase reactions. The effectiveness of inhibitor combinations as antioxidants versus a single inhibitor is illustrated at temperatures of 347°, 400°, and 500°F. The use of a small volume test to evaluate materials available in limited quantities is discussed. A series of samples which have been in storage for periods of 2 to 17 years are being revaluated for changes in properties due to storage. An insight into the mechanism of oxidation on or in the vicinity of hot metal surfaces is provided by quantitative oxidation studies in the controlled atmosphere panel coker. A series of successive tests in the panel cokers has been used to explore further the mechanism of coking and the variables governing the deposit. Deposition type tests in a single-pass lube rig are also discussed.

Midwest Research Institute. WADD TR 60-530.
CERAMIC BONDED SOLID-FILM LUBRICANTS. M. T. Lavik. AF 33(616)-6115. September 1960. ASTIA Document No. AD 249067.

Thirty-three compounds were screened for their oxidation resistance in air. Sixteen compounds indicate less than 50 percent conversion to oxides after 4 hrs. at 1000°F. The same compounds were screened for frictional properties from 80° to 1000°F at light loads. Seven of them indicated a friction coefficient below 0.50 over a large portion of the temperature range.

Four high temperature bonding agents were selected for bonding films of the most promising solid lubricants. Outstanding results were obtained at 1000°F with PbS films bonded with B_2O_3. Wear-lives as high as 50,000 revolutions were recorded at 10000°F, 100-lb. load and 370 rpm, with friction coefficients from 0.07 to 0.15 and negligible wear on the rub shoes. The wear-life of the PbS/B_2O_3 films falls off very rapidly with decreasing temperature. Films combining MoS_2 or graphite with PbS/B_2O_3 films have not shown good results at 1000°F.

Standard Oil Company. WADD TR 60-557, Part I.
DEVELOPMENT OF GREASE LUBRICANTS FOR HIGH TEMPERATURE BALL AND ROLLER BEARINGS OF ELECTRICAL EQUIPMENT. A. C. Borgo, K. R. Bunting, A. M. Dobry, J. C. Goossens, J. H. Klauwers, R. S. Barnes. AF 33(616)-6584. October 1960.

The objective of this contract is the development of grease systems for use in ball and roller bearings of

electrical equipment over an ultimate temperature range of -65°F to 900°F. The current program is aimed at the development of a -40°F to 700°F grease. The high temperature performance is the developing and screening fluids, thickeners and greases at 600°F. 600°F was chosen because long time performance at this temperature is required by the contract and it was close to the limit of significantly long performance of available grease and test equipment at the beginning of the contract.

Equipment was built or obtained for running loaded bearing tests, dropping points, evaporations, and roll stabilities at temperatures up to 900°F. Several types of bearings, said to be better for high temperature use than the MRC 204 S-17 bearings used in this period, were received at the end of this reporting period, but no tests have yet been run on them.

Fluids and thickeners, believed to be potentially useful in -40°F to 700°F greases, were synthesized or obtained from outside sources and screened in bench tests for adequately high thermal stability and low volatility. Thickeners were also screened for thickening ability over the temperature range. Materials that appeared promising after screening were made into greases and bearing tested. Fluids were thickened with the best high-temperature thickener available, and thickeners were dispersed in the best high-temperature fluid to obtain the greases for these tests.

Most bearing tests were run at 600°F, under modified CRC L-35 conditions, on Pope-Texas type spindles. Under these conditions, the best of the currently available greases, methylphenyl silicones, thickened with one of several high-melting organic thickeners ran for about 55 hours. Greases made with several experimental silicone fluids gave results that ranged from slightly worse to significantly better than those obtained with the best available greases. The best results were obtained with DC-QF-6-7024 Silicone Fluid which ran from 100 to 400 hours in various tests. Failures were due to the instability of the silicone fluids. The best phenoxyphenyl ether and chain-type polyphenyl greases ran as long as the best of the currently available greases. Failures were not due to fluid instability, but fluid volatility. Preparation of lower volatility fluids of these and related structures are under way. Surface treated silica, carbon black and glass fiber thickeners gave better results than the best of the currently used high temperature thickeners. In addition, some promising initial results were obtained with high melting polymers, complex sulfonyls and sulfonamides, and some organic pigments. None of these thickener systems has been completely perfected and even better results are to be expected as they are further developed.

Running similar bearing tests on MRC spindles gave results equivalent to those obtained on the Pope-Texas spindles, when similar load conditions were used. Under higher load conditions, shorter test runs were used. The DC-QF-6-7024 Fluid grease ran only 8 hours when the axial load was increased from five to fifty pounds. Load carrying additives improved bearing performance at higher loads. One additive in the QF-6-7024 grease lowered the five pound axial load running time to about 60 hours, but raised the fifty pound running time to over 30 hours. Another additive when added in this grease, did not affect the five pound load life, and ran for over 40 hours at fifty pounds load.

Increasing the temperature to 650°F decreased the running time.

Increasing the amount of grease filled into the bearing increased the running time.

No tests were run with relubrication, but the development and use of such a technique coupled with the use of better bearings is expected to increase bearing running times.

Midwest Research Institute. WADD TR 60-529. MECHANISM OF LUBRICATION OF GRAPHITE SINGLE CRYSTALS. Paul J. Bryant. AF 33(616)-6277. October 1960.

A research program has been initiated to investigate the friction mechanism of graphite. The effects of atmosphere, crystal orientation, etc., on the lubricative process are being observed.

A search for satisfactory graphite samples has been undertaken with graphite whiskers being considered first. The whisker structure was first found to be imperfect when it was physically removed from the growth matrix. A technique of selective oxidation was developed to extract the whiskers from the matrix without physical damage. Whiskers thus extracted, while still not ideal, are useful in some phases of the study. In particular, they contain a uniform inner core which is useful for surface friction, atmosphere, and wear damage studies.

Single crystals of graphite were produced by the thermal decomposition of aluminum carbide single crystals. These samples have the regular graphite crystalline orientations throughout, and are representative of the fundamental graphite system.

Aluminum-oxide whiskers were grown as a by-product of the technique for producing graphite single crystals. Their structure and the mechanism by which they were produced are of interest because they indicate the temperature conditions which existed during the production of graphite crystals.

X-ray diffraction analysis of the single crystals showed uniform crystallographic orientations and interatomic spacings in close agreement with those reported for natural graphite. The graphite whiskers show normal graphite spacing, but their crystal orientations were not uniform. The aluminum-oxide whiskers were found to consist entirely of the alpha phase and to contain no measurable impurities.

Structural features of the graphite whiskers and single crystals as determined by optical microscopy and induced damage experiments indicate the type of experiments for which the whiskers and single crystals are suitable.

WADD TR 60-536.
HIGH TEMPERATURE LUBRICANTS FOR ADVANCED GAS TURBINES. Harold W. Adams. November 1960. ASTIA Document No. AD 258596.

Selected polyesters derived from trimethylol propane proved to be satisfactory base stock fluids from -65°F to bulk oil temperatures of 400°F to 450°F, for advanced gas turbine engines. A combination "inhibitor package" consisting of 5-10-10 diphenylphenazasilane and phenyl alpha naphthy amine, was found to be most effective in these trimethylol propane fluids. The finished fluid formulations (resulting from the above base fluids and additive combinations) provided the necessary physical and chemical characteristics required for advanced gas turbine lubricants.

Shell Development Company. WADD TR 60-424. HIGH TEMPERATURE LUBRICATION IN THE PRESENCE OF NUCLEAR RADIATION. V. N. Borsoff, S. J. Beaubien, W. W. Kerlin. AF 33(616)-6658. November 1960. ASTIA Document No. AD 253208.

The work presented is an investigation of lubricating capabilities of unsubstituted polyphenyl ethers and a comparison of these ethers with conventional lubricants. The work consists of studying the performance of the lubricants in bearings and gears under severe thermal, oxidative and ionizing radiation stresses. Based on the evidence obtained, it appeared that all lubricants suffer an

appreciable decrease in load carrying capacity at elevated temperatures, but preserve their lubricating properties under the most severe environments, providing the flow of oil to the load bearing elements is not impeded in any manner. Since the flow of oil is highly affected by degradation due to heat, oxidation and ionizing radiation, the stabilities of unsubstituted polyphenyl ethers and MIL-L-9236A oil were studied. The results showed a great superiority of the polyphenyl ethers over the conventional oils. Regarding the effect of ionizing radiation, it was noted that at levels below 1×10^9 ergs/g C its effect is negligibly small and in this radiation range the main causes of lubricant degradation are heat and oxidation.

General Electric Company.
WADC TR 59-633, Part II.
HIGH TEMPERATURE INSTRUMENT OIL. Albert A. Schwartz, Harry R. Broadley, Jr., Robert S. Norman. AF 33(616)-5894. December 1960. ASTIA Document No. AD 258180.

Studies were made on lubricity improvers and oxidation inhibitors for silicone fluids, which had been found in our earlier work to be the most promising base stocks. Preliminary screening of the various fluid additive combinations were carried out using procedures developed in Part I of the program. Full scale performance tests in instrument bearings were run in air and in argon atmosphere. Significant improvement in performance in both the preliminary screening and the bearing tests was achieved with several of the lubricity additives. A vendor-developed dry lubricated ball bearing looked very promising in the bearing life tests.

Evaluation of the base stock, the most promising fluid formulation, and the vendor-developed dry lubricated ball bearing was carried out in a simulated gyro gimbal assembly and in high speed gyro motors designed to operate at 400°F. Significant improvement over the silicone base oil was obtained with the oil compounded with a lubricity improver, especially in the slow speed gimbal tests. Lubrication of the high speed gyro motors with these lubricants was not as satisfactory as the slower speed application.

The dry lubricated bearings developed by the outside vendor appear promising for moderate to high speed applications, such as the tachometer generator, where torque requirements are not critical. These bearings did not prove to be satisfactory in the gyro gimbal test since bearing torque increased rapidly during the first 200 hours of operation.

Cleveland Graphite Bronze. WADD TR 60-757. SILICONE LUBRICANT-CARBON SEAL INTERACTION AT ELEVATED TEMPERATURES. Thomas H. Koenig, E. James Vargo. AF 33(616)-6714. December 1960. ASTIA Document No. AD 258535.

The advent of higher power jet engines necessitated a suitable high temperature lubricant. Silicone oils became attractive candidates for this field primarily because of their thermal stability at high temperatures. However, one problem presented itself in the form of deposits at carbon seal areas. It was originally theorized that these deposits were formed by a basic incompatibility between silicone oil and the carbon used for mechanical seals.

Static immersion tests using 18 grades of carbon in combination with three silicone oils, one ester, and one silicone-ester blend at bath temperatures of 400°F, 500°F gave no indication that carbon has an effect on viscosity increase or high temperature breakdown of the oils tested. In tests where radical changes occurred in samples containing carbon, there were also radical changes in control samples not containing carbon. This point was also confirmed in dynamic tests.

In a test setup simulating sealing conditions, silicone sludges were formed at indicated interface temperatures of 280°F. This temperature is considerably below the oxidation and thermal decomposition temperatures of the lubricants. Localized hot spots were found to be the source of the high temperature. In one case, temperatures 300°F higher than the average were recorded.

Bench rig tests of shaft and face seals produced silicone sludge under conditions far less severe than those encountered in the field. Tests run with a nitrogen blanket show that excluding oxygen from the seal interface inhibits or stops the formation of silicone sludge. This method of seal protection will be satisfactory until the seal interface temperature reaches the thermal decomposition limit of the silicone oil, approximately 620°F.

WADD TR 60-770.
SYNTHESIS AND INVESTIGATION OF HIGH TEMPERATURE GREASE THICKENERS. John Q. Griffith, III, John B. Christian. December 1960. ASTIA Document No. AD 258380.

High melting imide solids synthesized from anthraquinone, pyromellitic acid, pyromellitic dianhydride, phthalic anhydride and various amines were investigated as potential high temperature grease thickeners. Several commercially available high melting solids such as asbestos, resins and amine anthroquinone thickening compounds were included in this investigation.

Greases were formulated from these materials by solvent, heating, and milling techniques. Physical characteristics of the resulting greases were also determined.

An imide thickener combination comprised of two separate components, the aluminum complex of pyromellitic benzoic acid and phthalimido benzoic acid proved to be the most promising material investigated. It proved to have good thermal stability, excellent gel characteristics and a melting point above 1200°F.

The commerically obtainable high melting point solids were generally ineffective as grease thickeners. Better methods of dispersion and of keeping article size uniform would greatly facilitate this work.

Ferrous Metals and Rare Metals

TR 4773.
VARIATION IN PROPERTIES OF COLD-ROLLED CORROSION-RESISTANT 18-8 STEEL SHEET AND EFFECTS OF LOW TEMPERATURE HEAT TREATMENT. B. Chasman, G. M. Martell, T. T. Oberg. May 1942.

TR 4799, Part I.
INVESTIGATION OF ALTERNATE STEELS. A. C. Willis, 2/Lt., Morse Hill. July 1942.

TR 4802.
HARDENABILITY OF STEEL. Morse Hill. July 1942.

TR 4799, Part 2.
INVESTIGATION OF ALTERNATE STEELS. Morse Hill. December 1942.

Cornell Aeronautical Laboratory, Inc.
TR 52-77.
DEVELOPMENT OF LOW ALLOY TI-B STEELS FOR HIGH TEMPERATURE SERVICE APPLICATIONS.
G. I. Guarnieri, L. W. Smith. W 33-038-ac-21094. April 1952. PB 123915. Order from LC, Mi $4.50, Ph $13.80.

Conservation of strategic metals may be accomplished in the production of jet aircraft engine parts provided suitable lean alloy substitutes are made available, capable of operating at elevated temperatures. In this respect, the Ti-B type of ferritic steels have been investigated and their creep and rupture properties evaluated and improved for service temperatures in the neighborhood of 1200°F. It was demonstrated, through testing the properties of a 600 pound heat of 3 Cr-1 Mo-Ti-B sheet steel with a 2.2 Ti/C ratio, that such a composition not only could be steel mill processed satisfactorily on a semi-commercial basis, but also that 1200°F rupture and creep strength properties, equivalent to the Cr-Ni stainless steels, could be obtained for several hundred hours of life.

A detailed investigation of this steel provided design type creep data for several conditions of heat treatment and hot rolling procedures. Other tests indicated that high hot strength properties could be retained in light gage sheet material provided surface decarburization was minimized during processing. Ceramic coated creep and rupture test specimens of this alloy displayed a life advantage over uncoated specimens at temperatures above 1200°F because of protection against oxidation. Heli-arc welded joints of the Cr-1 Mo-Ti-B sheet steel had inferior hot rupture strength with respect to the parent metal when no filler rod was used. A reasonable approach to parent metal high temperature strengths was obtained with the use of a 347 stainless steel filler rod with the weld bead left on.

As the result of studies made on a variety of compositions of 30 pound laboratory heats of Ti-B steels with varying C, Ti, B, Mo, Cr, W, and V, it was possible to determine the effect of these alloys on the high temperature strengths of the ferritic steels. With increasing quantities of titanium and carbon, marked gain in hot strength was obtained in both the plain C-Ti-B and 3 Cr-1 Mo-Ti-B steels, provided the Ti/C ratio was maintained in the neighborhood of two to five. Boron variations from 0.010 to 0.10 percent produced no significant effect on the high temperature strength properties at 1200°F. Molybdenum was most effective in improving the hot strength properties of the Ti-B steel while chromium served more in providing resistance to scaling and oxidation. Tungsten and vanadium, added to the 3 Cr-1 Mo-Ti-B steels to the maximum extent of about 0.15 percent individually or in combination, caused only minor gain in creep and rupture strength properties.

The unusual high temperature strength of the Ti-B steels is the result of their ability to form a hardened low temperature transformation product of acicular ferrite exceptionally resistant to tempering. This is accomplished as a result of the following:

(a) Solid solution hardening and diffusion interference effects of both boron and titanium.
(b) The retention of titanium and carbon in the supersaturated ferrite.
(c) The dispersion hardening effect of the precipitated titanium carbide.

TR 52-73.
MECHANICAL PROPERTIES OF A NEW HIGH-STRENGTH HIGH-TOUGHNESS GENERAL-PURPOSE ALLOY STEEL. William E. Dirkes, 1/Lt., Robert E. Bowman, Major, Edward L. Horne. August 1952. PB 111617. Order from OTS $0.75.

Mechanical properties of a chrome-nickel-molybdenum-vanadium general purpose medium carbon alloy steel (modified SAE 4330 plus vanadium), developed and fabricated by the Republic Steel Corporation, were determined by the Materials Laboratory, Research Division, Wright Air Development Center, to evaluate the suitability of the steel for aircraft applications where the combination of high strength and toughness are required. The proper- of the Republic alloy steel were compared with the properties of an alloy steel, designated as "Hy-Tuf", developed by the Crucible Steel Company for the same purpose and with the properties of the well established SAE 4340 general purpose alloy steel. When heat treated to the same nominal hardness and strength levels, the Republic steel had approximately the same percentages of elongation and reduction of area as the other steels. The Republic alloy steel showed superior Izod impact properties to those of Hy-Tuf and SAE 4340 alloy steels when each was heat treated to a tensile strength approximating 170,000 psi (Rockwell C39) both at room and low temperatures, but its impace properties were not as high as those for Hy-Tuf when heat treated to strength levels around 220,000 psi either at room or low temperatures. Both the Republic alloy steel and Hy-Tuf alloy steel had impace properties superior to 170,000 psi to 240,000 psi (Rockwell C39 to Rockwell C50), covered in this investigation, at room temperature and at low temperatures (-67°F or below). The end-quench hardenability curve for the Republic alloy steel fell along the minimum curve for SAE 4340 alloy up to a distance of 3/4 inch from the quenched end of the specimen, beyond which distance it approached the median of the hardenability range for SAE 4340. The Republic steel can be successfully welded by the same procedures used for welding SAE 4340 alloy steel.

TR 52-325, Part I.
ELEVATED TEMPERATURE FATIGUE PROPERTIES OF SAE 4340 STEEL. W. J. Trapp. December 1952. ASTIA Document No. AD 8716. PB 123193. Order from LC Mi $3.60, Ph $9.30.

This report presents the test procedures and results of a fatigue investigation at room and elevated temperatures on SAE 4340 steel, oil quenched and tempered to 160,000 psi in the unnotched and notched condition. The notch used in the investigation is a 60° V-notch with 0.010" radius and 0.025" depth.

The results, which are presented in form of S-N diagrams, normal and nondimensional modified Goodman and stress-range diagrams, reveal the effect of temperature and stress-ratio on the unnotched and notched fatigue properties.

The fatigue tests were supplemented by stress-rupture and creep-rupture tests and by dynamic creep-measurements. The investigation was conducted at room temperature, 600°, 800° and 1000°F.

In general, the fatigue strength was found to decrease with increasing temperature at all stress levels and all stress-ratios except for the life times between 10^5 and 15×10^6 cycles in the notched condition, where at 600° the value is lower than at 800° and even lower than at 1000°, dependent upon stress ratio. This can probably be related to an increase in brittleness in the 600° region, which is also confirmed by the fact, that the notch sensivity at 600° was found to be higher than at any of the other temperatures investigated.

The notch-sensitivity factor, based on maximum stress, is dependent upon temperature, stress-level and stress-ratio. It generally decreases with increasing stress level, increasing temperature and decreasing stress-ratio. The peak of notch-sensitivity is produced at completely reversed load for all temperatures.

The tests indicated that creep is dependent upon mean stress rather than upon maximum stress. Two distinct types of creep-time diagrams were obtained, determined by stress-ratio, mean stress and temperature. A fracture study revealed certain relations between the type of creep diagram and type of fracture, inter and transgranular.

TR 53-256.
FATIGUE OF ALLOY STEELS AT HIGH-STRESS LEVELS. Ture T. Oberg and Edward J. Ward. October 1953. ASTIA Document No. AD 23896. PB 128532. Order from LC Mi $2.70, Ph $4.80.

Several alloy steels used in aircraft have been investigated with the purpose of determining the fatigue strengths at stresses higher than are found in the stress-cycle fatigue diagrams as usually determined. The stress-cycle diagrams have been extended down to about 1000 cycles and the effects of internal heating at high stresses shown.

Syracuse University. TR 53-254, Part 1. SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT-TREATED TO HIGH STRENGTH LEVELS. George Sachs and Walter Beck. AF 33(616)-392. June 1954. PB 121700. Order from OTS $2.50.

In Part 1 data on hydrogen embrittlement of high-strength steels are presented and analyzed. Many failures in aircraft reported for steel parts having a strength above 200,000 psi and in addition, many failures of steel bolts heat treated to a strength considerably below 200,000 psi were found to be associated with changes in the basic mechanical characteristics of the steel caused by cadmium plating or chromium plating. Heat treating or baking applied to relieve this hydrogen embrittlement were frequently only partly effective.

Hydrogen in steel is highly mobile and this explains the unusual mechanical features of hydrogen em brittlement and the conflicting results of the many tests which were applied to disclose and measure hydrogen embrittlement. The normal strength of a high-strength steel is found to be rarely affected while its ductility may be greatly reduced. Consequently the strength of test specimens and parts which depends upon ductility may also be much reduced by hydrogen embrittlement. Furthermore, these effects are pronounced at low rates of straining or sustained loading but probably of no significance on impact loading.

The magnitude of hydrogen embrittlement also depends greatly upon numerous mechanical, chemical and electrochemical factors which are encountered in the making, shaping, heat treating, and finishing of aircraft parts.

Syracuse University. WADC TR 53-254 (Part 3). SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH STRENGTH LEVELS. George Sachs. AF 33(616)-392. July 1954. ASTIA Document No. AD 43818. PB 121667. Order from OTS $2.00.

Part III of the resport presents descriptions of actual failures encountered in high-strength steel aircraft parts and of the experimentation performed for the purpose of analysing the conditions of failure and determining the sources of it. Practically all of such information relating to aircraft parts has been supplied by aircraft and aircraft parts industries.

The attempt to systematically arrange this information leads to the conclusion that there exists a number of different sources of the failures in high-strength-steel aircraft parts. Furthermore, as a rule a failure resulted from a combination of several factors which deviated from those encountered in regular manufacture rather than from a single factor. From the analysis of the failure cases it may be deduced that a change in one particular factor would have been sufficient and was sufficient in a number of instances to prevent any further failures of the same part.

Syracuse University. WADC TR 53-254 (Part 2). SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH STRENGTH LEVELS. George Sachs. AF 33(616)-392. August 1954. PB 123090. Order from LC Mi $6.00, Ph $18.30.

In Part II the information available on fatigue characteristics of high-strength steels is assembled. The data obtained from various sources frequently show great differences even where the testing conditions were presumably identical. Still larger variations resulted from slight differences in testing or in steel conditions the nature of which was frequently unknown.

Contrary to common concepts, high-strength steels may exhibit endurance-strength values considerably superior to those of steels heat treated to a strength of 200,000 psi or lower. This also applies to such values in the presence of stress concentrations. It appears, in general, that any specific steel exhibits fatigue-strength characteristics which may be particularly unfavorable at certain tempering temperatures. High-hardenability high-strength steels usually possess endurance limits considerably above those of other steels heat-treated to high strength levels.

Syracuse University. WADC TR 53-254 (Part 4). SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH-STRENGTH LEVELS. George Sachs. AF 33(616)-392. August 1954. ASTIA Document No. AD 45578. PB 121504. Order from OTS $3.75.

Part 4 presents a general discussion of the factors which determine the selection of high-strength steels and assembles the data available for their static-strength and design characteristics. In addition, the information available on the effects of the numerous variables encountered in making, shaping and heat treating low-alloy steels and their significance for the strength properties of aircraft parts is discussed.

The regular strength characteristics of the steels, and especially their tensile strength, yield strength, elongation and reduction of area, reveal no indication of an embrittlement of steels heat treated to strength values in excess of 200 ksi, in general, and of those tempered at temperatures between 500 and 750°F, in particular. However, there is some indication that at very low testing temperatures steels heat treated within this tempering range also may exhibit a comparatively low ductility.

The response of static strength properties to other factors which adversely affect their performance, such as section size of slack quenching, is also limited and the practical significance of such effects is not clarified.

Syracuse University. WADC TR 53-254 (Part 5). SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH-STRENGTH LEVELS. George Sachs, E. P. Klier. AF 33(616)-392. September 1954. ASTIA Document No. AD 50288. PB 121505. Order from OTS $3.75

Part V summarises the results of impact tests and notch-tension tests on high-strength steels.

The impact strength of constructional low-alloy steels generally exhibits a minimum at tempering temperatures between 500 and 750°F for short tempering times. Therefore, steels tempered either at about 400°F or over

800°F are preferably used. Also, high-strength steels with optimum impact strength at a given strength level are obtained by holding the carbon content as low as possible. The effects of the numerous variables encountered in processing and heat treating steels on the impact strength are apparently not universal and not well clarified and frequently are obscured by secondary effects, such as the softening associated with slack quenching.

In contrast, the notch-strength in tension of high-strength steels appears to respond sensitively to a number of variables of different types such as magnitude of stress concentration, eccentricity of loading, section size of quenched and tested specimen, section size of product and method of quenching. The data available at present in these respects, are rather limited but appear to indicate universal trends.

In certain instances a parallelity between impace strength and notch strength has been noted.

Syracuse University. WADC TR 53-254 (Part 6). SURVEY OF LOW-ALLOY AIRCRAFT STEELS HEAT TREATED TO HIGH STRENGTH LEVELS. George Sachs and E. P. Klier. AF 33(616)-392. September 1954. ASTIA Document No. AD 52663. PB 121506. Order from OTS $0.50.

The appendix presents a number of suggestions for future research work on low-alloy aircraft steels heat treated to high strength levels.

Southern Research Institute. WADC TR 53-439. METALLURGICAL TESTING OF BORON STEELS. C. L. Dotson, J. R. Kattus, and F. R. O'Brien. AF 33(616)-468. October 1954. ASTIA Document No. AD 56078. PB 129526. Order from LC Mi $11.00, Ph $37.80.

Knowledge of the mechanical properties of many medium-carbon, boron-treated steels is insufficient for proper evaluation of the suitability of these steels for aircraft uses.

The hardenability, tensile, impact, notched tensile, and fatigue properties of boron steels 81B40, 98B40, and 81B30 have been determined and are reported. The hardenabilities of carburizing steels 94B17 and 43BV10 have been investigated for different carburized case depths and carbon contents. Impace properties of pseudo-carburized specimens have been determined. Detailed test results are reported for all investigations.

Results of this investigation can be used to determine the suitability of these boron treated steels for aircraft applications by comparision to the properties of alloy steels now used in the aircraft industry.

Massachusetts Institute of Technology. WADC TN 55-87. SOME EFFECTS OF SILICON ON THE MECHANICAL PROPERTIES OF HIGH STRENGTH STEELS. Chester H. Shih, B. L. Averbach, Morris Cohen. AF 33(616)-2012. November 1955.

The effects of increasing the silicon content in 4340 and 4325 steels to 1.5 percent have been observed as a function of tempering temperature. The softening on tempering is retarded by silicon, and it appears possible to obtain somewhat higher strengths in the silicon steels without a corresponding loss in ductility or in Charpy impact strength. At high strength levels the elastic limit is approximately one-half of the engineering yield strength; s licon additions have no influence on the maximum elastic limit. Although the introduction of silicon has several beneficial effects at high strength levels, undesirable impact properties may be introduced by tempering at high temperatures (temper embrittlement). Retained austenite data are presented as a function of cooling rate from the hardening temperature, and it is shown that substantial amounts of retained austenite may be developed on retarted cooling. There is also some indication that the fatigue endurance limits and the elastic limits may be related.

Battelle Memorial Institute. WADC TR 56-598. AN INVESTIGATION OF ABSORBED HYDROGEN IN ULTRA-HIGH-STRENGTH STEEL. Charles R. Simcoe, Arthur R. Elsea, George K. Manning. AF 33(616)-3202. November 1956.

A study was made of the relationship between current density and the permeation of hydrogen through a thin, steel membrane which was the cathode in an electrolytic cell. The established relationship was used to study the effect of the variation in hydrogen-absorption rate on the time for rupture to occur under sustained stress. It was found that the relationship between the rupture time and the rate at which hydrogen was absorbed by the steel could be expressed by the equation $T_f = KP^{-2/3}$ where T_f was the rupture time, K was constant for each applied stress, and P was the permeation rate.

A study was made of the incubation times for crack nucleation and of crack growth rates under various experimental conditions. Incubation times increased with decreasing applied stress. Crack growth rates also increased with decreasing applied stress, whereas, it was expected that they would decrease.

The effect of other factors, such as austenitizing temperature, prestrain, precharging, composition of the steel, and torsion loading, upon the rupture time also was studied.

National Bureau of Standards. WADC TR 56-396. TEMPER BRITTLENESS OF BORON-TREATED STEEL. Samuel J. Rosenberg. AF 33(616)-55-8. December 1956. ASTIA Document No. AD 110638. PB 121889.

Two series of steels, melted to the base composition of 8140, were studied to ascertain whether titanium and zirconium (present in many commercial boron addition agents) had any adverse effect upon the impact properties of the base steels, particularly with reference to temper brittleness. All steels were given the same hardening treatment (1/2 hour at 1600°F, oil quench) and were tempered 1 hour at 1200°F. The rate of cooling from the tempering temperature was varied so as to develop temper brittleness and isothermal embrittling treatments also were employed.

Southwest Research Institute. WADC TR 56-205. FATIGUE INVESTIGATION ON HIGH STRENGTH STEEL. J. K. Childs, M. M. Lemcoe. AF 33(616)-2513. July 1957. ASTIA Document No. AD 110474. PB 131371. Order from OTS $1.25.

A single heat of aircraft quality SAE 4340 steel, heat-treated to 190,000, 260,000 and 300,000 psi ultimate tensile strength, was tested by the increasing load method (similar to Prot's method) in axial loading to determine the variation in fatigue properties under each of the three mean stress conditions, sero, 60,000 and 90,000 psi. Conventional S-N curves were determined by axial load tests at 190,000 psi ultimate tensile strength, under sero mean stress and 90,000 psi mean stress, to provide factors for adjusting the Prot-type failure stresses to standard mean endurance limit values. In addition, Prot rotating beam tests were performed at 190,000 psi ultimate tensile strength.

The endurance limit under completely reversed axial stress increased with an increase in ultimate strength from 190,000 psi to 260,000 psi, but appeared to decrease when the same material was heat-treated to 300,000 psi. Under high mean tensile stress the 300,000 psi material may have a fatigue strength superior to that of the 260,000 psi material.

Massachusetts Institute of Technology. WADC TR 57-501.
MECHANICAL PROPERTIES OF SOME SILICON-MODIFIED HIGH STRENGTH STEELS. Carl J. Altstetter, Morris Cohen, B. L. Averbach. AF 33(616)-2012. December 1957. ASTIA Document No. AD 142265.

The mechanical properties of 43XX steels, containing 0.15%, 0.25% and 0.37% carbon, with an without 3% silicon, were determined as a function of tempering temperature. 4337 steels with 1.5% silicon and with variations in nickel, molybdenum and boron were also tested. The chief advantage of the silicon addition appears at high strength levels. Silicon retards the third stage of tempering, and an ultimate strength of 300,000 psi is developed in 4337 (3.0 Si) even after tempering as high as 600°F. The ductility and impact properties are not adversely affected as this strength level by the silicon. Silicon was also found to increase the as-quenched hardness, especially in the two lower carbon grades.

WADC TR 58-108, Pt. I.
THE EFFECT OF CADMIUM PLATING ON SAE 4340 STEEL IN THE PRESENCE OF STRESS CONCENTRATIONS AT ELEVATED TEMPERATURES. E. M. Kennedy, Jr., Major. March 1958. ASTIA Document No. 151075. PB 131814.

An investigation was carried out to determine the effect of cadmium plating on the tensile and stress rupture properties of high strength steel at elevated temperatures. Tests were conducted on SAE 4340 steel, and properties of this material were investigated in the annealed and heat treated conditions. The annealed material was investigated at 600°F and 611°F, effects on properties of the heat treated material were investigated at 500°F, 600°F and 611°F. The properties of cadmium plated steel in both conditions, annealed and heat treated, were compared with the properties of unplated steel subjected to the same test conditions.

Ohio State University Research Foundation. WADC TR 56-555.
DEVELOPMENT OF NEW MECHANISMS AND TECHNIQUES FOR OBTAINING STEELS IN THE ULTRA-HIGH STRENGTH RANGE. Damian Gullotti, Erwin Eichen, Joseph W. Spretnak. AF 33(616)-2879. May 1958. ASTIA Document No. AD 130755. PB 131238.

Two possible methods of obtaining steels in the high strength range were investigated, namely, the superimposition of plastic deformation on the transformation of austenite to martensite, and the production of high nitrogen-low carbon (0.25% maximum) martensite. In the deformation of austenites prior to transformation, increases in tensile strength up to 10% and in elastic limit up to 30% were obtained. In steels of base composition C-0.20, Si-1.60, Mn 0.75, Cr-1.40, increasing the nitrogen content from 0.004 to 0.009% increased the 0.2% offset yield strength about 6% and the tensile strength about 3.5%. The ductility was essentially unaffected by this increase in nitrogen content.

Research Chemicals Inc. WADC TR 57-666 Part I. SELECTION AND EVALUATION OF RARE OR UNUSUAL METALS FOR APPLICATION TO ADVANCED WEAPONS SYSTEMS PART I. A LITERATURE SURVEY. Bernard Love. AF 33(616)-5293. June 1958. ASTIA Document No. AD 155685. PB 151311. Order from OTS $3.00.

A survey has been made of the unclassified literature relating to the rare earth elements, hafnium, rhodium, and ruthenium. The best available data has been compiled for the abundance and distribution of elements, the methods of recovery from ores and purification, the reduction to metal, and the physical, chemical, and mechanical properties of the elements and their important compounds.

Massachusetts Institute of Technology. WADC TR 57-720 Part I.
THE EFFECT OF PRESTRAIN AND RETEMPERING ON ULTRA-HIGH STRENGTH STEEL. Edward T. Stephenson, Benjamin L. Averbach, Morris Cohen. AF 33(616)-2012. November 1958. ASTIA Document No. AD 206076.

The mechanical properties of pretempered, prestrained, and retempered ultra-high strength steel are reported here. Pretempering temperatures were varied from 400 to 1200°F, prestrains from 0 to 7% elongation, and retempering temperatures from 70 to 1200°F.

It is found that prestrain and retempering may cause a significant increase in the yield stress and tensile strength, but with an accompanying loss of ductility and toughness. On increasing the retempering temperature, a maximum occurs in the strength along with a minimum in the ductility and toughness. The temperature of the maximum shifts from 300 to 700°F with increasing pretempering temperature, and the minimum behaves similarly.

Any tempering-prestrain combination which increases the strength appreciably also decreases the elongation-to-maximum-load to practically zero. This behavior severely limits the possible application of prestrained ultra-high strength steel.

Prestrain and retempering (below the pretempering temperature) have no observable effect on the fracture stress, the hardness, or the electron microstructure.

Several phenomena are considered in an attempt to account for the results of this investigation. Substructure seemed to be the most promising model for explaining the observed behavior. However, the evidence is inconclusive.

Research Chemicals Inc. WADC TR 57-666 Part II. SELECTION AND EVALUATION OF RARE OR UNUSUAL METALS PART II. THE METALLURGY OR YTTRIUM AND THE RARE EARTH METALS. Bernard Love. AF 33(616)-5905. March 1959. ASTIA Document No. AD 211847. PB 151825. Order from OTS $3.00.

Studies have been made of the properties of yttrium and the rare earth metals. These have included metallographic studies, oxidation and corrosion resistance studies, mechanical property determination and studies of alloys of titanium and beryllium with rare earth additions.

Improved methods for the production of pure metals are described. A complete metallographic procedure has been developed. Procedures for the chemical and spectrographic analysis of metals and alloys are provided including a method for oxygen analysis.

Corrosion studies include determination of the rate of moisture absorption of the rare earth oxides under controlled conditions of temperature and humidity, and determination of the rate of atmospheric corrosion of the metals in dry and humid atmospheres at 35 and 95°C, and at 200, 400, and 600°C. The rate of atmospheric corrosion was found to vary widely; from europium, a fresh surface of which starts to oxidise immediately on exposure to air, to samarium for which oxidation was very slow even at the elevated temperatures.

Mechanical properties of case and wrought rare earths are presented for both room and elevated temperatures. Tensile properties vary widely. Yttrium and the heavy rare earths appear to have the best mechanical properties, and maintain a considerable percentage of room temperature strength to temperatures of 427°C (800°F).

The methods of preparation, analysis, and examination of titanium and beryllium alloys containing up to 10% rare earth elements are presented. Tentative partial constitutional diagrams are constructed. Very low solubility was found except for lanthanum which had an indicated solubility between 0.2 and 1.5 weight percent.

Massachusetts Institute of Technology. WADC TR 59-57.
THE EFFECT OF SILICON ON THE TEMPERING OF HIGH STRENGTH STEELS. C. J. Altstetter, B. L. Averbach, Morris Cohen. AF 33(616)-5161. May 1959. ASTIA Document No. AD 214426.

The tempering of steels with a base composition of AISI 43XX and up to 3% silicon was studied by measuring longitudinal mechanical properties and kinetic behavior. The kinetic analysis was performed on precision-length measurements after isothermal tempering treatments from 72° to 850°F; mechanical properties were evaluated after one hour tempers at temperatures up to 1300°F.

It was found that silicon affects the temperature at which the first-stage decomposition products transform to cementite and ferrite, although silicon has no marked influence on the first stage itself. In steels without added silicon, the transformation of low-carbon martensite and epsilon carbide to ferrite and cementite (which is the third stage in iron-carbon alloys) is split into two substages because of the stabilizing effect of the alloying elements on epsilon carbide. The first substage is the transformation of low-carbon martensite to thin-plate cementite and ferrite, and the second is the resolution of epsilon carbide and the precipitation of cementite. In silicon steels, the last two reactions occur simultaneously because of the higher transformation temperatures.

The changes in the matrix and carbide phases are reflected in the mechanical properties. A decrease in matrix carbon content causes a decrease in hardness and ultimate strength. Likewise, an agglomeration of carbide also causes a decrease in these properties. The influence of silicon on the as-quenched hardness is considered to be due, at least in part, to its effect on quench-tempering. The "500°F embrittlement phenomenon" is attributed to the morphology of the first-formed cementite. Silicon permits tempering to higher temperature by inhibiting both the softening effect of carbon depletion from the matrix as well as the embrittling effect of cementite precipitation.

Metals and Brazing Alloys

TR 4796.
AIRCRAFT QUALITY CASTING STANDARDS. Robert Katz. July 1942.

TR 4814.
STUDY OF MATERIALS USED IN AIR FRAME OF JUNKERS 88 AIRPLANE. Morse Hill. August 1942.

TR 5925.
PROCEDURES USED IN PRECISION CASTING FOUNDRY. William F. Davenport, 1/Lt. and Adolph Strott. September 1949.

TR 6191.
PRECOAT MATERIALS FOR INVESTMENT CASTING. William F. Davenport, 1/Lt. and Adolph Strott. August 1950.

TR 6222.
STUDY OF INVESTMENTS FOR PRECISION CASTING PROCESS. William F. Davenport, 1/Lt. and Adolph Strott. October 1950.

TR 6678.
SURVEY OF CRITICAL AND STRATEGIC METALS. Howard J. Siegel, 2/Lt. November 1951.

Data are presented pertaining to three groups of critical metals (structural, ferroalloy, and plating metals) relevant to applications, production (world and domestic), and substitutes for these metals.

University of California. TR 52-101, Part 1.
EQUIPMENT FOR TESTING THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS. Lawrence A. Shepard and John E. Dorn. AF 33(038)-11502. July 1952. ASTIA Document No. AD 167643. PB 124052. Order from LC Mi $2.70, Ph $4.80.

Very little is known about the separate or combined effects of intermittent heating and stressing on the elevated temperature creep and creep-rupture characteristics of aircraft structural metals. Such information is important since aircraft and engines will be subject to these conditions. Between flights, loads and temperatures are low; during flights, they are high. Exact service conditions cannot be reproduced in the laboratory, but arbitrary cycles have been chosen for initial work. In view of the need for more complete information on the effects of such intermittent heating and stressing on the creep and creep-rupture properties of structural sheet materials, an experimental program on this subject was initiated. (Possible theoretical analyses of the results will be made later.) One of the major problems of the program was the development of suitable equipment for the investigation. A description of four creep testing machines, specially designed for this program, with automatic electronic control units is given herein. This equipment is designed to produce any combination, separately or simultaneously, of intermittent heating and stressing of creep-rupture specimens, in or out of phase.

University of Illinois. TR 52-89, Part 1.
PLASTIC BEHAVIOR OF ENGINEERING MATERIALS. PART 1 - AXIAL TENSION AND BENDING INTERACTION CURVES FOR MEMBERS LOADED INELASTICALLY. D. O. Brush, O. M. Sidebottom, and J. O. Smith. AF 33(038)-15677. August 1952. PB 116839. Order from LC Mi $2.75, Ph $6.50.

This paper presents a theoretical method for constructing dimensionless interaction curves for members subjected to combined tension and bending loads that produce inelastic strains, and presents experimental results which verify the theory. Each interaction curve represents the total range of the ratios of axial load to bending moment which will cause inelastic strains to extend a given depth in the member. Experimental interaction

curves were obtained from eccentrically loaded tension members of rectangular cross sections made from three strain hardening materials, namely, annealed rail steel and aluminum alloys 24S-T4 and 75S-T6. Good agreement was found between theory and experiment. In order to design a member subjected to combined axial and bending loads by use of the interaction curves, the lateral deflection of the member must be estimated. Three orders of approximation for the lateral deflection of eccentrically loaded tension members are presented. The problem of combined bending and axial compressive loads is discussed and research based on the methods of analysis developed in this investigation is suggested for solving the buckling load of a member subjected to combined bending and axial compressive loads. Some illustrative problems are solved in the appendix which show how the results of this investigation may be used.

Battelle Memorial Institute. TR 6731, Part 1. SHORT-TIME CREEP PROPERTIES OF STRUCTURAL SHEET MATERIALS FOR AIRCRAFT AND MISSILES. J. A. Van Echo, L. C. Page, W. F. Simmons, and H. C. Cross. AF 33(038)-8743. August 1952.

This project was undertaken to determine the short-time creep strengths of several aircraft structural sheet materials at times up to approximately 100 minutes.

The materials tested included two magnesium alloys, three aluminum alloys, four steels, and two high-temperature alloys. Each group of materials was tested over the useful temperature range for the material in that group.

The testing procedure was to load the specimen cold, heat to the desired temperature by resistance heating, and hold at the test temperature until the test was completed. A slide-wire extensometer was used and the total extension of the specimen was recorded on a high-speed General Electric recorder.

The data are presented as time-deformation curves, design curves, and curves comparing the materials after one, ten, and sixty minutes of loading. Among the light alloys, 24S-T had the highest strength at all test temperatures. Steels containing titanium and boron had high strength but were very brittle. The high-temperature alloy L-605 in the hot-rolled condition had very good properties between 1200° and 2000°F.

Armour Research Foundation of Illinois Institute of Technology. TR 6517, Part 2. DETERMINATION OF PHYSICAL PROPERTIES OF FERROUS AND NON-FERROUS STRUCTURAL SHEET MATERIALS AT ELEVATED TEMPERATURES. D. D. Doerr. AF 33(038)-8681. December 1952.

In order to establish important design criteria, the compressive, bearing and shear properties have been determined for (1) XA78S-T6 aluminum alloy sheet (clad), and FS-1a magnesium alloy at room temperature and at elevated temperatures ranging from 212° to 600°F, for exposure periods between 0.5 and 1,000 hours, (2) Heat Treat Nos. 1 and 2 of SAE 8630 alloy steel, SAE 4130 alloy steel, annealed stainless steel, and half-hard stainless steel, at room temperature and at several elevated temperatures between 400° and 1200°F for exposure periods ranging from 0.5 to 100 hours. These properties have been compared with the tensile data obtained under corresponding conditions in an attempt to establish whether a correlation exists which would permit prediction of the compressive, bearing and shear characteristics from the tensile properties of the individual material at room and elevated temperatures.

The test specimens, equipment, and procedures are described in detail. The test results are presented in the form of tables and curves to illustrate the effect of temperature and exposure time on the physical properties of the various materials under investigation.

Armour Research Foundation,Illinois Institute of Technology. TR 6517, Supp. 1. Part 1. DETERMINATION OF PHYSICAL PROPERTIES OF NON-FERROUS STRUCTURAL SHEET MATERIALS AT ELEVATED TEMPERATURES. PART 1, SUPPLEMENT 1 - TYPICAL STRESS VS. STRAIN AND STRESS VS. DEFORMATION CURVES. D. D. Doerr. AF 33(038)-8681. February 1953.

This supplement has been prepared to present typical stress-strain curves in tension and compression, and stress-deformation curves in bearing for all materials tested on the basic contract. These curves were drawn from the original data obtained by laboratory tests. In presenting this information on the aluminum and magnesium alloys, families of curves were drawn for each test temperature in which time is a parameter. Therefore, each figure consists of five curves which represent the stress-strain or stress-deformation relationships existing at each elevated temperature for exposure times of 0.5, 2, 10, 100, and 1000 hours. For the titanium materials, each figure consists of five curves illustrating the stress-strain or stress-deformation relationships which exist at each of the two exposure periods at temperatures of 78, 400, 600, 800, and 1000°F.

These curves are presented for (1) 24S-T3 aluminum alloy at room temperature and at elevated temperatures between 212° and 700°F with exposure periods ranging from 0.5 to 1000 hours, (2) 75S-T6 aluminum alloy, FS-1H and Mh magnesium alloys at room temperature and at elevated temperatures ranging from 300 to 600°F for exposure periods between 0.5 and 1000 hours, and (3) annealed and cold-rolled titanium materials at room temperature and at several elevated temperatures between 400° and 1000°F for exposure periods of 0.5 and 100 hours.

Battelle Memorial Institute. TR 6731, Part 2. SHORT-TIME CREEP PROPERTIES OF STRUCTURAL MATERIALS FOR AIRCRAFT AND MISSILES. METHOD OF TESTING FOR SHORT-TIME HIGH-TEMPERATURE CREEP PROPERTIES OF SHEET MATERIALS. J. A. VanEcho, Ward F. Simmons, H. C. Cross. AF 33(038)-8743. May 1953.

This report describes the methods employed in the testing of aircraft structural sheet materials for times ranging from a few seconds up to approximately 100 minutes. The methods and procedures described herein, which were used in obtaining test results and design curves published in AF Technical Report 6731, Part 1, are presented in detail. Such information is no longer generally available and modifications have been incorporated in their current usage.

This report covers all phases of the testing procedures, including the test specimen shape and size, testing frame and loading system, main heating power supply and control, temperature-measurement and control, and strain-measurement and recording system.

Methods of reporting test results are shown for a previously tested aluminum alloy. These results are given in forms of time-deformation curves, design curves and stress-total deformation curves.

Univ. of California.
WADC TR 52-101 Part 2.
EQUIPMENT FOR TESTING THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS. Lawrence A. Shepard, Carl D. Wiseman, C. Dean Starr, John E. Dorn. AF 33(038)-11502. August 1954. PB 121265. Order from OTS $0.75.

Four creep testing units were constructed at the University of California to test the creep strength of aircraft metals under intermittent loading and heating conditions. The equipment was designed to permit operations of loading, unloading, heating and cooling of the test specimen to occur automatically and periodically on a preset cycle. Special care was taken to provide smooth and vibration-free function of the equipment. Provisions were made for the accurate and continuous recording of both strains and temperature throughout the test.

Stevens Inst. of Technology. WADC TR 54-183. THE INVESTIGATION OF POROUS MEDIA PREPARED FROM SPHERICAL METAL POWDER PARTICLES. Frank W. Heck, Donald P. Ferriss, Lambert H. Mott, Gregory J. Comstock. AF 33(616)-396. March 1955. ASTIA Document No. AD 66388. PB 133576. Order from LC, Mi $11.10, Ph $41.10.

The processing of spherical stainless steel powder particles by compacting and sintering methods is described. Room temperature air permeability and tensile strength and stress rupture data at 1100°F are reported.

Porous specimens were prepared from six particle size fractions to densities ranging from 75% to 90% of the density of the solid material and the thicknesses ranging from 0.040 inch to 0.200 inch by various combinations of compacting and coining pressures. Sintering time, temperature, and atmosphere were held constant. Methods of separating spherical particles from an aggregate of particle shapes are discussed.

The permeability data are presented in the form of a D'Arcy coefficient of permeability and also as a relationship between the pressure differential across the compact and the volumetric rate of air flow through the compact.

Horizons, Inc. WADC TR 54-598. RETAINER MATERIALS FOR AIRCRAFT GAS TURBINE BEARINGS. Eugene J. Bucur, F. Clifton Wagner. AF 33(616)-2099. March 1955. ASTIA Document No. AD 74305. PB 131349. Order from OTS $1.50.

A study of the elevated temperature wear resistance of potential cage materials for aircraft turbojet bearings was conducted using a special wear testing machine to simulate roughly the conditions to which such bearings were subjected in service. All tests were conducted in the Mechanical Metallurgy Department of Horizons Incorporated. It has been found that:

1. Several alloy compositions have been developed which have superior bearing properties than "S" Monel and iron-silicon bronze. All of the promising materials except one contain silver as a major alloying element.
2. It has been established that the addition of from 2 to 4% silicon is distinctly beneficial to the wear properties of several classes of metallic alloys
3. It has been shown that the alloy composition can be varied considerably with respect to the strong load supporting phase as long as silver is contained in the soft matrix.

Battelle Memorial Institute. WADC TR 55-34. AN INVESTIGATION OF MATERIALS FOR CAGES FOR AIRCRAFT GAS-TURBINE ROLLING-CONTACT BEARINGS. W. A. Glaeser, C. M. Allen, S. L. Fawcett. AF 33(616)-2100. April 1955.

An investigation has been carried out to develop new materials for cages for rolling-contact bearings used in jet aircraft. Two machines have been constructed to evaluate prospective materials. There are the rubbing-button-test machine and the cage-material testing machine. The rubbing-button machine is designed to screen the various materials in the simplest way possible, evaluating their resistance to galling and to seizure under thin-film lubrication at high rubbing velocities. The cage-material testing machine was designed to further evaluate promising materials from the screening tests under conditions simulating actual turbine-bearing operation. Material comparisons are based on the performance of silicon-iron bronze in these machines. At present, this cage material is used more widely than any other material for turbine bearings.

Two promising materials have been developed in the course of this research. These are chromized steel and boron nitride cermet. These materials have exhibited good corrosion resistance to hot MIL-L-7808 products used in the lubrication of turbine bearings.

An initial series of runs using the cage-testing machine with silicon-iron bronze specimens has proved the machine to be a valuable tool for simulating cage-pocket wear by the rolling elements. Comparison of cage specimens from this machine with cages from a bearing operated in a high-speed turbine showed marked similarity in the wear patterns. The loads on the cage pockets were measured with a strain-sensing device.

Battelle Memorial Institute. AFTR 6731 Part 3. SHORT-TIME CREEP PROPERTIES OF STRUCTURAL SHEET MATERIALS FOR AIRCRAFT AND MISSILES. John A. Van Echo, W. F. Wirth, Ward F. Simmons. AF 33(038)-8743. May 1955.

Short-time, high-temperature creep properties are reported for several structural aircraft sheet materials. The materials include a titanium alloy, three Alclad aluminum alloys, four low-alloy steels, and two stainless steels. Each group of materials was tested over a useful temperature range. The time of interest for each material ranged from about 1/2 to 120 minutes.

The data obtained are reported as (1) time-deformation curves, (2) design curves including total deformations, (3) stress-total deformation curves which rate the materials after 1, 10, and 60 minutes in test, and (4) creep-design curves. The creep-design curves contain only actual creep deformation in the range of 0.1 to 5 percent.

This report is Part 3 of a project that was undertaken to obtain the aforementioned data.

Battelle Memorial Institute. AFTR 6731 Part 4. SHORT-TIME CREEP PROPERTIES OF STRUCTURAL SHEET MATERIALS FOR AIRCRAFT AND MISSILES. John A. Van Echo, Damian V. Gullotti, Ward F. Simmons, James R. Bibler. AF 33(038)-8743. January 1956.

Creep properties are reported for several commercial sheet materials including RC-70 titanium, 61S-T6 and 75S-T6 aluminum alloys, SAE 4130 steel, the heat-resistant Alloy Steel A-286, and two stainless steels--17-7PH and Type 410. The test results were obtained under special short-time (1/2 to 120 minutes), rapid-heating (50 to 250°F per second) test conditions to simulate service conditions. Each alloy was tested over the useful temperature range for the particular material.

Applications of these alloys under special short-time conditions might include skin material for supersonic

vehicles, combustion chambers for rockets and missiles, and other structural parts in aircraft and missiles.

This report is Part 4 of a project that was undertaken to obtain the creep properties of numerous sheet materials. Previous work was similarly done on a wide range of other commercial and some experimental sheet materials.

Battelle Memorial Inst. WADC TR 56-83.
AN INVESTIGATION OF THE EFFECTS OF HYDROGEN ON THE BRITTLE FAILURE OF HIGH-STRENGTH STEELS. Edward R. Slaughter, E. Ellis Fletcher, Arthur R. Elsea, George K. Manning. AF 33(616)-2103. April 1956. PB 121357. Order from OTS $1.50.

The effect of hydrogen on the delayed, brittle failure of high-strength steel was investigated by the use of room-temperature stress-rupture tests on unnotched, cathodically charged specimens. The principal variables in these tests were stress, strength level, structure, composition, and hydrogen content. The time for failure to occur decreased as the strength level of the steel or the applied stress was increased, but changes in structure or composition had little effect on the time to failure.

The martensite and bainite reactions were found to be unaffected by the variations in hydrogen content.

The diffusion of hydrogen through cathodically charged steel was investigated. In this study, stress was found to have no measurable effect on either the diffusion or the distribution of hydrogen in steel.

Hydrogen contents of steel cathodically charged under various conditions were determined.

Crucible Steel Company of America.
WADC TR 54-472 Part 2.
DEVELOPMENT OF AUSTENITIC IRON-BASE SHEET ALLOY. Roy R. Rothermel. AF 33(616)-2047 May 1956. PB 121438. Order from OTS $0.79.

Results of the investigation of the weldability of a type II, AD30 alloy (conducted in the welding laboratory of the contractor) indicate that this material can be welded when adequate measures are taken to limit stresses set up during welding and where proper protection from the atmosphere is afforded the material during welding.

Satisfactory welds were made in representative types of weld joints by the metallic arc process using types 310 and 312 mod. (2-3% Mo) electrodes and by the inert gas process using AD30 filler.

Satisfactory welds were obtained by the butt flash-welding process.

Bend test results of various welded specimens show good ductility, and evidence of formability of a welded section in the as-welded, solution treated condition.

Short-time tensile test results indicate welded specimens to have strengths (at a 1500°F testing temperature) comparable to the AD30 base material.

Univ. of Minnesota. WADC TR 56-282.
THE EFFECT OF STRESS ON MICROHARDNESS. F. H. Vitovec. AF 33(616)-2803. August 1956. ASTIA Document No. AD 97201. PB 121534.

Microhardness is investigated as a function of test load for mild steel, Armco iron, copper, brass, zinc and cadmium. At light loads a discontinuity of slope in the hardness-load curve is observed which can be attributed to subgrain structure. A tension-compression fixture was developed and used to study the effect of stress on the microhardness of mild steel, brass, and copper specimens. For initially stress-free specimens, tensile stress causes a decrease in microhardness over a certain range of test load, and compressive stress results in a slight increase. The effect of stress on microhardness is complicated by the discontinuous slope in the hardness-load curve introduced by subgrain structure effects.

Horizons Inc. WADC TR 56-294.
RETAINER MATERIALS FOR AIRCRAFT GAS-TURBINE BEARINGS. Peter F. Mataich, F. Clifton Wagner. AF 33(616)-2949. August 1956. ASTIA Document No. AD 97235. PB 121935. Order from OTS $1.00.

This study was made with the purpose of developing new cage materials to be used in gas-turbine jet engines. The materials being used presently are operating near their upper temperature limits and the demands for greater efficiency and power will necessitate the use of materials with high temperature wear characteristics superior to those presently being used.

The development work was concentrated on nickel base silver impregnated materials produced by powder metallurgy techniques. Various boride, carbide, and silicide additions were made to the nickel base, and several compositions were found which had superior wear characteristics. In addition to this work a series of oil evaluation tests were made, and the wear rates of both the new and the standard materials were determined while using these oils as lubricants.

The Ohio State Univ. Research Foundation.
WADC TR 56-242.
STRESS CORROSION CRACKING IN TYPE 403 STAINLESS STEEL. Mars G. Fontana. AF 33(616)-2849. August 1956. ASTIA Document No. AD 97215. PB 121414.

The effect of austenitizing temperature on the hardness, impact strength, and microstructure of Types 403, 402, and 431 stainless steel was determined. The optimum combination of these properties occurred with austenitizing temperatures of 1725°, 1850° and 1900°F respectively for the 403, 420, and 431 materials. Tempered structures displayed minimums in the impact strength, tempering temperature curves at 1000°, 900° and 1000°F for the 403, 420 and 431 materials respectively.

Metallographic studies were made of stress-corrosion specimens tested in a 1:1 HCl and water solution containing 1% SeO_2. These studies showed that pitting was initiated at MnS inclusions and that cracking was associated with the pits. Also, pitting characteristics were dependent on tempering temperatures and therefore are related to the microstructure of the alloy. Cracking does not occur in Type 403 alloy when it is tempered at 1050°F or higher. This temperature is lowered to 900°F for the Type 420 material, and it is less than 700°F for Type 431 stainless steel stressed to 75,000 psi and tested in the above solution.

Electron microscopy and diffraction studies were made of tempered Type 403 stainless steel. These studies indicate several carbides to be present in the alloy after tempering in the 1000° - 1200°F temperature range. Diffraction patterns vary for specimens tempered in this range.

National Bureau of Standards. WADC TR 56-402.
RESEARCH ON EFFECTS OF PRESTRAINING AND NOTCH SHARPNESS ON THE NOTCH STRENGTH OF MATERIALS. G. W. Geil, N. L. Carwile. AF 33(616)-55-9. October 1956. ASTIA Document No. AD 110436. PB 121782.

A general study was made on the effect of prestraining in tension at +25°C on the tensile properties at -196° and +100°C of notched and unnotched specimens of (1) a commercially pure titanium, Ti-75A, and (2) a 4% Al, 4% Mn titanium alloy, C130AM. Included in this general study were (1) a preliminary investigation on both materials to determine the effect of the notch sharpness, as varied by a change in root radius or notch depth, on the tensile

properties, (2) a study of the combined effect of notch sharpness (as varied by a change in notch depth) and prestrain at +25°C on the tensile properties of notched specimens of these materials at -196° or +100°C, and (3) a determination of the impact properties of Charpy V-notch specimens of both metals over the temperature range of -196° to +300°C.

Prestraining in tension at +25°C had considerable effect on the ductility retained by both the notched and unnotched specimens of each material at -196° or +100°C. Furthermore, the retained true strain at initial fracture (total true strain at initial fracture in the second stage of test at -196° or +100°C minus the true strain at the end of the first stage of test at +25°C) decreased with increase in the prestrain at +25°C. The results indicate that Ti-75A and C130AM are not subject to "rheotropic embrittlement" as the retained ductility of the specimens at -196°C was not improved by prestraining at +25°C.

Prestraining in tension at +25°C has little, if any effect on the tensile strength or notch strength of Ti-75A or C130AM, except for some impairment in the notch strength of the deep-notched specimens of C130AM that did not retain any appreciable ductility at -196°C.

The change from a ductile to brittle behavior in the impact tests on Charpy V-notch specimens (based on the energy absorbed in fracturing the specimens) occurred over temperature ranges of approximately +150° to +100°C and +200° to +100°C for Ti-75A and C130AM, respectively.

Battelle Memorial Institute. WADC TR 56-486. EVALUATION OF POROUS MATERIALS FOR BOUNDARY-LAYER CONTROL. David E. Debeau. AF 33(600)-28835. November 1956. ASTIA Document No. AD 110582. PB 121851.

Criteria were determined from designers for comparing various commercial permeable sheet materials for use in boundary-layer control associated with high-lift systems. Sintered metal powders, special woven and sintered wire materials, a compressed glass-fiber product, woven wire cloths, and perforated metal sheets were evaluated and compared for the following properties: average permeability, uniformity of permeability, resistance to clogging and corrosion, mechanical properties including room temperature tensile strength, modulus of elasticity, Poisson's ratio, and minimum bend radius. An evaluation of economic factors, including production facilities, product limitations, requirements for and availability of raw materials, and cost of product, was carried out on those permeable sheet materials which attained most nearly the physical and mechanical properties desired by the aircraft designers.

Univ. of Illinois. WADC TR 56-330 Part I. THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS. Part I: Eccentrically-Loaded Tension Members Having Angle- and T-Sections. O. M. Sidebottom, M. E. Clark. AF 33(616)-2753. April 1957. ASTIA Document No. AD 118178. PB 136061.

This paper presents the results of an analytical and experimental investigation for the determination of the load necessary to produce any given depth of yielding in angle- and T-section members subjected to eccentric tensile loading. This load is found analytically from the intersection of a constant depth of yielding interaction curve and a moment-load curve. General load and moment expressions used in the construction of the interaction curves were derived for cross-sections composed of rectangular elements. The primary assumption in these derivations was that for small inelastic strains the stress-strain diagrams of the material could be represented by two straight lines. The moment-load expressions were derived on the assumption that each eccentrically loaded member deformed into a segment of a circle.

In the experimental investigation eccentric load tests were made on angle- and T-section members made from various classes of materials. The three materials used were SAE 4340 alloy steel, type 304 stainless steel, and 7075-T6 aluminum alloy. The angle-section members were tested with the principal axes orientated in three different positions with respect to the loading plane. In general the results of these tests corroborated the theoretical analyses. It was found that deviations from the theory could be explained by variations in the stress-strain properties of the material.

Supplementary tests were performed to determine the effect of pure bending loading and the effect of time on the inelastic behavior under sustained and alternating load schedules. Pure bending test results indicated that the theory proposed was conservative in the majority of cases. The results of the sustained and alternating load tests indicated that time was not an important variable so long as the material properties used in the analysis were obtained from specimens subjected to loading schedules similar to those used in testing the eccentrically loaded member.

Univ. of Illinois. WADC TR 56-330 Part II. THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS. Part II: Inelastic Behavior of Aluminum Alloy I-Beams with Rectangular Web Section Cutouts. Will J. Worley, Shuji Taira. AF 33(616)2753. April 1957. ASTIA Document No. AD 118185. PB 131028.

This report contains an exploratory investigation of the plastic bending of aluminum alloy I-beams with rectangular web section cutouts. The mechanism method of analysis employing the upper bound theorem was employed in predicting the ultimate loads of the various beams. The experimental results are in good agreement with the predicted ultimate loads.

Univ. of Illinois. WADC TR 56-330 Part IV. THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS. Part IV: Eccentrically-Loaded Columns Having Angle- and T-Sections. M. E. Clark, O. M. Sidebottom. AF 33(616)-2753. May 1957. ASTIA Document No. AD 130765. PB 131252.

This paper presents the results of an analytical and experimental investigation for the determination of the load necessary to produce any given depth of yielding in angle and T-section columns subjected to eccentric loads. This load is found analytically from the intersection of a constant depth of yielding interaction curve and the moment-load curve (see Part I of this Report). The collapse load of the column can be taken as the maximum load given by the moment-load curve.

In the experimental investigation, tests were made on eccentrically-loaded angle- and T-section columns made from type 304 stainless steel. The angle-section columns were so oriented that some buckled about the minimum axis of inertia and some about the maximum axis of inertia. The effects of variations in initial eccentricity and slenderness ratio on the collapse load were investigated. In all cases the experimental and theoretical moment-load curves were of the same general shape; the experimental collapse load being less than the theoretical by 2 to 16 percent. Since

the theory is based on assumptions which make it conservative, the fact that the experiments showed it to be unconservative was attributed to the time-sensitive behavior of the stainless steel material. The stress-strain properties obtained from tension and compression tests did not represent the stress-strain behavior of the material in the column. When these stress-strain properties were adjusted by a 28 percent reduction in the modulus of elasticity, the experimental collapse load varied from 9 percent greater to 11 percent less than the theoretical value with most of the values comparing very favorably.

Univ. of Illinois. WADC TR 56-330 Part V. THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS. Part V: Inelastic Behavior of Aluminum Alloy I-Beams with Elliptical Web Section Cutouts. Will J. Worley. AF 33(616)-2753. May 1957. ASTIA Document No. AD 130768. PB 131245.

This report represents an extension of WADC Technical Report 56-330, Part II, April, 1956.

This extension was undertaken to investigate the plastic bending behavior of aluminum alloy I-beams with elliptical web section cutouts. As in the above report, the mechanism method of analysis employing the Upper Bound Theorem was used in predicting the ultimate loads of the various beams. This procedure could not be applied in all cases since several of the beams failed by buckling failures in the web sections. The I-beams were loaded in pure bending using quarter point loading and in combined shear and bending using center point loading. The span lengths were varied for a given depth of I-beam in order to investigate the effects of shear on the failure mechanism. Where the mechanism analysis applies, it permits the prediction of loads which are in good agreement with the experimental results.

Univ. of Illinois. WADC TR 56-330 Part VI. THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS. Part VI: A Digital Computer Analysis of Bending Moment-Axial Load Interaction Curves. Y. Maeda, P. Van Lierde, O. M. Sidebottom, M. E. Clark. AF 33(616)-2753. May 1957. ASTIA Document No. AD 118300. PB 131182.

This paper presents the results of a study undertaken to determine the feasibility of using the digital computer to make the time-consuming calculations necessary to construct moment-load interaction curves. A strip method of numerical integration was set up to solve the equations of equilibrium for the members; this method allowed a generalization of the procedure to all shapes of cross-section.

In setting up the problem for the digital computer, it was not convenient to work with the actual stress distributions. Instead, it was found that the actual stress distribution could be represented by three linear stress distributions having the same stress gradient. Load- and moment-functions were computed from these stress distributions and the actual load and moment in the section were determined from these load- and moment functions.

Load- and moment-functions for some 86 different conditions for T-, angle-, and channel-section members were determined using the digital computer. The great number of additional computations required to construct the interaction curves once the load-and moment-functions are available renders the digital computer method reported herein of questionable use. Further study of the problem could possibly produce methods by which these additional computations could be made by the computer and thereby make the method more efficient and useful.

Univ. of Illinois. WADC TR 56-330 Part III. THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS. Part III: The Plastic Bending of Tapered Members. Montgomerie C. Steele, Hassan A. Hassan. AF 33(616)-2753. June 1957. ASTIA Document No. AD 130865. PB 131373.

This report contains an exploratory investigation of the plastic bending of tapered members. Two analytical approaches are employed. The first considers a rigorous treatment by the classical theories of elasticity and plasticity while the second resorts to the more tractable mechanics of materials methods. A limited amount of experimental work is presented in support of theory.

WADC TR 57-131. THE SAFETY OF AIRCRAFT STRUCTURES. A. M. Freudenthal. AF 33(616)-2729. July 1957. ASTIA Document No. AD 130910. PB 131382.

The concept of structural safety of airframes is analyzed on the basis of its relation to the probability of structural failure, with a view of establishing procedures of quantitative evaluation of safety factors for a predetermined "acceptable" risk of failure.

The difference in the approach to the concept of safety for ultimate strength and for fatigue is discussed, considering recent developments in fatigue research, particularly the results of fatigue tests under random loading, and methods of safety analysis for both conditions are proposed.

In this analysis the "limit load" or "limit load factor" is a basic concept. It should, however be noted that this concept is not identical with the conventional structural design criterion of the same name. The difference is fundamental: while in conventional design the "limit load" is a derivative concept obtained simply by dividing the "ultimate load" by an arbitrary "safety" factor, usually 1.5, the concept as used here is the primary load criterion defining, independently of the ultimate load, a limiting condition of service by the aid of which fatigue design and design for limit load can be correlated.

Since it is a purpose of the present analysis to discuss and develop procedures for the rational evaluation of safety factors, expediently defined in terms of ratios of the ultimate to the limit load factor, it is obvious that both load factors have to be independently derived from operational criteria. Thus, while the meaning of the concept of "ultimate load" used in this report does not differ significantly from its meaning in conventional design, the "limit load" concept is significantly different.

WADC TR 57-311. EFFECT OF LOADING RATE ON THE STRENGTH OF SINGLE AND MULTIPLE RIVETED JOINTS. Richard F. Klinger. September 1957. ASTIA Document No. AD 131098. PB 131513.

Slow and rapid loading tensile shear tests were conducted at room temperature on single and multiple rivet lap and butt joints. The joints were manufactured with a standard pneumatic type rivet squeezer using 2117-T3 aluminum alloy rivets, 1/8 inch diameter, and Alclad 2024-T3 aluminum alloy sheet, 0.064" thick. The ultimate strengths of the joints were determined with times to the ultimate load of 0.02 and 60 seconds. All failures were by rivet shear.

The results indicate that the effect of rapid loading is to increase the strength of single riveted joints that this effect is diminished as the number of rivets in the joint increases and that when the number of rivets in the joint is four and above the effect of rapid loading is to decrease the joint strength slightly.

Univ. of Illinois and Convair.
WADC TR 56-330 Part VII.
THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS. Part VII: Inelastic Behavior of Aluminum Alloy I-Beams with Elliptic-Type Web Section Cutouts. Will J. Worley, Fred D. Breuer. AF 33(616)-2753. December 1957. ASTIA Document No. AD 142217. PB 131556.

This report represents a continuation of WADC Technical Report 56-330, Part II, April 1957 and Part V, May 1957.

This continuation was undertaken to investigate the fully plastic bending behavior of aluminum alloy I-beams with elliptic-type web section cutouts. As in the two earlier reports, the mechanism method of analysis employing the Upper Bound Theorem was used in predicting the ultimate loads of the various beams. The I-beams were loaded as simple beams with center loading. The mathematical relations were developed to enable solution by means of an electronic digital computing machine, the Illiac. The results obtained on the Illiac indicated that a diamond shape web section cutout was the most favorable type for resisting fully plastic bending. Tests were conducted with substantiated the results obtained on the Illiac.

Armour Research Foundation. WADC TR 57-648.
DEVELOPMENT OF OXIDATION AND LIQUID SODIUM RESISTANT BRAZING ALLOYS. Domenic A. Canonico, Harry Schwartzbart. AF 33(600)-3406. March 1958. ASTIA Document No. AD 151013. PB 131745.

The first year's work has been completed at Armour Research Foundation on a program which has as its objectives: (1) the development of filler metals for brazing type 310 stainless steel to Inconal, the brazements to be resistant to oxidation and attack by molten sodium at 1650°F, and (2) the development of a simple test for the ductility of a brazed joint.

Three metals -- iron, chromium, and nickel -- were selected, from oxidation and sodium resistance considerations, as the base metals from which to develop acceptable brazing alloys. From a study of all the available pertinent phase diagrams 91 alloys were devised, of which 16 satisfied the flow temperature and flowability requirements of the program. The flow temperature was between 1750°F and 1900°F. Minimum flowability was three inches of length along a six-inch T-specimen.

T-specimens brazed with these 16 alloys were subjected to an oxidizing atmosphere for 500 hours at 1650°F. Four alloys successfully withstood oxidation to a depth of less than 0.003 inches.

A test for ductility of brazed joints, based on the premise that the load necessary to cause cracking in the fillet of a braze is a measure of the ductility of the brazing alloy, has been developed. The test has shown itself to be capable of differentiating two filler alloys, although the large amount of scatter obtained required that a statistical analysis of the data be applied. Further development should reduce the scatter.

Stanford Research Institute. WADC TR 57-695.
DETERMINATION OF THE MECHANICAL PROPERTIES OF A HIGH PURITY LEAD AND A 0.058% COPPER-LEAD ALLOY. Thomas E. Tietz. AF 33(616)-3785. April 1958. ASTIA Document No. 151165. PB 131818.

The mechanical properties of a high purity lead and a 0.058% copper-lead alloy were determined at test temperatures of 100, 175, 250, and 325°F.

Tensile properties evaluated included the ultimate strength, elongation, modulus of elasticity, proportional limit, and yield strength. Compression properties evaluated were the modulus of elasticity, proportional limit, and yield strength. Ultimate shear strength and the bearing yield strength and ultimate bearing strength were determined. Stress-creep time curves were obtained for total strain values of 0.2, 0.5, 1.0, and 2.0%, for creep time of from 1 hour to 500 hours.

The data obtained are summarized in graphic and tabular form, in the Experimental Results section of this report.

Univ. of Illinois. WADC TR 56-330 Part VIII.
THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS. Part VIII: Eccentrically-Loaded Tension Members Made of Two Stainless Steels Tested at Elevated Temperatures. O. M. Sidebottom, M. E. Clark, S. Dharmarajan. AF 33(616)-2753. October 1958. ASTIA Document No. 202495. PB 151673. Order from OTS $1.75.

This report presents the results of an analytical and experimental investigation for the determination of the load necessary to produce a specified amount of inelastic deformation in rectangular-section members subjected to eccentric tensile loads at elevated temperatures. Two different theories were used. In one theory each isochronous stress-strain diagram was represented by two straight lines, and the load and deflection of the eccentrically loaded members were obtained from interaction curves and moment-load curves. In the other theory each isochronous stress-strain diagram was represented by a hyperbolic sine curve, and the resultant theory is called the hyperbolic sine theory.

In the experimental investigation, tests were made on eccentrically-loaded rectangular-section members made of type 304 stainless steel at a test temperature of 1000°F and 17-7 PH stainless steel at test temperatures of 1000°F and 1200°F. Some of the 17-7PH stainless steel specimens were given a precipitation hardening treatment and some were tested in the untreated condition.

The type 304 stainless steel did not creep at 1000°F. The interaction curve--moment-load curve theory was found to give satisfactory results. The heat treated 17-7PH stainless steel was found to have higher strength than the untreated, and it was found to exhibit a small amount of inelastic deformation for zero time but a larger amount of creep in 30 minutes when tested at 1000°F. The untreated 17-7PH was found to creep a small amount at 1000°F and a much greater amount at 1200°F. In each case the isochronous stress-strain diagram could be represented by a hyperbolic sine curve. It was found that the load and/or deflection for the eccentrically loaded members could be adequately represented by either the hyperbolic sine theory or the interaction curve--moment-load theory. The former gave the best results.

Univ. of Illinois. WADC TR 56-330 Part IX.
THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS. Part IX - T-Section Eccentrically-Loaded Tension Members Made of Type 304 Stainless Steel and Tested at 1000°F. O. M. Sidebottom, S. Dharmarajan. AF 33(616)-2753. October 1958. ASTIA Document No. AD 202496. PB 151674. Order from OTS $0.50.

This report presents the results of an analytical and experimental investigation for the determination of the load necessary to produce a specified amount of inelastic deformation in T-section members subjected to eccentric tensile loads at 1000 F. The interaction curve--moment-load curve theory was used in predicting the load and deformation of the eccentrically-loaded members.

The material used in making the test members was type 304 stainless steel. Creep tension tests indicated that this material was extremely time-insensitive, at least for

the first 30 minutes. The stress-strain diagram of the material at 1000°F was similar in shape to that at room temperature.

The comparison between the theory and experiment for the T-section eccentrically-loaded members was excellent for those members with sufficiently small initial eccentricity that yielding did not occur in the most stressed compression fibers. For those members with larger eccentricity, the experimental moment-load curves fell appreciably below the theoretical curve over the range of the curve for which yielding had occurred on the compression side of the member. It is believed that this discrepancy results from the fact that the yield stress in compression was less than in tension.

WADC TR 58-168.
STRESS RELAXATION IN STRUCTURAL MATERIALS. Alfred M. Freudenthal. AF 33(616)-2729. October 1958. ASTIA Document No. AD 202497. PB 151516.

The phenomenon of stress-relaxation is discussed in relation to its physical origin, its mathematical representation, its experimental determination and its design significance, with particular emphasis on the interrelation between creep and relaxation in metals and in polymers.

While the relaxation behavior of materials is of primary importance in design, particularly with regard to flight structures operating under conditions of varying temperatures, it appears that neither the present methods of direct "relaxation testing" nor the conversion of creep data into relaxation data provide useful and reliable information concerning the relaxation behavior of the material.

Univ. of Michigan. WADC TR 57-58 Part II.
NOTCH SENSITIVITY OF AIRCRAFT STRUCTURAL AND ENGINE ALLOYS. Part II: Further Studies with A-286 Alloy. Howard R. Voorhees, James W. Freeman. AF 33(616)-3380. January 1959. ASTIA Document No. AD 207850. PB 151738. Order from OTS $1.75.

Sufficient data on a single lot of A-286 alloy were obtained at 1200 F to permit future evaluation of an analysis for notched-bar rupture life in terms of normal creep-rupture properties.

Circumferential notches with theoretical stress concentration factors (K_t) of 3.0 or less increased rupture time of materials with a 2200°F solution temperature, despite smooth-bar elongations as low as 0.5% or below.

Notch behavior varied widely with notch geometry in tests on specimens with 1800°F solution temperature. Transition to notch sensitivity at longer time periods was, however, positively demonstrated for several notch geometries.

The creep rate of a smooth bar at any time in a variable-stress test appeared to be a unique function of the existing stress level and the cumulative portion of rupture life already expended. No exact criterion was found for rupture of A-286 alloy after creep under variable simple tension; for engineering purposes the portion of rupture life consumed in a time interval (Δt) during which creep deformation (ΔC) occurred could be evaluated by $\frac{(\Delta t)(\Delta C)}{(R)(D)}$, where R and D are the rupture time and creep deformation at rupture in a normal test at the existing stress.

Firm criteria for creep rupture under a general state of variable complex stress are still lacking for the desired evaluation and extension of the proposed calculation methods.

The Brush Beryllium Company.
WADC TR 58-478 Part I.
DEVELOPMENT OF WROUGHT BERYLLIUM ALLOYS OF IMPROVED PROPERTIES. John G. Klein, Leslie M. Perelman, Wallace W. Beaver. DO 33(616)-57-19. February 1959. ASTIA Document No. AD 208663. PB 151711.

A review of the literature on alloying and other structural modifications and their effect on mechanical properties of beryllium is included in this report.

Primary consideration is given to mechanical and physical properties of QMV beryllium, both sintered and wrought, beryllium-rich alloys, and structurally modified metal; fabrication procedures are described.

A discussion of background and methods used to study preferred crystallographic orientation in wrought beryllium is appended.

Alloyd Research Corporation
WADC TR 58-457 Part I.
RESEARCH ON TECHNIQUES FOR THE PRODUCTION OF ULTRA-PURE BERYLLIUM. Joseph L. Lukesh, Laurence M. Schetky, Henry S. Spacil, Malcolm Basche. AF 33(616)-5300. February 1959. ASTIA Document No. AD 208662. PB 151878. Order from OTS $1.25.

The brittleness problem of beryllium is described and the empirical and theoretical approaches to its solution discussed. The Alloyd Research Corporation program for the production of very high purity beryllium is described in detail. Experimental results are outlined, conclusions are drawn, and recommendations for future work presented.

WADC TR 58-615 Part I.
A COMPENDIUM OF CONSTITUTIONAL TERNARY DIAGRAMS OF THE METALLIC SYSTEMS. Dr. W. Guertler, Berlin, Germany. AF 61(052)-74. March 1959. ASTIA Document No. AD 210719.

The purpose of this project is to collect and present a complete collection of ternary phase diagrams of the various metallic systems.

The ternary diagrams presented are built from available binary diagrams. In each instance the most reliable binary diagram has been selected.

The basis for the designation of the pages is the order of atomic numbers. It is thus possible to attribute to each ternary alloy three numbers below 100 corresponding to the three combining elements and to assemble them in order of magnitude from the smallest to the highest figure. Consequently each system can be located easily.

American Brake Shoe Co. WADC TR 59-63.
DEVELOPMENT OF LOW ALLOY STEEL COMPOSITIONS SUITABLE FOR HIGH STRENGTH STEEL CASTINGS. Hugo R. Larson, Ronald C. Campbell, Herbert W. Lloyd. AF 33(616)-5299. June 1959. PB 161044. Order from OTS $2.50.

Cast high strength alloy steels were investigated to develop compositions which had 12% elongation, 30% reduction of area and 15 ft. lb. impact strength when heat treated to 180,000 psi tensile strength. In addition, the ultra high strength level was studied to determine the tensile strength at which a reduction of area in excess of 10% could be maintained.

A series of alloys based primarily on 4340 were poured. The optimum carbon content was established at approximately 0.35%. A lower nickel alloy similar to 8740 had comparable ductility and impact properties to 4340, but showed a consistently higher yield strength. Reduction of area and impact requirements were satisfactorily met, but

elongation values of 12% were not obtained consistently.

High purity raw materials had a definite beneficial effect on ductility both at the 180,000 psi and the ultra high strength level.

Two vacuum melted heats met all of the above requirements quite easily.

Air hardening die type steels were also investigated. Exceptionally good properties were obtained with high purity raw materials and a base analysis of about .30% carbon, 5% chromium, 1.50% molybdenum and 0.50% vanadium.

Simple plate castings of varying thickness and length were poured to investigate the feeding characteristics of the 8740 type steel. The castings were made in ceramic and sand molds and with and without end chills. Tensile bars cut from the castings had properties which correlated well with radiographic soundness of thin slices cut adjacent to the tensile specimens.

Both the 8740 and the 5% chromium steels were poured in three typical airframe castings. Tensile properties in the castings were not as good as from test bars. Although this was expected, the in-casting properties of these particular castings could probably be improved if time were available to revise patterns and rigging systems.

Armour Research Foundation. WADC TR 59-86.
DEVELOPMENT OF ULTRA-HIGH STRENGTH, TEMPER RESISTANT STEELS DESIGNED FOR IMPROVEMENT OF FATIGUE PROPERTIES THROUGH RELIEF OF RESIDUAL STRESS. Harvey B. Nudelman, John P. Sheehan. AF 33(616)-3299. June 1959. PB 151971. Order from OTS $1.00.

Various experimental alloy steels were investigated with reference to temper resistance, which was evaluated on the basis of hardness measurements. Molybdenum was the major alloying element used for improving temper resistance in steels having carbon contents in the range of 0.30 to 0.55 percent. Chromium, vanadium, and tungsten were utilized in minor amounts as alloying elements. It was found that austenitizing at 2200°F was required to dissolve all of the carbides and provide a fully martensitic structure upon oil quenching or air cooling. Alloys were developed capable of maintaining hardnesses of Rc 40 to Rc 59 after tempering for one hour in the range of 1250°F to 1300°F.

Metallurgy Division, National Bureau of Standards.
WADC TR 58-653.
A STUDY OF 17-7 PH STAINLESS STEEL. Nesbit L. Carwile, Samuel J. Rosenberg. AF 33(616)-57-7. June 1959. ASTIA Document No. AD 216528. PB 161089. Order from OTS $1.25.

A study was made of the mechanical properties and microstructures of 17-7 PH stainless steel as affected by heat treatment. Although optimum room temperature mechanical properties were obtained by a treatment slightly different from the TH 950 and TH 1050 treatments recommended by the manufacturer, the best tensile and stress-rupture properties at elevated temperatures (600 and 800°F) were obtained with the TH 950 treatment.

When this steel in either the TH 950 or TH 1050 condition was subjected to prolonged exposure at 800°F, either with or without stress, a definite embrittlement resulted. The material may be restored to approximately its original properties by re-aging.

Although extensive metallographic and X-ray examinations were made to ascertain the mechanism of aging and the precipitation-hardening compound, no definite conclusions were reached. The microstructure of this steel is extremely complex and could be a fertile field for study.

Case Institute of Technology.
WADC TR 59-223.
RESEARCH ON STRAIN AGING EFFECTS IN TITANIUM. Harold S. Gurev, William M. Baldwin, Jr. AF 33(616)-5691. September 1959. ASTIA Document No. AD 229863. PB 161304. Order from OTS $1.75.

The effects of varying interstitial element content (oxygen, carbon and nitrogen) on the tensile properties of an alpha-beta titanium alloy, Ti-140A, were determined at test temperatures ranging from room temperature to above 1000°F and at strain rates from 0.05 to 19,000 in/in/min. The stress rupture properties of such alloys and similar interstitial bearing modifications of an alpha alloy, A-110AT, were also determined between 400°F and 1000°F.

The tensile properties of the alpha-beta modifications were strongly dependent on the nitrogen content alone, while the appearance of a strain rate dependent high temperature embrittlement in an alloy modification heat was governed by the identity of the principal interstitial present. The controlling mechanism for this high temperature embrittlement was found, by comparison of activation energies for the processes, to be the diffusion of the principal interstitial element in, probably, the beta phase. The relatively interstitial free heat also exhibited such embrittlement, but its cause could not be linked to interstitial diffusion. Correlation of embrittlement with manifestations of strain aging noted during testing was not consistent.

A time dependent embrittlement was also discovered during stress rupture testing of alpha alloy modifications at 800°F and 1000°F and in alpha-beta modifications at 400°F.

Crucible Steel Co. of America.
WADC TR 59-390.
DEVELOPMENT OF A CORROSION-RESISTANT BEARING STEEL FOR SERVICE IN AIRCRAFT AT TEMPERATURES UP TO 1000°F. G. Steven, T. V. Philip. AF 33(616)-5428, Phase B. October 1959. PB 161338. Order from OTS $1.75.

The present study is an extension of that completed in the development of the high-temperature bearing alloy WB49 (See WADC TR 57-343, Part II). In the current work corrosion resistance was added to the high initial hardness, adequate temper resistance (in the range 600 to 900°F), and good dimensional stability of the alloy.

To produce secondary hardening, the base composition of a quench hardenable stainless steel, 440C, was modified with carbide-formers V, W, and Mo. Cobalt was added to counteract the ferrite phase field expansion caused by these elements at the austenitizing temperature. By increasing the carbide solubility at high temperatures and thereby raising the chromium content in the matrix, cobalt also enhances the corrosion resistance.

As a result of an evaluation of forty-nine air-melted and seventeen vacuum-melted steels, the following composition is recommended for use as a stainless bearing steel for 500 hours of operation at temperatures up to 900°F:

Alloy Desig.	C	Mn	Si	Cr	V	W	Mo	Co
WADC 65	1.10 1.15	0.15 max.	0.15 max.	13.5 14.5	2.5 3.0	2.0 2.5	3.75 4.25	5.0 5.5

For best performance, the steel should be heat-treated as follows: heat to 2200°F, oil quench, and refrigerate to minus 100°F. Rockwell "C" 66 is achieved (2% retained austenite) by tempering the steel for 1 + 1 + 2 hours at 1000°F with intermediate subzero cooling after the first and second draws. However, tempering twice for 2 hours at 1000°F also results in a useful structure. After 500 hours at 900°F the hardness drops to R_c 65 (hot hardness of R_c 57), and the structure contracts 13 in./in.

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After three cycles of a water vapor corrosion test, the surface of fully heat-treated WADD 66 is only slightly more pitted than that of annealed 430 stainless steel.

Crucible Steel Co. of America.
WADC TR 59-353.
DEVELOPMENT OF HIGH-TEMPERATURE IRON-BASE ALLOYS. A. Kasak, V. K. Chandhok, E. J. Dulis. AF 33(616)-5428, Phase A. October 1959. PB 161337. Order from OTS $2.00.

An investigation was conducted to develop steels that have a combination of very high strengths at ambient temperature and at temperatures approaching 1200°F. The results show that with proper balancing of the composition and optimum heat-treatment the objective of the program could be attained.

At temperatures up to 1100°F, the steels developed under this project had higher strengths than any other steel known today. The highest strengths were obtained on steels that contained 0.35 to 0.40% C, 7 to 9% Cr, 0.5 to 1.0% V, 5 to 7% Mo, 5 to 8% Co, up to 1% Ti, up to 1% Ta & Cb, up to 1.5% W, and up to 0.01% B. For example, one steel that contained 0.36% C, 7.72% Cr, 1.15% V, 5.72% Mo, 4.93% Co, and 0.009% B had a tensile strength of 310,000 psi at room temperature and 201,000 psi at 1100°F; the 100-hour creep-rupture strength at 1100°F was 98,000 psi. Also, excellent strengths were obtained at 1200°F.

In some cases, beneficial effects on strength and ductility were derived from using a novel heat-treating schedule.

Microscopic studies indicated that the fracture behavior and the creep-rupture strength were significantly influenced by the formation and coalescence of carbides at the grain boundaries.

WADC TR 59-263.
EFFECT OF HEAT TREATMENT ON THE METALLURGICAL AND MECHANICAL PROPERTIES OF 7AL-3MO TITANIUM ALLOY. Paul L. Hendricks. November 1959. PB 161363. Order from OTS $2.50

Metallurgical and mechanical properties of 7Al-3Mo titanium alloy were determined on a production size heat. The investigation included heat treatment, the determination of tensile properties, stress rupture, creep stability, compressive yield, modulus, and impact strength. The conditions under which testing was accomplished varied from room temperature to 1000°F.

Manufacturing Laboratories, Inc.
WADC TR 59-342.
INVESTIGATION OF THE STRENGTH AND DUCTILITY RELATIONSHIPS IN TITANIUM-ALUMINUM ALLOYS BETWEEN 6 AND 15% ALUMINUM FOR APPLICATION AT ELEVATED TEMPERATURES. B. S. Lement, G. T. Hahn, K. Kreder. AF 33(616)-5948. November 1959. PB 161424. Order from OTS $1.75.

An investigation of the strength and ductility relationships in a series of Ti-Al alloys varying from 6 to 15% aluminum were carried out. The activation energy for the embrittlement reaction in a Ti-8Al alloy was found to be about 34,000 cal/mol, which is compatible with present knowledge of diffusion of aluminum or interstitial elements. More complete evidence was obtained that striations represent sub-boundaries that form as a result of the beta-alpha transformation and at which micro-segregation occurs. Striations can be eliminated by cold working and recrystallising, or by quenching and recrystallizing. Rapid quenching results in a martensitic structure which exhibits high resistance to embrittlement on subsequent aging at 750°F in a Ti-8Al alloy.

In the embrittled condition, fracture takes place by transgranular cleavage. The primary cause of embrittlement appears to be high resistant to plastic flow caused by aluminum content and microsegregation. Recommendations are given for extending the useful range of aluminum content in Ti-Al alloys for commercial applications.

Armour Research Institute. WADC TR 59-500.
BERYLLIUM RESEARCH FOR DEVELOPMENT IN THE AREA OF CASTING. F. A. Crossley, A. G. Metcalfe, W. H. Graft. AF 33(616)-5911. February 1960. PB 161754. Order from OTS $2.50.

Various aspects of the casting of beryllium have been investigated to obtain information and understanding leading to the development of sound, fine-grained cast material. Areas investigated were: (1) X-ray determination of the direction of columnar growth in cast beryllium; (2) consumable arc casting of beryllium; (3) reported allotropy of beryllium by thermal analysis; (4) determination of grain-refining inoculants; and (5) application of vibration to cast beryllium for grain refinement.

Evidence in support of the reported allotropy of beryllium was obtained. The pertinent findings were the following: (1) the occurrence of a very pronounced thermal arrest 5 to 10°C below the solidification temperature of beryllium; (2) metallographic observations of transformation markings; and (3) lack of a preferred orientation for columnar grains in cast beryllium.

Consumable arc-melting experiments showed promise. Significant zones of equiaxed grains were obtained in an ingot prepared by a special technique. Vibration of cast, induction-melted beryllium was also promising. One ingot cast under vibration at 5g acceleration and 60 cps showed substantial zones of relatively fine, equiaxed crystals. Representative grain size measurements gave 616 grains/in.^{2} for this ingot compared to 316 grains/in.^{2} for a static (standard) casting. Interrupted rotation was demonstrated to be very effective in grain refining aluminum and is considered to be promising for beryllium.

A rust-colored tantalum nitride (identity uncertain), tungsten carbide, and possibly titanium diboride apparently nucleated beryllium solidification to produce grain refinement. Also, an alloying addition of 1 atomic pct of germanium produced grain refinement. The germanium alloy showed very high fluidity relative to other beryllium and beryllium alloy melts. The grain refinement was apparently due to some insoluble particles becoming effective nucleating agents in the Be-1 a/o Ge alloy.

Univ. of Michigan Research Institute
WADC TR 59-681.
FURTHER INVESTIGATIONS OF THE EFFECT OF PRIOR CREEP ON MECHANICAL PROPERTIES OF C110M TITANIUM WITH EMPHASIS ON THE BAUSCHINGER EFFECT. Jeremy V. Gluck, James W. Freeman. AF 33(616)-3368. March 1960. ASTIA Document No. AD 236917. PB 171946. Order from OTS $1.75.

A study of the effect of prior creep at 650° to 800°F on the short-time mechanical properties of C110M sheet showed property changes characteristic of the Bauschinger effect. After creep in tension, the tensile yield strength was increased and the compressive yield strength was decreased.

Creep-exposure at 700°F was found to cause Bauschinger effects almost as large as those reported in the literature for cold-stretching. The magnitude of the effect depended to some extent on the direction of the applied stress with respect to the rolling direction of the sheet, possibly due to a Bauschinger effect present in the original sheet as well as preferred orientation effects. The time of

creep-exposure also governed the extent of the effect. A study of variable strain paths indicated that there was no apparent difference between the effects of short-time plastic strain and creep strain in inducing a Bauschinger effect. However, for a given deformation, recovery effects caused a reduction in the effect as the creep time or temperature was increased. Periods of exposure at no load at 700°F were found to be effective in removing the Bauschinger effect.

The test material was also found to be subject to a structural instability during testing which accounted for increased strength and decreased ductility. The instability was a stress-activated breakdown of non-equilibrium beta to form a secondary alpha phase. Strain hardening during testing was at most a minor factor.

Battelle Memorial Institute. WADC TR 59-595. METALLURGICAL AND MECHANICAL CHARACTERISTICS OF HIGH-PURITY TITANIUM-BASE ALLOYS. R. W. Douglass, F. C. Holden, Horace R. Ogden, R. I. Jaffee. AF 33(616)-5462. March 1960. PB 161817. Order from OTS $3.00.

The relationships between mechanical properties, alloy composition, microstructure, and thermal history have been studied for high-purity titanium-base alloys. The alloy systems investigated include Ti-V, Ti-Cr, and Ti-W binary alloys, and Ti-Al-V, Ti-Al-Cr, Ti-O-V, Ti-O-Cr, and Ti-O-Mn ternary alloys. Mechanical-property data include tensile and flow properties, impact behavior, hardness, aging, and cooling-rate data. The effects of dispersions on the creep of Ti-W, Ti-Cu, and Ti-Si alloys were investigated. The metallurgical principles involved here and in previous work are discussed.

Alloyd Research Corporation. WADC TR 58-457, Part II. RESEARCH ON TECHNIQUES FOR THE PRODUCTION OF ULTRA-PURE BERYLLIUM. Malcolm Basche, Laurence M. Schetky. AF 33(616)-5300. March 1960. PB 161877. Order from OTS $1.50.

This report covers work done during the past year aimed at the production of high purity beryllium. The investigation concentrated on the following three approaches:

1. Zone purification in moderate and high vacuum
2. Distillation under high vacuum
3. Purification through halide reduction techniques.

As a result of this work, it has been determined that purification by zone melting in moderate or high vacuum is not feasible. Purification by vacuum distillation, zone purification in a high purity inert atmosphere, and by halide reduction techniques, do appear promising and further work is recommended.

Avco Corporation. WADC TR 59-695, Part II. BERYLLIUM JOINING WADC SPONSORED PROGRAM. E. M. Passmore. AF 33(616)-5913. April 1960. PB 161831. Order from OTS $2.75.

Joining of beryllium plates and rods by braze welding, fusion welding, and pressure welding was investigated, with the objective of developing improved methods for applications at both room and elevated temperatures. From the work described in this report, it can be concluded that braze welding with silver filler metal and pressure welding without filler offer the most promise as useful joining techniques for beryllium.

It was found that room temperature joint strengths of aluminum-12w/o silicon braze welds, made by the argon shielded, a-c tungsten arc process, are in the range of 20,000 to 25,000 psi. While the low melting point of the aluminum-silicon filler metal would seriously limit its high temperature use, silver braze welds are more promising in this respect. Room temperature joint strengths of about 21,000 psi are retained up to about 1000°F. Even at 1300°F, the strength is 11,000 psi, corresponding to a joint efficiency of 83 percent, compared to the tensile strength of the base material at that temperature.

Hot tearing during argon shielded, tungsten arc welding can be minimized by preheating the beryllium and using low arc travel speeds combined with low currents. Maintenance of base material aluminum content at a low level is advisable, since aluminum was found to promote hot tearing in the composition range present in commercially pure beryllium.

Oxidation of beryllium during welding can be effectively controlled by the use of a dry box evacuated and filled with pure argon and by the use of argon as shielding gas for the arc. While porosity occurred during welding some lots of beryllium, it was absent in others and no correlation with composition was found. Neither shielding gas differences, vacuum outgassing, nor preheating was found to affect porosity.

Joint efficiencies of 100 percent can be attained by pressure welding at temperatures less than 1650°F (900°C) and weld deformations less than 0.5 percent. Maximum strengths were about 40,000 psi for hot-pressed and 55,000 to 60,000 psi for extruded beryllium. Both welding temperature and surface roughness were found to affect joint strength. Room temperature tensile strengths increased rapidly with an increase in the pressure welding temperature up to about 1560°F (850°C), then decreased above 1830 due to grain growth.

Surfaces finished with 180 grit, silicon carbide paper produced weaker joints than did metallographically polished surfaces, especially at welding temperatures up to 1920°F (1050°C). Under optimum welding conditions, however, joint strength is limited apparently only by the strength of the base material.

Armour Research Foundation. WADC TR 59-705. EVALUATION AND ALLOY DEVELOPMENT OF HOT-WORK DIE STEELS FOR STRUCTURAL PURPOSES. Michael Schneider, John P. Sheehan. AF 33(616)-5633. May 1960. PB 171008. Order from OTS $2.25.

A program to investigate the tensile properties of three commercial hot-work die steels, Vasojet 1000, Potomac M, and Peerless 56 has been carried out. The results indicate that in sheet form these materials are essentially equivalent and develop the following nominal peak properties when air cooled from 1850°F and double tempered at 1000°F: 295,000 psi ultimate tensile strength; 233,000 psi yield strength at 0.2% offset; and 7% elongation in 2 inch gage length. Those materials which had been cross rolled showed little or no difference between longitudinal and transverse properties. Furthermore, no significant differences were obtained in longitudinal tensile properties by vacuum arc remelting of commercial bar stock.

Approximately 30 experimental alloy steels were melted, in which most of the commmon alloying elements were investigated to determine their effects on tensile properties. Of these, several developed yield strengths of 280,000-290,000 psi, ultimate tensile strengths of 340,000-360,000 psi and tensile elongations of 3-5% in 2 inch gage length on sheet material approximately 0.080 inch thick.

New York University. WADD TR 60-95. FURTHER STUDIES ON ACTIVE-EUTECTOID ALLOYS ON TITANIUM. R. F. Bunshah, D. Osterberg, E. Ence, H. Margolin. AF 33(616)-5655. May 1960. PB 161964. Order from OTS $2.00.

This report covers further work succeeding on previous investigations on active eutectoid titanium-copper alloys in which decomposition of the beta phase to alpha plus compound occurs rapidly. The current program is divided into two parts. Part I deals with the properties of active eutectoid alloys. The effect of stepwise additions of Al and/or Zr to binary Ti-5Cu, Ti-6Cu, Ti-6Ni, Ti-2Ni-2Cu and Ti-4Ni-2Cu alloys on the tensile strength at room and elevated temperatures, creep-rupture properties and thermal stability under stress were studied. Several alloys exhibited outstanding short time tensile strengths up to 1200°F much superior to the best competition. The creep rupture properties were correspondingly good. These alloys which exhibited highest strengths also showed instability. Stability could be restored partially by some sacrifice in strength.

This and the previous work has demonstrated that a new class of titanium alloys, i.e., of the alpha-compound type have been developed. They have potential utility in applications where short time elevated temperature strength properties are the prime consideration particularly for one cycle operation. Part II is concerned with the role played by Zr in strengthening these alloys. Constitutional diagram studies on the Ti-Zr and the 750°C section of the Ti-Cu-Zr systems were undertaken.

Armour Research Foundation. WADD TR 60-99. INVESTIGATION OF THE Ti-Al-Cb SYSTEM AS A SOURCE OF ALLOYS FOR USE AT 1200°-1800°F. Joseph B. McAndrew, Charles R. Simcoe. AF 33(616)-6125. May 1960.

The work reported was an experimental study of titanium base alloys containing major additions of both aluminum and columbium, with attention primarily directed toward the properties of such alloys in the temperature range of 1200°F to 1800°F.

Alloys were prepared with aluminum content of 5 to 17.5%, plus 15 to 30% columbium, and were investigated with respect to forgeability, density, oxidation resistance at 1000°C (1832°F), hardness, short-time tensile properties at room temperature and elevated temperatures, heat treatment response, and microstructure. Several alloys were also subjected to bend, impact and stress-rupture tests. The refinement, precision, or extent of testing was not such as would produce data appropriate for design purposes, since the program was intended to yield findings suitable for research uses.

A considerable range of properties was exhibited by alloys of various compositions. Several alloys of high aluminum content had exceptionally high strength-to-density ratios in short-time tensile tests at temperatures up to 1600°F (871°C). Forgeability, good oxidation resistance, and low density were attractive features shared by all of the alloys. Many of them showed considerable response to heat treatment.

Analysis of the data indicates that the Ti-Al-Cb alloys offer a very promising field in which to conduct further research.

Armour Research Foundation. WADD TR 60-120. STUDY OF FATIGUE PROPERTIES OF ULTRA-HIGH STRENGTH STEEL. Harvey B. Nudelman, John P. Sheehan. AF 33(616)-6290. June 1960. PB 171056. Order from OTS $1.75.

A temper-resistant, high-strength steel was investigated with reference to fatigue strength. This alloy was prepared by two different melting techniques. The first method consisted of induction melting in air using standard deoxidation techniques. The second method involved the application of a special deoxidation practice to induction melting; silicon-free steel was deoxidized with carbon and aluminum to minimize the presence of silicate inclusions. These alloys were tested in fatigue using the Prot accelerated method. The results of the samples melted with standard practice indicated that fatigue strength is improved slightly by tempering at elevated temperature. The application of a special melting process showed that a very significant increase in the Prot fracture stress to ultimate tensile strength ratio (E_p/UTS) can be obtained by eliminating the presence of silicon and deoxidizing with carbon and aluminum.

Battelle Memorial Institute. WADD TR 60-258. A STUDY OF THE TITANIUM-LIQUID OXYGEN PYROPHORIC REACTION. J. D. Jackson, P. D. Miller, W. K. Boyd, F. W. Fink. AF 33(616)-6345. June 1960. PB 171017. Order from OTS $1.50.

A review of the literature indicates that titanium is impact sensitive under liquid oxygen below the acceptable limit for other metals.

An experimental program was begun to determine the mechanism of the titanium-LOX reaction. Several factors were investigated singly in a controlled manner using unalloyed titanium (75A) and an alloy (6Al-4V) that were carefully cleaned. The factors were:

1. Exposure of a fresh surface by fracture and tearing
2. Deformation by impact using steel balls
3. Impact of smooth specially cleaned flat surfaces
4. LOX pressure and velocity
5. Galling

The results from this program indicate that no one of the above, per se, is a primary cause of this reaction. A proposed mechanism is that heat generated by impact produces gaseous oxygen, which is compressed at local sites. A fresh surface exposed by the impact reacts with the high-pressure gaseous oxygen. Propagation is dependent on the amount of heat generated and the rate of heat loss from the affected area.

Lockheed Missiles and Space Division, California Division of Lockheed Aircraft Corporation. WADD TR 60-116. BERYLLIUM CRACK PROPAGATION AND EFFECTS OF SURFACE CONDITION. C. O. Matthews, M. I. Jacobson, W. E. Jahaman, W. V. Ward. AF 33(616)-5978. July 1960. PB 171088. Order from OTS $3.00.

The mechanical properties of beryllium sheet with various surface finishes were investigated. The best properties were obtained on sheet which had been etched to remove surface defects caused by maching.

Ductility was low in all cases, being limited by the presence of notches and by preferred orientation in the sheet. Ductility was increased by heating to 400°F to 600°F.

The fatigue endurance limit was as high as the static tensile strength, and was improved by etching. Impact tests were found to be most suitable for distinguishing between various surfaces.

Theoretical analyses were made of the effect of including residual stress in the Griffith crack theory, and of the dynamic stress at the leading edge of a crack in a uniaxially stressed plate.

American Brake Shoe Company. WADC TR 59-63, Part II.

DEVELOPMENT OF LOW ALLOY STEEL COMPOSITIONS SUITABLE FOR HIGH STRENGTH STEEL CASTING. Hugo R. Larson, Ronald C. Campbell, Herbert W. Lloyd. AF 33(616)-6485. July 1960. PB 171065. Order from OTS $2.00.

Cast high strength steels were investigated in an effort to facilitate the production of high integrity, high strength castings for aircraft and missile applications.

A study was made of heat treatments and compositions for optimum properties in heavy sections. Acceptable properties were developed in the modified 8735 and 4335 alloys in two and three inch sections, but the problem of poor ductility in heavy sections of 5% chromium steel was not solved.

Spectrographic analyses and sulfur additions to high purity heats failed to reveal the factors responsible for improved properties in high purity heats. Fatigue properties of normal and high purity and vacuum melted low alloy steels were rather disappointing. An endurance limit of approximately 55,000-60,000 psi was observed.

Excellent properties were developed at room temperature and 1000°F in the 5% Cr air hardening steel with a 3% tungsten addition and proper heat treatment. The effect of soundness and feeding distance on the properties of the 5% Cr air hardening steel was also studied.

Finally, analysis of production heats indicated good reproducibility of properties in cast high strength steels. High purity production heats were shown to be somewhat superior to normal purity heats.

WADC TN 59-326.

THE STATUS OF RESEARCH AND DEVELOPMENT FOR HIGH STRENGTH AIRCRAFT STEELS. E. M. Kennedy, Jr. July 1960. ASTIA Document No. AD 226520. PB 152299. Order from LC, Mi $3.00, Ph $6.30.

New aircraft and missile developments have required the development and use of structural steels having optimum strength and ductility over a wide range of temperatures.

In this survey an attempt is made to point up to some of the significant areas of investigation in which efforts are being expended toward developing and improving steels having tensile strengths in excess of 260,000 psi. In order to complete the survey of the field of interest, the sources known to have research and development interest in this area were contacted either by personal visitation or through official correspondence.

The results of the contacts made were gratifying and the many investigations and studies being carried on are commented upon in the text of this report.

New York University. WADD TR 60-316.

BINARY AND TERNARY DIAGRAMS. Elmars Ence, Paul A. Farrar, Harold Margolin. AF 33(616)-5704. August 1960. PB 171154. Order from OTS $2.25.

I. Ti-Al-Cr System -- The Ti-rich corner of the Ti-Al-Cr system has been reinvestigated from 600-1400°C (up to 40 wt percent Al and 32 wt percent Cr). Partial isothermal sections have been constructed at 1400, 1200, 1100, 1000, 800 and 600°C. The phases encountered in the portion of the system investigated are Ti_3Al, Ti_2Al, TiAl and $TiCr_2$. A miscibility gap in the field is produced by the interaction of the Ti_2Al and the $TiCr_2$ fields.

II. Ti-Al-V System -- The Ti-Al-V system has been reinvestigated from 50 to 100 wt percent titanium and from 500°C to 1400°C, using X-ray diffraction and metallographic techniques. Isothermal sections were delineated at 500, 600, 700, 800, 900, 1000, 1100, 1200, 1300 and 1400°C. Vertical sections at 2, 4, 6, 10, and 16 wt percent aluminum; 2, 4, 6, 10, and 16 wt percent vanadium and 90 wt percent Ti were constructed. The phases encountered in the portion of the system reinvestigated Ti_3Al, Ti_2Al and TiAl.

The Brush Beryllium Company. WADC TR 58-478, Part II.

DEVELOPMENT OF WROUGHT BERYLLIUM ALLOYS OF IMPROVED PROPERTIES. John G. Klein, Leslie M. Perelman, Wallace W. Beaver. DO 33(616)-57-19. September 1960. PB 171389. Order from OTS $2.75.

Mechanical and physical properties are reported for extruded and/or rolled products fabricated from beryllium-rich alloys of silver, tin, cadmium, zinc, nickel, and copper, as well as beryllium fabricated from subsievesize powder and powder of higher than normal beryllium oxide content.

WADD TR 60-425.

MECHANICAL PROPERTIES OF BERYLLIUM. A. E. Riesen, R. T. Ault. September 1960. PB 171411. Order from OTS $1.00.

The test procedures and results of a mechanical properties' determination program which included tensile, torsion, pin shear, creep rupture, and fatigue data on two lots of beryllium are presented. One lot of material was hot pressed with a BeO content of 1.45%; the other lot was hot pressed and hot extruded and contained 1.55% BeO.

Emphasis was placed on the fatigue and creep rupture data. Fatigue tests were conducted at room and elevated temperatures with stress ratios of A and 0.67. Both lots of material showed surprising strength under fatigue loading conditions whereas beryllium's inherent brittleness manifested itself under static loading conditions by the brittle fractures.

The hot pressed-hot extruded material was uniformly stronger and therefore more desirable for design purposes than the material that was only hot pressed.

New York University. WADD TR 60-443.

PARTITIONING AND MICRODISTRIBUTION OF INTERSTITIAL ALLOYING IN TITANIUM. Irving B. Cadoff, Joseph Winter. AF 33(616)-5506. September 1960. PB 171412. Order from OTS $1.25.

The partitioning or segregation of excess interstitial solutes at the grain boundaries in alpha phase and beta phase, titanium alloys was investigated. Internal friction spectra of dilute alloys of titanium with oxygen, nitrogen and carbon were obtained. From these spectra the activation energy for grain boundary stress relaxation, the net interaction energy of a solute atom with a grain boundary, and the excess solute concentration were calculated.

As in previous investigations of this type the partitioning of excess solute was found to obey a relation of the form:

$$C_s = C_o \exp^{V} . s/kT$$

The interaction energies were found to lie in the range 0.15 to 0.40 eV for binary interstitial alloys. The expected behavior of increase in activation energy, decrease in peak height and decrease in peak temperature with increasing solute concentration was observed. In the case of carbon and nitrogen alloys the activation energy increase exhibited a saturation effect. While this was not observed in oxygen alloys, the nature of the curve indicated approach to

saturation. In the range of saturation, the carbon alloys developed a second peak, the composition at which the peak was fully evident being close to the solubility limit of carbon in alpha titanium. From the nature of the curves obtained it may be inferred that the double peak may be associated with the formation of the delta phase, which causes local depletion of the carbon atoms segregated at the grain boundary.

Tensile measurements indicated that the increase of excess solute at grain boundaries and dislocations brought about by either increasing the bulk concentration or decreasing the equilibrium annealing temperature resulted in an increase in yield strength. The effect of excess solute on yield stress was found to be parallel to its effect on the internal friction spectra.

WADD TR 60-542.
STUDY OF RATE CONTROLLING PROCESS FOR COMPRESSIVE DEFORMATION OF HIGH PURITY ALUMINUM. A. E. Riesen. September 1960. PB 171488. Order from OTS $0.75.

A unique testing technique for determining the rate-controlling process for compressive deformation of high purity aluminum over the temperature range of 297°K to 358°K is discussed, and an attempt is made to correlate the activation energy for compression with some recognized rate-controlling process for tensile deformation of aluminum. It is shown that the compressive activation energy obtained, 5,400 cal/mol., cannot be related to any known tensile deformation process.

The results of this study indicate a need for further experimental investigations to define the compression activation energy spectrum for aluminum over a broad temperature range.

Massachusetts Institute of Technology.
WADD TR 60-340.
VOID FORMATION AND GRAIN BOUNDARY SLIDING IN ALUMINUM-MAGNESIUM SOLID SOLUTION ALLOYS. Arthur W. Mullendore, Nicholas J. Grant. AF 33(616)-5926. September 1960. PB 171399. Order from OTS $1.00.

Void formation and growth during creep were observed in aluminum -- 1.92 percent magnesium and aluminum -- 5.10 percent magnesium as a function of temperature, stress, and time. The origin of voids is related to the development of an irregular (serrated) grain boundary and to grain boundary sliding. Grain boundary sliding is shown to result largely from crystal slip crossing the grain boundary.

Nuclear Metals, Inc., Massachusetts Institute of Technology, Westinghouse Research Laboratories. WADD TR 60-132.
REFRACTORY METAL CONSTITUTION DIAGRAMS. A. R. Kaufmann, E. J. Rapperport, M. F. Smith, J. Wulff, J. Brophy, N. J. Grant, B. C. Giessen, A. Taylor, N. Doyle. AF 33(616)-6023. October 1960. PB 171391. Order from OTS $3.00.

Nine binary constitution diagrams and one ternary constitution diagram of some of the refractory metals are presented. The binary diagrams include Hf-Mo, Hf-Re, Hf-W, Nb-Re, Os-Ta, Os-W, Re-Ta, Ru-Ta, Ru-W, and the ternary is Re-Ta-W.

Care was taken to obtain reliable diagrams. In particular, the purity of the constituents (99.9 percent plus) was protected at all times, and the temperatures were measured to an accuracy within 20°C.

Polytechnic Institute of Brooklyn. WADD TN 59-424, Part II.
PRELIMINARY ANALYSIS OF THE CAPABILITIES OF A COMPOSITE SLAB FOR AN ADVANCED HEAT-SINK DESIGN. Paul A. Libby. AF 33(616)-5944. January 1961. ASTIA Document No. AD 253542. PB 171824. Order from OTS $1.75.

Presented here are the results of a numerical analysis of the capability of a composite slab of beryllium oxide (BeO) and beryllium (Be) to absorb the heating associated with the reentry of a high performance ballistic missile. The trajectory considered corresponds to a ballistic factor (W/C_DA) equal to 2,000 psf, to a reentry velocity of 20,000 fps, and to a reentry angle of 20°. The thermal properties of the metals were considered temperature dependent; surface radiation was included. An implicit system of numerical integration was applied.

The numerical results indicate that the maximum permissible heat transfer rates for the trajectory are obtained with a relatively thin slab of beryllium oxide. The addition of beryllium to this slab may be required for structural and thermal shock considerations but does not greatly improve the heat-sink capabilities.

The permissible values of the heat transfer parameter are applied to a slender cone with a 20° half angle and with a spherical cap of 0.25 ft nose radius. The laminar and turbulent heat transfer data for such a cone were available. It is shown that for laminar flow no heat transfer reduction is required on the cone while for turbulent flow a reduction to 1/2 is required. The possibility of utilizing a mass transfer system in order to reduce the heat transfer on the spherical cap and on the cone in the case of turbulent flow is briefly discussed.

High Temperature Metals

TR 5435.
COMPARISON OF HIGH TEMPERATURE PROPERTIES OF TURBINE WHEEL ALLOYS OF LOW ALLOY CONTENT. Isaac Perlmutter. March 1946.

TR 5649.
STRESS CORROSION OF HEAT RESISTANT ALLOYS AT ELEVATED TEMPERATURES. Isaac Perlmutter. November 1947.

TR 5712.
INVESTIGATION OF SHEET MATERIALS FOR APPLICATION AT HIGH TEMPERATURES. Isaac Perlmutter and W. H. Rector. July 1948.

TR 5716 (R).
SERVICE FAILURE OF TURBINE BUCKETS. Isaac Perlmutter and H. K. Adenstedt. July 1948.

Allegheny Ludlum Steel Corporation. TR 5731.
THE DEVELOPMENT OF SHEET MATERIALS FOR HIGH TEMPERATURE APPLICATIONS. Gunther Mohling and Joseph B. Meierdirks. W33-038-ac-16519. November 1948.

Crucible Steel Company of America. TR 5730.
RESEARCH ON HIGH TEMPERATURE SHEET MATERIAL. William Murphy and L. L. Ferrall. W33-038-ac-16677. November 1948.

Cornell Aeronautical Laboratory, Inc. TR 5929.
UTILIZATION OF LOW ALLOY MATERIALS FOR HIGH TEMPERATURE SERVICE APPLICATIONS. James Miller, L. W. Smith, and Phillip K. Porter. W33-038-ac-21094. June 1949.

TR 5893.
INVESTIGATION OF THE STRESS RUPTURE PROPERTIES AT 1500°F OF A NUMBER OF HIGH TEMPERATURE ALLOYS. Melvin E. Fields, Capt., and William H. Rector. July 1949.

Syracuse University. TR 5930.
DYNAMIC CREEP AND RUPTURE PROPERTIES OF TEMPERATURE RESISTANT MATERIALS UNDER TENSILE FATIGUE STRESS. B. J. Lazan. W33-038-ac-15941(17507). February 1950.

TR 5936, Part 1.
ELEVATED TEMPERATURE FATIGUE TESTING OF TURBINE BUCKETS, PART 1 -- CALCULATIONS OF NATURAL FREQUENCIES AND STRESSES, AND PROPOSED TESTING METHODS. Dr. A. Herzog. May 1950.

Battelle Memorial Institute. TR 5949.
U-36 ALLOY DETERMINATION OF DESIGN DATA. Ward F. Simmons. AF 33(038)-956. May 1950

TR 6188.
STRESS RUPTURE TESTS ON SHEET ALLOYS FOR HIGH TEMPERATURE APPLICATIONS. Isaac Perlmutter. July 1950.

Allegheny Ludlum Steel Corporation. TR 6219.
FORGING BUCKET ALLOYS AND CAST BUCKET ALLOYS. Gunther H. Mohling. AF 33(038)-2040. October 1950.

Allegheny Ludlum Steel Corporation. TR 6615.
THE DEVELOPMENT OF FORGING AND CASTING ALLOYS FOR TURBINE BUCKETS. Ralph P. DeVries, Jr., and Gunther Mohling, Ph.D. AF 33(038)-11669. August 1951.

This project was undertaken with the object of further investigating the forging and cast turbine bucket alloys developed under the preceding contract AF 33(038)-2040. The effects of various compositional variations were studied on the forging alloy V-912, 34.9 cobalt, 6.5 tungsten, 1.6 columbium, 20.0 chromium, 20.0 nickel, 3.0 molybdenum, 0.3 silicon, 1.0 manganese, 0.3 carbon, balance iron, and the cast alloy W-834, 29.5 cobalt, 8.0 tungsten, 0.5 carbon, 25.0 chromium, 20.0 nickel, 3.0 molybdenum, 0.5 silicon, 1.0 manganese, balance iron.

It was found that the percentage of elements in these alloys was very close to the optimum ratios to produce sufficient stress rupture and room temperature strengths required of turbine bucket alloys.

TR 6640.
SILICA-KAOLIN PRECOAT FOR INVESTMENT CASTING. William F. Davenport, Captain, USAF, Adolph Strott. October 1951.

An investigation was conducted to study the feasibility of using kaolin as the source for alumina in the recently discovered alumina-silica precoat, or mold facing material.

Precoats were prepared using kaolin as the alumina carrier in both molecular and percentile variations. These were evaluated by casting a heat resistant type alloy, a stainless steel, and an alloy steel into the investment molds faced with each particular precoat composition. Kaolin was found to be satisfactory as the alumina source although some shifting of previously determined composition limits was noted. No difference in precoat properties was found to exist between molecular and percentile variations other than that which was accounted for due to percentile differences.

U. S. Bureau of Mines. TR 52-54.
MALLEABLE CHROMIUM AND ITS ALLOYS. Earl T. Hayes. AF 33(038)-50-1084-E. March 1952.

Syracuse University. TR 52-243.
DAMPING, ELASTICITY, AND FATIGUE PROPERTIES OF TEMPERATURE-RESISTANT MATERIALS. B. J. Lazan and L. J. Demer. AF 33(038)-18903. November 1952.

The damping, elasticity and fatigue properties of several temperature-resistant materials were investigated in rotating cantilever-beam testing equipment. The room and elevated temperature tests were designed to reveal changes in damping energy and dynamic modulus of elasticity during constant cyclic stress fatigue tests at engineering stress levels. Usual S-N fatigue curves are presented in addition to a series of new diagrams designed to show the effects of both stress magnitude and stress history on the damping and elasticity properties. Two methods for comparing the damping energies of a group of materials are offered and the merits of each discussed. Diagrams are also presented to facilitate comparison of the elasticity properties among materials tested at a given temperature.

Syracuse University. TR 52-227.
PROPERTIES OF TEMPERATURE-RESISTANT MATERIALS UNDER TENSILE AND COMPRESSIVE FATIGUE STRESS. B. J. Lazan, E. Westberg. AF 33(038)-18903. November 1952.

Newly developed gripe and machine improvements are described for fatigue loading under direct stress (tension-compression) ratios of alternating to mean stress from zero to infinity. Data are presented to indicate the uniformity of stress distribution possible with these gripe and accuracy of the average stress. Dynamic creep, rupture and ductility data are reported on N-155, S-590 and Vitallium at 1350° and 1500°F under direct stress combinations from static to reversed loading. Stress range diagrams (alternating stress versus mean stress) are presented to indicate the stress combination which will produce rupture and various degrees of creep in 5 to 1500 hours. Percent elongation data are analyzed in terms of alternating-to-mean stress ratio and stress magnitude, and it is shown that both are significant variables. Elongation up to the start of third stage of creep is also analyzed in terms of stress ratio and stress magnitude and only stress magnitude was found to be significant. The implications of these findings are discussed.

Syracuse University. TR 52-253.
METALLOGRAPHIC STUDIES ON N-155 SPECIMENS EXPOSED TO STATIC AND DYNAMIC STRESS AT ELEVATED TEMPERATURES. F. R. Morral and B. J. Lazan. AF 33(038)-18903. December 1952.

Work reported herein represents a continuation of earlier metallographic studies on heat-resistant alloys. The specimens studied in this program were N-155 previously tested under fatigue stress having various stress ratios. An attempt was made to determine the effect of each of the four test variables; time, temperature, static stress, alternating stress. Visual and macroscopic classifications of the fractures were attempted and microscopic structure and microhardness data are reported.

University of Minnesota. TR 53-70.
DAMPING, ELASTICITY, AND FATIGUE PROPERTIES OF UNNOTCHED AND NOTCHED N-155 AT ROOM TEMPERATURE AND ELEVATED TEMPERATURES. L. J. Demer, B. J. Lazan. AF 33(038)-18903. February 1953. PB 111618. Order from OTS $2.00.

Data are presented on the damping, elasticity, and fatigue properties of N-155 under rotating bending stress. Unnotched and notched specimens at room temperature, 1350°, and 1500°F are included in this study. Properties at the three temperatures are compared on two bases, equal stress and equal stress ratio. The change in vertical deflection and run-out for notched apecimens are associated with the first evidence of the formation of a fatigue crack. Curves for these first-evidence-of-crack points plotted against stress are compared with the usual S-N fracture diagrams. Effective stress concentration factors are determined from both the fracture and first-evidence-of-crack data and the latter values are found to be nearly independent of the number of fatigue cycles. Data are presented for both unnotched and notched specimens on the dynamic proportional limit and its relationship to the fatigue strength. Equations are developed for interpreting rotating cantilever beam data so that the effective length of specimen fillets, specific damping energy, and dynamic modulus of elasticity may be calculated.

University of Minnesota. TR 52-226, Part 2.
INVESTIGATION OF AXIAL LOADING FATIGUE PROPERTIES OF HEAT RESISTANT ALLOY N-155, PART 2. AN EXPLORATORY INVESTIGATION OF THE EFFECT OF TEMPERATURE, TIME, AND STRESS ON FRACTURE CHARACTERISTICS AND METALLOGRAPHIC STRUCTURE OF N-155 AND HARDNESS OF N-155 AND S-816. Fred W. DeMoney. AF 33(038)-18903. February 1953.

An exploratory study of effect of the test variables on the macroscopic appearance of the fracture surface was continued. Fracture profiles were investigated microscopically and an attempt made to quantitatively analyze the nature of the fracture profile. From this analysis is shown that the tendency for an intercrystalline fracture to occur decreases with increasing stress and stress ratio. Investigation of the metallographic structure and hardness of unstressed N-155 verifies prior work concerning the precipitation hardening characteristics of this material. Rockwell "B" hardness tests were conducted on both the surface and longitudinal sections of the stressed specimens in the longitudinal variable stress regions. Tukon Vickers diamond pyramid hardness tests were also conducted on transverse sections of rotating beam fatigue specimens in the transverse variable stress regions. The study of effect of stress variables, while not completely investigated, indicate an acceleration of the precipitation hardening phenomenon as revealed by hardness tests. The acceleration of the precipitation hardening phenomenon is not observed in the metallographic structure of the material.

TR 5936, Part 2.
ELEVATED TEMPERATURE FATIGUE TESTING OF TURBINE BUCKETS, Part 2. FATIGUE TESTS OF TURBINE BUCKETS UNDER STATIC AXIAL AND SUPERIMPOSED VIBRATIONAL BENDING LOADS. Dr. Albrecht Herzog, Ing. March 1953.

The fatigue investigation of turbine blades in a special device, permitting the application of static axial and superimposed vibrational bending loads by means of an electromagnet, is discussed. The excitation of the vibration of the axially loaded bucket was adjusted to the fundamental frequency of this system. The most important factors causing damping effects and energy losses during vibration are considered. Special attention is given to the measuring methods for obtaining stress and deflection values of the vibrating bucket under dynamic load. The tests were conducted at room and at elevated temperatures. The results are discussed and compared with fatigue tests under similar conditions using simple specimens and standard testing devices.

University of Minnesota. TR 52-226, Part 1.
INVESTIGATION OF AXIAL LOADING FATIGUE PROPERTIES OF HEAT RESISTANT ALLOY N-155. B. J. Lazan, F. DeMoney. AF 33(038)-20840. March 1953.

Dynamic creep and rupture data are presented on N-155 at room temperature and 1000°F, correlated to some extent with prior work. Temperature increases were observed in certain temperature and stress ranges immediately after the application of alternating stress to a test specimen. These are discussed in terms of the internal damping capacity of the material and the possibility of utilizing these observed temperature increases as a qualitative indication of damping is suggested. A vibration analysis of the Minnesota direct stress fatigue machine is presented and used to correlate calibration data procured by three independent methods.

U. S. Bureau of Mines. TR 53-272.
CHROMIUM-BASE ALLOYS. Richard G. Nelson, Harry G. Anderson. AF 33(038)-50-1084 E. August 1953.

Binary alloys of 60 Cr-40 Fe with oxygen analyses of 0.01 to 0.03 show elongations of 15 and 17% in the hot worked and heat treated conditions. Similar alloys with 0.2% oxygen show up to 14% elongation in the heat treated conditions with little or no elongation in the hot worked state. Alloys with 70% chromium show evidence of ductility. Addition of deoxidizers lowers the ductility by causing intergranular failure. Creep rupture values are given for 60 and 70% chromium balance iron alloys. High chromium alloys can be worked at relatively low temperatures.

University of Michigan. TR 53-277, Part 1.
HIGH-TEMPERATURE PROPERTIES OF FOUR LOW-ALLOY STEELS FOR JET-ENGINE TURBINE WHEELS. Arthur Zonder, Adron I. Rush, James W. Freeman. AF 33(038)-13496. November 1953. PB 135130. Order from LC, Mi $4.50, Ph $12.30.

Properties at 1000°, 1100° and 1200°F are reported for jet-engine turbine wheels made from four low-alloy hardenable steels. The steels were SAE 4340, 1.25 Cr-Mo-Si-V ("17-22A"S), 3 Cr-Mo-W-V (H-40), and 12 Cr-Mo-W-V (C-422). The wheels were contour forged from commercial heats. Wheels of each alloy were heat

treated by normalizing, oil quenching, and by an interrupted quench. All were tempered to an aim hardness of 280 to 320 Brinell.

There was very little difference in strength between the alloys for the wheels with the best heat treatment at short time periods. The 4340 steel fell off rapidly in strength with time. The "17-22A"S steel had the highest strength at 1000°F, but fell off with time at 1100° and 1200°F. The H-40 and C-422 steels maintained their strength better with time and temperature. Heat treatments which produced intermediate transformation products in the structures of the steels resulted in highest strength, except for C-422 alloy. The latter 12 Cr alloy could not be heat treated to such structures. The treatments producing the best properties were normalizing for 4340 steel, oil quenching for "17-22A"S steel, oil quenching for H-40, and oil quenching for C-422 alloy.

Allegheny Ludlum Steel Corporation. TR 53-274. CASTING AND FORGING TURBINE BUCKET ALLOYS. R. K. Pitler, and W. W. Dyrkacz AF 18(600)-149. December 1953. PB 134470. Order from LC, Mi $3.90, Ph $10.80.

Wrought nickel-base alloys and chromium-manganese austenitic steels, as well as cast iron-nickel-cobalt-chromium alloys were investigated with an aim toward lowering the strategic alloy content of materials for gas turbine service at temperatures of 1200° to 1600°F. The best nickel-base alloys contained around 10% cobalt and over 5% molybdenum in addition to the age-hardening agents, titanium and aluminum. The austenitic steels, containing 17% manganese and 12% chromium showed promise for service at 1200°F. The addition of boron contributed materially to the high-temperature properties of the cast alloys.

Battelle Memorial Institute. TR 53-451. THE DEVELOPMENT OF A FORGEABLE HIGH-STRENGTH, HIGH-TEMPERATURE, CHROMIUM-RICH, CHROMIUM-IRON ALLOY. D. P. Moon, I. A. Blank, A. M. Hall. AF 33(616)-222. January 1954. PB 121112. Order from OTS $0.75.

The development of a forgeable high-strength, high-temperature, chromium-rich, chromium-iron alloy by Battelle Memorial Institute is described. Experimental alloys were produced by induction melting 4- and 12-pound charges of commercially available melting stock, casting into rammed zirconite molds, and forging to 5/8-inch-square bars for testing. Stress-rupture properties of alloys containing approximately 70 Cr, 30 Fe, 9 Mo, 2 to 3 Ti, and up to 1/2 Al (parts by weight) were excellent, but room-temperature ductility was poor. Some ductility was obtained after heat treatment in warm-rolled strip of the compositions 55 Cr, 45 Fe, 1 Ti and 60 Cr, 40 Fe, and 1 Ti. It is concluded that the 70Cr-30Fe-base alloy containing about 9Mo, 2 to 3 Ti, and up to 1/2Al shows promise as a material of construction for turbine buckets in turbojet aircraft.

University of California. TR 54-40. THE EFFECT OF GRAIN SIZE ON THE FATIGUE AND CREEP PROPERTIES OF STAINLESS STEEL AND INCONEL AT ELEVATED TEMPERATURES. A. T. Robinson, J. E. Dorn. AF 33(038)-22608. April 1954.

This investigation was undertaken to evaluate the effect of grain size, as produced by annealing, on the high temperature creep and fatigue properties of inconel and an 18-8 stainless steel. In order to isolate the effects of grain size and annealing from other metallurgical effects, an attempt was made to select materials which were not particularly prone to extraneous changes such as precipitation or spheroidisation of intermediate phases. But, in order to estimate the practical utility of practicing annealing for grain size control of high temperature alloys, the two representative alloys inconel and type 304 extra low carbon 18-8 stainless steel were chosen. Unfortunately both of these materials were found to exhibit structural changes during annealing which might have affected their creep and fatigue properties. The results indicate that to clearly delineate the effect of grain size on the properties of metals it will be necessary to use metals in which all auxiliary microstructural changes are absent.

University of California. TR 53-336, Part 1. THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS. PART 1 -- INTERMITTENT STRESSING. Lawrence A. Shepard, C. Dean Starr, Carl D. Wiseman, John E. Dorn. AF 33(038)-11502. May 1954. ASTIA Document No. AD 23715. PB 135149. Order fro LC, Mi $6.00, Ph $18.30.

Constant and intermittent load creep tests were performed on clad aluminum alloys 75S-T6 and 24S-T3 at 300°, 450° and 600°F, and on cold rolled titanium at 400°F. Constant load creep tests were also conducted on annealed titanium Ti-75-A at 400°F.

Two groups of intermittent load tests were performed, each having a full cycle of two hours. One group was maintained under load for one hour out of every two, and the second for one and one-half hours out of every two.

An empirical method for estimating and correlating creep curves for various stresses at constant temperature has been determined and shown to be applicable to both static and intermittent creep of a number of metals and alloys.

On the basis of total test time, constant load creep was more rapid than creep under either of the two load cycles.

The two alternate types of interrupted loading cycles used in the present investigation gave practically the same creep strain versus net time under load curves. Undoubtedly other types of on and off loading periods would have revealed differences in the creep behavior.

University of California. WADC TR 53-336, Part 2. THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS. Lawrence A. Shepard, C. Dean Starr, Carl D. Wiseman, John E. Dorn. AF 33(038)-11502. July 1954. PB 131016. Order from OTS $1.00.

Intermittent temperature, constant load creep tests were performed on clad aluminum alloys 75S-T6 and 24S-T3 at 450°F, and the test data are reported herein.

Aluminum alloy 75S-T6 creep specimens were held at 450°F for 1/2 hours out of every two hour temperature cycle. During the remaining half hour of the two hour temperature cycle the specimens were cooled to room temperature, held at room temperature, and then reheated to 450°F prior to the start of the next 2 hour cycle. The 24S-T3 aluminum alloy specimens were held at 450°F for 1 hour out of every two. During the remaining hour of the two hour temperature cycle the specimens were cooled to room temperature, held at room temperature and then reheated to 450°F prior to the start of the next two hour cycle. All specimens were maintained under constant load throughout the test.

Comparison of cyclic temperature creep data obtained in this investigation with isothermal data under

the same conditions of stress reveal that approximately equivalent plastic strains are achieved when the comparison is made on the basis of net time at test temperature.

A survey of existing literature on metallic creep under cyclic temperature conditions is also included.

University of California. WADC TR 53-336. Part 3. THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS. Lawrence A. Shepard, C. Dean Starr, Carl D. Wiseman, John E. Dorn. AF 33(038)-11502. July 1954. PB 130 204. Order from LC, Mi $2.70, Ph $4.80.

Tests on the combined effect of intermittent loading and intermittent heating on the creep strength of aluminum alloy 75S-T6 at 450°F were performed.

In all constant and intermittent tests the specimens were initially heated to test temperature in a three hour period. Two types of combined intermittent loading and heating cycles were used after the initial heating and loading which incorporated 1-1/2 hour periods of stressing and heating within a two hour interval. In one type, the "in phase" condition, the time of load application and the acquisition of test temperature were simultaneous. In the other type, the "out of phase" condition, cooling preceded unloading by 45 minutes.

Within the limits of scatter, simultaneous cycling of the load and temperature in phase produced little effect other than to extend the creep test by the time periods at room temperature and no load. In the case of out of phase cycling of the load and temperature, an acceleration of the creep rate was obtained as compared with isothermal, constant load creep tests assuming that little or no creep should occur at no load, or when the specimen is below test temperature.

University of Michigan. WADC TR 54-206. THERMAL-SHOCK INVESTIGATION. T. A. Hunter, L. L. Thomas, A. R. Bobrowsky. AF 33(038)-21254. September 1954. PB 121109. Order from OTS $2.75.

A program of investigation has been undertaken to evaluate the resistance of various materials to thermal shocking. A preliminary analysis of thermal-shock damage has been carried out on a theoretical basis. The results of this theoretical work indicate that the scope of the problem is so wide that purely analytical methods must be supplemented by experimental data.

An experimental program has therefore been set up to test fourteen materials for their relative resistance to severe repeated thermal shock from the temperature range of 1600° to 2000°F. A suitable apparatus has been constructed and a standard specimen shape has been devised which give reasonable reproducibility of results. Excursions into the subjects of previous specimen history, mechanical fatigue, and thermal wiggling have also been made.

Battelle Memorial Institute. WADC TR 53-451, Part 2. THE DEVELOPMENT OF A FORGEABLE HIGH-STRENGTH, HIGH-TEMPERATURE, CHROMIUM-RICH, CHROMIUM-IRON ALLOY. D. P. Moon, H. A. Blank, A. M. Hall. AF 33(616)-222. October 1954. PB 121111. Order from OTS $0.75.

The second year's work on the development of a forgeable high-strength, high-temperature, chromium-rich, chromium-iron alloy is described. Experimental alloys were produced by induction melting charges of commercially available melting stock, casting into molds, and fabricating by various hot-working methods. The intended composition of the alloys produced during this period was 70 parts chromium, 30 parts iron, 6 to 9 parts molybdenum, 2 to 3 parts titanium, and up to 1/2 part aluminum. This composition had been found, in the previous year's work, to possess excellent stress-rupture properties at 1500°F.

The forging qualities of 4- and 12-pound ingots were found to improve as their soundness was improved by the use of preheated alloy charges and slag materials. A portion of this material was successfully hot rolled to thin strip. Little success was achieved in attempts to forge larger ingots. No room-temperature ductility as indicated by bend tests, was found in rolled strip before or after heat treating. However, the heat treatments affected the hardness and the microstructure. Specimens machined from forged bars of the alloy exhibited remarkable thermal-shock properties when tested at 1800° to 2000°F.

Battelle Memorial Institute. WADC TR 54-391. INVESTIGATION ON NOTCH SENSITIVITY OF HEAT-RESISTANT ALLOYS AT ELEVATED TEMPERATURE (RUPTURE STRENGTH OF NOTCHED BARS AT HIGH TEMPERATURES). R. L. Carlson, R. J. MacDonald, W. F. Simmons. AF 18(600)-61. October 1954.

Stress-rupture tests were conducted on notched and unnotched or plain bars of S-816, Inconel "X" Type 550, and Waspaloy alloys at test temperatures ranging from 1200° to 1600°F. The notched specimens had 50 percent, 60-degree, V-notches with the root radii ranging from 0.005 inch to 0.100 inch. In some tests, as many as three notches of different root radii were used.

The test results indicated that S-816 alloy was notch strengthened by all of the notches used, in the temperature range from 1350°F to 1600°F. Inconel "X" Type 550 was always notch strengthened by all of the notches only at the test temperature of 1600°F. Waspaloy was always notch strengthened by all notches only at the temperature of 1500°F. Both Inconel "X" Type 550 and Waspaloy could be notch strengthened for some test conditions (notch sharpness and time) at temperatures below 1600°F and 1500°F, respectively.

Factors considered to have an influence on stress-rupture behavior have been studied and the results are included. The factors investigated are notch geometry, notched and unnotched ductility, the modes of deformation and fracture, metallurgical changes, and surface condition. The influence of some of these factors can vary considerably from alloy to alloy. It does not appear possible, therefore, to evaluate the notch and unnotched stress-rupture behavior of a given alloy completely by any simple method. Rather, an evaluation should be based upon the combined consideration of those factors that are influential in each individual case.

Allegheny Ludlum Steel Corporation. WADC TR 54-276. RESEARCH AND DEVELOPMENT OF WROUGHT AND CAST HIGH TEMPERATURE ALLOYS. R. R. MacFarlane, R. S. DeFries, E. E. Reynolds, W. W. Dyrkacz. AF 18 (600)-149. October 1954. ASTIA Document No. AD 58695. PB 129969. Order from LC, Mi $5.40, Ph $15.30.

Study of wrought and cast Co-base and Fe-base alloys was conducted with the object of development of better high-temperature alloys having a minimum strategic alloy content. An alloy containing 10Ni, 10 Cr, 10 W, 5 Mo, and 1 Cb + Ta was outstanding in rupture properties for the wrought Co-base alloys at 1500°to 1700°F. An 18 Mn, 12 Cr, 3 Mo, .8V alloy had a good combination of properties for application at 1200°F for the wrought Fe-base alloys. Thermal shock properties were best for the

cast alloys containing the highest Co. No correlation was apparent between thermal shock characteristics and other commonly measured properties.

Sam Tour & Co., Inc. WADC TR 54-451, Part 1. EVALUATION OF SURFACE TREATMENTS FOR LOW-ALLOY STEELS (PART 1. TEST METHOD FOR HEAT-RESISTANT CORROSION PROTECTIVE COATINGS ON STEEL). Sam Tour. AF 33(616)-406. November 1954. PB 121087. Order from OTS $0.75.

The test procudure involves two types of artificially created environments. The first step is a cyclic or repeated exposure for one week at the desired temperature in an atmosphere of products of combustion. The second step is a cyclic exposure for one week in an alternate condensation corrosion test unit at room temperature or slightly above.

The atmosphere containing products of combustion is obtained by the use of a kerosene torch, kerosene plus suitable additives and an excess of air. The atmosphere produced in this manner is conducted into a full muffle in a temperature controlled furnace. The panels are hung on racks in the furnace muffle where they are heated to the desired temperature for 6 hours each day for five continuous days. During the remaining 18 hours of each day they remain in the closed muffle with no heat applied to the furnace and no artificial atmosphere being introduced into the muffle.

The alternate condensation test equipment has a large turntable revolving about 15 times per hour and on which the specimen panels are mounted. The turntable carries the panels successively through (a) a tunnel where they are cooled with dry air, (b) a tunnel where they encounter warm moist air so as to collect a layer of condensate and (c) an open space where the condensate may evaporate into the room atmosphere.

Sixteen different coatings have been tested. Some of the coatings were tested at temperatures of 600°, 800° and 1000°F, others, at 1000°, 1200° and 1400°F in the atmosphere of combustion products of kerosene with additives. Eight of the sixteen coatings mentioned above have been tested at 1000°F in the atmosphere of the combustion products of kerosene without additives.

When the additives were not used, different corrosion effects were observed. These panels withstood the testing conditions in the furnace and in the alternate condensation test better than when the additives were used.

It should be pointed out that this test method is specialized and rather severe; the success or failure of a coating in this test does not imply similar results under other conditions of service or tests.

Sam Tour & Company, Inc. WADC TR 54-451, Part 2.
EVALUATION OF SURFACE TREATMENTS FOR LOW ALLOY STEELS. (PART 2. PAINT CHROMIZING, PAINT SILICONIZING, AND COATING OF TITANIUM-BORON LOW ALLOY STEEL). Sam Tour. AF 33(616)-406. November 1954. PB 121088. Order from OTS $0.75.

Diffusion coatings produced by chromizing or siliconizing offer considerable promise as heat resistant corrosion protective coatings for use on low to medium carbon, plain carbon or low alloy steels for service at temperatures up to 1200°F. Either of these types of coatings can be applied by the paint process.

The paint used consists of a suspension or slurry containing (a) the desired metal powder (chromium or silicon), (b) a fluoride, (c) glass frits of low and high melting points, (d) a liquid vehicle containing suspension agents and binders, and (e) a volatile thinner.

Substantially no pre-preparation of the steel surface is necessary. The paint is applied by brush, dip or spray. Several coats with intermediate air drying are required. After preheat, the work is heated or fired in an open furnace. Temperatures of 1900° to 1950°F for 1 to 3 hours are required for chromizing. Temperatures of 1750° to 1850°F for 10 to 30 minutes are required for siliconizing. In either case, as furnace heating proceeds, the binders in the paint burn away and the glass frits melt to form a molten protective, but reactive, blanket enveloping the work and protecting it from oxidation as the chromizing or siliconizing reaction takes place. Upon removal from the furnace and cooling, the glass freezes, shrinks and cracks away, leaving the treated surfaces relatively clean.

The low alloy titanium-boron steels can be paint chromized or paint siliconized. The coatings provide protection against scaling during solution heat treatment at temperatures as high as 2100°F.

The chromized cases on low carbon steel are continuous, uniform, ductile layers that withstand bending and forming operations and can be welded. The siliconized cases are hard, brittle and zonal in nature, with a thin interfacial zone that is continuous.

Further development work on both the paint chromizing and the paint siliconizing processes is outlined and recommended.

Crucible Steel Company of America. WADC TR 54-472.
DEVELOPMENT OF AUSTENITIC IRON-BASE SHEET ALLOY. Alfred G. Allten, Edward Sadowski, Allan Simon, Peter Lillys. AF 33(616)-2047. December 1954.

Two types of high-phosphorus austenitic precipitation-hardening steels were investigated to determine their suitability as sheet alloys for jet engine components. One of the steels which contained approximately 0.4 C, 8.0 Mn, .2P, 8.0 Ni, 19.5 Cr, 1.2 Mo, .2N was tested in sheet form. This steel largely met the objectives of the investigation, in that it contained not less than 50% iron, and was stronger than N-155 at 1200°, 1350° and 1500°F. Also, this steel had oxidation resistance only slightly inferior to that of N-155 at 1700°F. However, the steel was not readily weldable, and further investigation of this property would be required to properly evaluate the steel.

University of Michigan. WADC TR 53-277, Part 2. HIGH-TEMPERATURE PROPERTIES OF FOUR LOW-ALLOY STEELS FOR JET-ENGINE TURBINE WHEELS. Adron I. Rush and James W. Freeman. AF 33(038)-13496. February 1955. ASTIA Document No AD 64926. PB 133 856. Order from LC, Mi$6.30, Ph $19.80.

The relationships between type of microstructure and properties at 700°F to 1200°F were surveyed for four low alloyed steels. The steels were SAE 4340, 1.25 Cr-Mo-Si-V ("17-22A"S), 3 Cr-Mo-W-V (H-40), and 13 Cr-Mo-W-V (C-422). Near pure structures were produced by isothermal transformation at a series of temperatures. Martensitic structures were produced by oil quenching. Normalized specimens were also included. Maximum Brinell hardness was kept at 280-320 by tempering the structures which had higher hardness as transformed.

The results indicated that bainitic structures had maximum strength over the temperature range. Tempered martensite in general had intermediate to low strengths. Pearlites were relatively weak at low temperatures, but became similar to the bainites at the higher temperatures. There was considerable variation between

high and low temperature bainite and between fine and coarse pearlite. Normalized materials apparently have generally high levels of strength because the usual structures developed are predominantly bainite.

In most cases, rather wide variations in structure were possible with rather uniform properties. There were, however, usually a predominantly strong and an abnormally weak structure within the generalizations. Alloy content controlled the level of strength for a given structure. Thus, while martensitic structures compared unfavorably to the bainites for SAE 4340 and "17-22A"S, the martensitic structure of the C-422 alloy was superior to the lower alloyed steels at the higher temperature and longer time periods. Reasonably good correlations were developed between the structures and properties of turbine wheels of the four alloys on the basis of the results of the survey. The general results from the survey appear to be useful for general guidance in heat treating alloys for high temperature service. However, the survey is very limited and care should be used in extending the data until all the factors have been investigated.

University of Minnesota. WADC TR 54-488, Part 1.
STRESS-RUPTURE, FATIGUE AND NOTCH SENSITIVITY PROPERTIES OF HIGH TEMPERATURE ALLOYS. Franz H. Vitovec, Benjamin J. Lazan. AF 33(038)-20840. February 1955.

Fatigue and stress-rupture data obtained under various combinations of mean and alternating axial stress and static creep data are presented and discussed for S-816 alloy at 75°, 1350°, 1500° and 1650°F. Tests were performed under axial stress on unnotched specimens and specimens having theoretical notch sensitivity factors of 2.4 and 3.4. The data are presented as S-N curves and stress range diagrams to show the effect of specimen notch, temperature, alternating-to-mean stress ratio, and stress magnitude on the fatigue and stress-rupture properties.

The role of both creep and fatigue as factors in rupture is discussed with particular reference to temperature and alternating-to-mean stress ratio.

U. S. Bureau of Mines. WADC TR 53-272, Part 2.
CHROMIUM-BASE ALLOYS. Haruo Kato, Earl T. Hayes. PO 33(038)-50-1084 E. April 1955.

The high temperature stress rupture properties of alloys of 60Cr-40Fe to which was added either molybdenum, nickel or cobalt were investigated. Improvements in alloy preparation involving the use of high purity stock and consumable arc melting lowered the oxygen content from approximately 3000 ppm to 300 ppm and improved ductility. These alloys showed very short creep rupture life at 1500°F and 20,000 psi.

Climax Molybdenum Company of Michigan. WADC TR 55-96.
DEVELOPMENT OF MOLYBDENUM NOZZLE BLADES. D. V. Doane. AF 33(600)-23851. April 1955. ASTIA Document No. AD 75793. PB 123113. Order from LC, Mi $2.40, Ph $3.30.

This report describes the development of methods to produce coated molybdenum gas turbine guide vanes (nozzle blades) conforming as closely as possible to USAF Drawing X52D9613. Under this contract 60 vanes have been fabricated and 6 vanes have been coated, using two different coating procedures. The detailed fabrication procedures, coating experiments, and detailed coating procedures are presented in the report.

Allegheny Ludlum Steel Corp. WADC TR 55-23. DEVELOPMENT OF WROUGHT AND CAST ALLOYS FOR HIGH TEMPERATURE APPLICATIONS. R. R. MacFarlane, R. S. DeFries, E. E. Reynolds, W. W. Dyrkacz. AF 33(616)-2463. April 1955. PB 111891. Order from OTS $2.50.

Developmental studies were conducted on wrought Fe-base and both wrought and cast Co-base alloys for applications at high temperatures. A heat treatable, Fe-base, austenitic alloy containing Mn and Cr was modified with B to give excellent stress-rupture properties at 1200°F. Oxidation resistance was greatly improved by small Al additions. A wrought Co-base alloy with good stress-rupture properties at 1600° and 1700°F and improved oxidation resistance was developed. Composition levels giving optimum properties were determined for the cast Co-base alloys. Modifications involving B were investigated in both wrought and cast Co-base alloys.

University of Michigan. WADC TR 55-470, Part 1. AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEELS AND HIGH-NICKEL ALLOYS. Clarence A. Siebert, Maurice J. Sinnott, Lynn H. De Smyter, Robert E. Keith. AF 33(616)-353. June 1955. ASTIA Document No. AD 84494. PB 131471. Order from OTS $2.75.

Chromel alloys ASM, ARM and D, and type 310 stainless steels were oxidized for 100-hour periods in the stressed condition. The above alloys and Inconel were oxidized for times up to 500 hours in the unstressed condition. Intergranular oxidation measurements were obtained microscopically. The influence of stress was to cause an increase in the intergranular penetration increased with increasing time and temperature. Increasing the water-vapor content of the air increased the intergranular penetration slightly. The effect of a preferred orientation decreased the intergranular penetration slightly. The weight gained during oxidation was determined. It was found that in the alloys tested that a plot of the square of the specific weight gain versus temperature resulted in a straight-line relationship. Visual and magnetic examinations were made on the oxidized specimens and their oxides. No correlation between these observations and oxidation properties could be determined. X-ray diffraction patterns were made on representative oxides. This analysis showed the scales encountered to be of a protective nature by Randall and Robbs criteria of the presence of Cr_2O_3 or high-parameter spinel. Electron diffraction examination of the subsurface structure was performed on type 310 stainless steels oxidized in the unstressed condition. It was found that the oxidation products in the subsurface region were substantially the same as the surface oxides as determined by X-ray diffraction techniques.

University of Michigan. WADC TR 54-120, Part 2. AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEELS AND HIGH-NICKEL ALLOYS. Clarence A. Siebert, Maurice J. Sinnott, Lynn H. De Smyter, Robert E. Keith. AF 33(616)-353. June 1955.
(Abstract Same as Above)

National Research Corporation. WADC TR 55-218. INVESTIGATION OF NICKEL BASE PRECIPITATION HARDENING ALLOYS. David I. Siniser. AF 33(616)-2144. September 1955. PB 111791. Order from OTS $1.25.

Vacuum-melted Waspaloy was made with controlled carbon content and varying ratios of titanium to aluminum. All nominal compositions were within the chemical specification ranges for commercial Waspaloy. Standard melting and casting procedures were satisfactory, except

that an unusually large hot top appears necessary for producing sound ingots. All ingots forged readily. Increasing Ti/Al ratio from about 1.5 to 3.0 had no appreciable effect on rupture life or minimum creep rate at 1500°F for stresses from 30,000 to 35,000 psi. Both elongation and reduction of area at failure appeared to decrease with increasing Ti/Al ratio. None of the alloys were notch sensitive in stress-rupture at 52,500 psi and 1350°F. There was an indication of a minimum notch sensitivity at a Ti/Al ratio of about 2.0 for both stabilized and unstabilized vacuum-melted Waspaloy.

The Babcock & Wilcox Company Research Center. WADC TN 55-290. DEVELOPMENT OF CAST IRON-BASE ALLOYS OF AUSTENITIC TYPE FOR HIGH HEAT-RESISTANCE AND SCALE-RESISTANCE. F. Eberle, W. E. Leyda, W. Feduska, F. B. Snyder. AF 33(616)-2413. October 1955.

For the purpose of developing iron-base substitute alloys for the cobalt-base alloy H.S. 21, distinguished by

1. a minimum iron content of 45%
2. a fully austenitic microstructure
3. adequate oxidation resistance at 2000°F
4. a creep-rupture strength at 1600° and 1800°F similar to that of H.S. 21, and
5. suitability for fabrication by the investment-casting or shell-molding process

over 100 investment-cast experimental compositions were studied. A base composition containing 45% Fe, 20% Cr, and 27% Ni was found to possess the required lattice structure and oxidation resistance and to retain these characteristics when strengthened by the addition of 4-7.5% of carbide- and compound-forming elements. For strengthening purposes, dual and triple additions of Ta, Cb, Ti, Zr, Be, Mo, and W, at concentration ratios of 1:1:1 (1:1), 2:1:1 (2:1), 1:2:1 (1:2), and 1:1:2 were investigated. The most-promising composition found was an alloy containing:

0.52C, 1.11Mo, 0.7Si, 19.8Cr, 27.8Ni, 2.02Cb, 0.63Ta, 2.2W, 0.05CeLa.

Its rupture strength at 1600° and 1800°F was equal to about 80-85% that of H.S. 21. Preliminary tests indicated the possibility of raising the strength level of this alloy further by increasing the tungsten content relative to that of CbTa and by replacing part of the nickel by cobalt.

Southern Research Institute. WADC TR 55-391. TENSILE, FRACTURE, AND SHORT-TIME CREEP PROPERTIES OF AIRCRAFT-STRUCTURAL MATERIALS AT VERY HIGH TEMPERATURES AFTER RAPID HEATING. J. Robert Kattus, Clifford L. Dotson. AF 33(616)-2837. December 1955. ASTIA Document No. AD 110560.

The tensile, fracture, and short-time creep properties of electrolytic copper sheet, ingot iron sheet, tantalum sheet, molybdenum sheet, and four types of graphite -- types GBE, 3474-D, 7087, and GBH -- were determined at temperatures up to the melting points of the metals and up to 5200°F for the graphite. High rates of heating were used in all tests, and the tensile tests were conducted at moderate to rapid strain rates after relatively short holding times at the test temperatures.

The metals decreased in tensile strength continuously with increasing temperatures, but they did retain some strength at temperatures approaching the melting points. The graphite materials decreased little in strength between room temperature and 5200°F.

The copper and iron were capable of supporting a stress of 200 psi for five minutes at temperatures near the melting points. For five minutes at temperatures between 4600°F and 4800°F, each type of graphite supported a load equivalent to two-thirds of the room-temperature strength. Because of deterioration of the specimens due to oxidation, molybdenum and tantalum were not able to support a stress of 200 psi for as long as one minute at temperatures near their melting points. At maximum temperatures of 1900°F and 2200°F, the molybdenum and tantalum, respectively, were capable of supporting a stress of 10,000 psi for five minutes.

WADC TR 54-391, Suppl. 1. INVESTIGATION ON NOTCH SENSITIVITY OF HEAT RESISTANT ALLOYS AT ELEVATED TEMPERATURES, Suppl. 1. APPARENT VOLUME INCREASE DUE TO TRIAXIAL STRESSES. W. E. Dirkes, Capt., USAF. January 1956.

The volume of structural metals is generally considered to remain constant even with extremely high stresses applied. Some data recently published indicate significant changes in volume under stress-rupture test conditions for specimens having stress gradients. More recent analysis of the same data and specimens together with additional measurements from these specimens show that significant volume changes did not actually occur, but that plane cross-sections of specimens did not always remain plane when subjected to stress gradients.

University of Michigan. WADC TR 55-388. A SURVEY OF THE EFFECT OF AUSTENITIZING TEMPERATURE AND RATE OF CONDITIONOUS COOLING ON THE STRUCTURE AND 700°F to 1200°F PROPERTIES OF THREE LOW-ALLOYED STEELS. Kenneth P. MacKay, A. Phillip Coldren, Adron I. Rush, James W. Freeman. AF 33(038)-13496. January 1956. PB 121149. Order from OTS $2.00.

The relationships between microstructures formed with various cooling rates and austenitizing temperatures and properties at 700° to 1200°F were surveyed for three low-alloyed steels. The steels were Ni-Cr-Mo-(SAE 4340), 1.25Cr-Mo-V ("17-22A"S), and 3Cr-Mo-W-V (H-40). Martensite, martensitic-bainitic, bainitic, and bainitic-ferritic structures were produced by oil quenching 1-inch rounds and air cooling 1-inch and simulated 3- and 6-inch rounds. Modifications of the oil-quenched and normalized structures of the 1-inch rounds resulted when the austenitizing temperature was raised from 1750° to 2100°F. The hardness level was maintained at 280-320 BHN by tempering the structures which had higher hardnesses in the as-transformed condition.

The results indicated that the fully bainitic structures which were predominantly upper bainite had maximum strength over the range of testing temperature used. In general, such structures were found in the larger, normalized sections and with the higher austenitizing temperature. Since low values of ductility resulted from the higher temperature treatments, however, the best combinations of strength and ductility were obtained when the largest sections were normalized from the lower austenitizing temperatures (1750°F for SAE 4340 and "17-22A"S, and 1950°F for H-40).

Regarding the effect of varying the cooling rates of normalized bars, it was found that increases in strength occurred for all three steels as the effective bar diameter was increased from 1 inch to 6 inches. The effect of raising the austenitizing temperature from 1750° to 2100°F was to increase the strength, with the H-40 steel being affected the most. Ductility was lowered for all three steels as the heat-treating temperature was raised.

Sylvania Electric Products, Inc. WADC TR 55-501.

DEVELOPMENT OF HEAT RESISTANT ALLOYS BY POWDER METALLURGY TECHNIQUES. G. Zuromsky, L. Sama, H. S. Kalish, L. L. Seigle. AF 33(616)-2401. March 1956. PB 121356. Order from OTS $2.00.

Powder metallurgical techniques were utilized in the production of Cr-W-Co alloys, to meet a stress rupture specification of 100 hours life at 870°C and 25,000 psi. Attempts to produce chromium-base alloys with room temperature ductility and/or high density were unsuccessful. The use of a master alloy improved the densities of the cobalt-base alloys. Stress-rupture properties of the cobalt-base alloys at 870°C and 25,000 psi appeared to approach those of the weaker Stellites but failed to meet the 100-hour requirement. Nickel additions lowered the stress-rupture strength but gave increased ductility. Attempts to retain carbon as an alloying addition were unsuccessful.

Universal-Cyclops Steel Corporation. WADC TR 56-114.

INVESTIGATION OF THE EFFECTS OF INCONGRUOUS ELEMENTS AND THE INTERACTION EFFECTS OF THESE ELEMENTS ON HIGH TEMPERATURE STRENGTH OF FE-CO-NI-CR ALLOYS. Thomas L. Robertshaw, Francis M. Richmond. AF 33(616)-2777. April 1956. PB 121379. Order from OTS $1.75.

An investigation was made of the effect of various hardening elements on the high temperature properties of vacuum melted alloys having a base composition of 60 atomic % Ni, 20 atomic % Cr, 10 atomic % Fe and 10 atomic % Co. Stress-rupture properties of these alloys at 1600°F were largely dependent on (1) the vacuum obtained during melting, (2) the fabrication methods, and (3) the total amount of hardener. On the basis of atomic misfit, the hardening elements B, Zr and Cb were equivalent to Mo in increasing the rupture strength of these alloys. On this same basis, however, Ti was more potent and Al less potent than Mo. A maximum in stress-rupture stress occurred at a hardener level of about 35 equivalent atomic percent Mo.

It was found that, in general, a high level of strength was more likely to occur at a given hardener level when greater variety of hardening elements were added.

P. R. Mallory & Co., Inc. WADC TR 56-190.

A STUDY OF THE POSSIBILITY OF REINFORCING HIGH-TEMPERATURE ALLOYS BY ADDITION OF REFRACTORY POWDERS. John D. Burney. AF 33(616)-2959. May 1956. PB 121474. Order from OTS $1.25.

A study of the possibility of reinforcing 80 Ni-20 Cr alloy by the addition of such refractory oxides as Al_2O_3, TiO_2, ZrO_2, Cr_2O_3 and SiO_2 was made.

Several powder metallurgical fabrication techniques were investigated such as (1) pressing and sintering, (2) pressing, sintering and repressing, (3) pressing, sintering followed by hot working, (4) hot pressing and (5) liquid phase sintering.

In the "press-sinter" method of fabrication, bars with 1.0% TiO_2 showed better stress-rupture properties than bars with any of the other oxide additions investigated. Rupture times were 7.45 and 8.7 hours at 1600°F and 7000 psi.

Hot forging techniques were found to be applicable for working bars containing oxide additions of 1.0% or less.

An 80 Ni-20 Cr alloy reinforced with 1% Al_2O_3 and densified by a liquid phase sintering technique resulted in improved stress-rupture properties compared to wrought 80 Ni-20 Cr alloy. This material had a rupture life at 1600°F and 7000 psi in excess of 268 hours; while at 1800°F and 6650 psi, the rupture life was almost 47 hours.

The room temperature impact and ultimate tensile strength appeared adequate and the oxidation resistance and thermal shock properties were good.

Cornell Aeronautical Laboratory, Inc. WADC TR 54-270, Part 2.

INVESTIGATION OF THE COMPRESSIVE, BEARING, AND SHEAR CREEP-RUPTURE PROPERTIES OF AIRCRAFT STRUCTURAL METALS AND JOINTS AT ELEVATED TEMPERATURES. Frank J. Vawter, Glen J. Guarnieri, Luke A. Yerkovich, George Derrick. AF 33(616)-190. September 1956. ASTIA Document No. AD 97283. PB 121656.

The establishment of high-temperature creep and rupture properties of materials is a prerequisite for design if exposure to elevated temperature in service is anticipated. These properties, which are determined from the conventional tensile creep test, are of questionable value if stress conditions other than tension are encountered. To supplement these existing tensile creep and rupture data, this project was initiated to determine the high-temperature creep strengths of a number of structural aircraft alloys when subjected to compression, bearing, and shear stresses.

Tensile creep data are included for A-70 commercially-pure titanium, C-110M titanium alloy and SAE 4130 alloy steel sheet. Bearing creep data are presented for A-70 and C-110M titanium, 4130 steel and type 321 stainless steel. In addition, results of shear-pin deformation tests on 2117-T4 aluminum, Monel and type 301 stainless steel wire and compression creep test results for 2024-T3 aluminum sheet and plate and C-110M titanium alloy sheet are included.

University of Michigan. WADC TR 54-175, Part 3.

NOTCH SENSITIVITY OF HEAT-RESISTANT ALLOYS AT ELEVATED TEMPERATURES, Part 3. Final Data and Correlations. Howard R. Voorhees, James W. Freeman. AF 18(600)-62. September 1956. ASTIA Document No. AD 97253. PB 121791.

Earlier parts of this report summarized rupture lives for smooth and notched round specimens of three heat-resistant alloys, together with pertinent experimental data on tensile stress-strain properties and creep-relaxation characteristics for these alloys. Similar types of data have now been obtained for flat specimens, and for two other types of material (a Cr-Si-Mo-V steel and an age-hardening aluminum alloy).

Results gathered during the over-all program lead to the following conclusions:

1. Elevated-temperature rupture characteristics of notched specimens under a steady tensile load appear to depend on three major factors:
   a. The distribution and level of the initial stress pattern, determined by the notch configuration and tensile characteristics of the alloy.
   b. The rate at which variable creep rates at different locations in the cross section are able to relax the peak stress originally concentrated near the notch. Under multiaxial stressing the effective stress can easily become less than the nominal value for alloys with low creep resistance.
   c. Rupture characteristics of the material at the prevailing stresses and for the prior history experienced by different fibers in the notched bar. If too large a portion of the total life is used up

at the initial high stresses, the remaining service will be short even if the final stress is low.

2. It appears that rupture life of a structure in the presence of a concentrated complex stress involves no new factors beyond those which determine rupture under simple stress. If one can predict the stress-strain-time history at points throughout a body, the time until rupture at any point seems amenable to calculation from data obtained with smooth specimens. The major uncertainty is choice of the proper criterion for initiation of rupture following creep under variable complex stresses.
3. For some alloys a small amount of plastic deformation near the notch when the load is added may radically alter subsequent creep-rupture characteristics. This may be the major factor in notch weakening under some conditions.

Allegheny Ludlum Steel Corp. WADC TR 56-327.
INVESTIGATION OF FORGED COBALT BASE ALLOYS FOR HIGH TEMPERATURE APPLICATIONS.
R. R. MacFarlane, R. K. Pitler, E. E. Reynolds.
AF 33(616)-2882. October 1956. ASTIA Document No. AD 110409. PB 121723.

Developmental studies were made of a wrought Co-base alloy for applications at high temperatures. Additions of Al improved oxidation resistance and lowered rupture strength. B additions up to .2 percent increased rupture strength and ductility. This increase was limited by a necessarily low solution temperature of 2150°F since, at 2200°F, eutectic melting of a B phase occurred. Ti additions resulted in a large increase in room temperature hardness through an aging reaction but yielded no improvement otherwise. Attempts at improving the workability of unforgeable air melted modifications by vacuum consumable electrode and vacuum induction melting techniques were not successful.

Univ. of Michigan. WADC TR 55-470 Part 2.
AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEELS AND HIGH-NICKEL ALLOYS. C Clarence A. Siebert, Maurice J. Sinnott, Lynn H. DeSmyter, Harry M. Ferrari. AF 33(616)-353. Oct. 1956. ASTIA Document No. AD 110440. PB 121 795.

Specimens from Chromel ASM, Hastelloy B, and commercial and vacuum-melted type 310 stainless steel alloys were oxidized for 100-hour periods in the stressed condition. Intergranular oxidation measurements were obtained microscopically. In general, the intergranular penetrations increased rapidly with stress after a certain minimum value was reached. This minimum value, denoted as the threshold stress, was determined for each alloy at various temperatures. The weight gained during oxidation was determined. It was found that most of the alloys tested followed the parabolic oxidation law.

The Babcock & Wilcox Company Research Center.
WADC TN 55-290 Part II.
DEVELOPMENT OF CAST IRON-BASE ALLOYS OF AUSTENITIC TYPE FOR HIGH HEAT-RESISTANCE AND SCALE-RESISTANCE. F. Eberle, J. H. Hoke, W. E. Leyda. AF 33(616)-2413. January 1957. ASTIA Document No. AD 110716. PB 121950.

Iron-base alloys containing a minimum iron content of 45%, a minimum carbon content of 0.50%, a chromium content of not less than 20%, and between 27 and 32% nickel, part of which may be replaced by cobalt, can develop a creep-rupture strength at 1600° to 1800°F approaching that of the cobalt-base alloy H. S. 21 when they are strengthened by not more than 5% additions of Cb, Ta, Mo, and W in certain combinations and relative concentrations. These high-creep-rupture-strength iron-base alloys are equal to H. S. 21 in oxidation resistance at 2000°F, but inferior to the former in short-time cold and hot tensile strength, in cold ductility, and in resistance to thermal shock and thermal fatigue. The possibility of improving the latter by lowering the carbon content and compensating the resulting decrease in rupture strength with suitable modifications in composition has not been explored.

Climax Molybdenum Company of Michigan.
WADC TR 56-454.
INVESTIGATION OF THE EFFECTS OF HOT-COLD WORK ON THE PROPERTIES OF MOLYBDENUM ALLOYS. M. Semchyshen, Robert Q. Barr. AF 33(616)-2861. January 1957. ASTIA Document No. AD 110708. PB 121976.

Two alloys, molybdenum-0.28% niobium and molybdenum-0.50% titanium, were investigated from the standpoint of effect of fabrication variables on the degree of strain hardening and the resultant mechanical properties and on subsequent resistance to softening with exposure to elevated temperatures. Fabrication was accomplished either by rolling or forging.

The hardness of the rolled bars increased rapidly with increasing amounts of reduction by rolling up to reductions of about 20%, then increased at a lower rate at successively higher reductions. When rolled bars were subjected to one-hour exposures at elevated temperatures, those sustaining the highest reductions displayed rapid decline in hardness and complete recrystallization in the range from 2250 to 2400°F for the niobium alloy and 2600° to 2800°F for the titanium alloy. Bars sustaining less reduction by rolling generally displayed a gradual approach to minimum hardness with increasing exposure temperature.

Forging studies revealed a rather complex dependence of degree of strain hardening upon composition, specimen condition prior to forging, forging temperature, and amount of reduction. Specimens in the strain hardened condition prior to forging developed substantially higher hardness than specimens partially or fully recrystallized prior to forging. At the highest forging temperature investigated, 2400°F, specimens from the niobium alloy were less severely strain hardened than comparable specimens form the titanium alloy, owing to the relatively greater structural stability of the latter. A relationship was evolved between as-forged hardness and softening on exposure to elevated temperatures.

Tensile tests at room temperature and 1800°F taken from specimens containing a gradient in the degree of strain hardening invariably failed at locations corresponding to minimum strain hardening. Certain creep-rupture tests at 1800°F, however, failed at locations corresponding to maximum strain hardening. Examination of these specimens indicated that partial recrystallization and softening had occurred under the combined influence of time, temperature and stress at locations where no structural changes had taken place after exposures under similar conditions of time and temperature only.

Univ. of Michigan. WADC TR 57-40.
AN INVESTIGATION OF THREE FERRITIC STEELS FOR HIGH-TEMPERATURE APPLICATION. A. Phillip Coldren, James W. Freeman. AF 33(616)-3239. APRIL 1957. ASTIA Document No. AD 118204. PB 131069.

An investigation was carried out to survey the relationships between microstructure and properties at 700° to 1100°F for low-alloy, hardenable steels. A Ni-Cr-Mo

(SAE 4340) and two Cr-Mo-V ("17-22-A"S and "17-22-A"V) steels were studied. The results, together with those presented in References 1 and 2 for previous work on 4340 and "17-22-A"S steels, correlate properties with microstructure for three temperatures of isothermal transformation in the pearlite region and three in the bainite region. Oil-quenched and normalized structures were included for comparison. The properties were evaluated for these structures when tempered to both the 300 and 350 Brinell hardness levels. Data for the "17-22-A'V steel in the form of TTT diagram obtained to establish heat-treating condition is included.

These data are supplemented by similar studies for continuous cooling down to rates simulating the air cooling of a 6-inch round. The influence of increasing the the heat-treating temperature was also studied. Controlled mixed structures were developed by transforming about 50 percent in the upper bainitic range and about 50 percent in the lower bainitic range. The results of a very limited start for a study of hot working condition effects is included.

The results are correlated and analyzed in the report. Optimum structures (or treatments) vary with both the alloy and test conditions. In general, bainitic structures of one type or another gave best properties. However, for the Cr-Mo-V steels, pearlites formed at relatively low temperatures were often as strong or stronger than the bainites. Tempered martensite was never the strongest structure and most often was the weakest.

General Electric Department. WADC TR 57-154. EFFECTS OF PROCESSING VARIABLES ON FRACTURE AND DISC-BURSTING CHARACTERISTICS OF FOUR HIGH TEMPERATURE MATERIALS. Richard Sakamoto. AF 33(616)-2778. May 1957. ASTIA Document No. AD 118346.

Generalization of notch sensitivity characteristics of materials evaluated by conventional notch rupture data without due regard to the specific application may represent an unjustified material penalty and unnecessarily preclude the use of a material for a specific application.

Elevated temperature spin test data for several alloys is included which indicate that certain alloys are notch insensitive in thin disc tests even though they show significant strength reduction in the conventional notch rupture tests. This difference in behavior is attributed to the greater constraint existing in the notched cylindrical specimen and indicates that the constraint factor may be fully as significant as the stress concentration.

Ford Motor Company. WADC TR 57-298 Part I. IRON-ALUMINUM ALLOY SYSTEMS. Part I - Fundamental Studies and Alloy Development. F. X. Kayser. AF 33(600)-32448. May 1959. ASTIA Document No. AD 130823. PB 137502.

An extensive program is being carried on to study the mechanical and physical behavior of recrystallized iron-aluminum alloys containing up to 18.5 weight percent aluminum. Data are presented illustrating the effect of aluminum on the stress-rupture and tensile properties. It is concluded that the recrystallized binary alloys are too low in hot strength to consider for use in moderate stress application at temperatures in excess of 1000°F. Special attention has been given to the development of higher hot strength alloys. The effect of titanium on hot strength is demonstrated.

The effects observed of order-disorder transformations on mechanical properties are of fundamental interest. Some interesting and unique magnetic phenomenon have been discovered in the binary alloys among which is a continuous change from ferro-magnetic to anti-ferromagnetic behavior.

Ford Motor Co. WADC TR 57-298. Part II. IRON-ALUMINUM ALLOY SYSTEMS, Part II - Some Investigations in Air-Melting and Application of Iron-Aluminum Alloys. Joseph J. Clark, William J. Zager. AF 33(600)-32448. May 1957. ASTIA Document No. AD 130824. PB 138158.

During the period April 1956 to April 1957 the Process Development Department of the Manufacturing Engineering Office of Ford Motor Company air-melted a series of heats of iron-aluminum alloys in the 6 to 10% aluminum range. The melting and deoxidation of these heats, performed in indirect-arc type furnaces, are described. Mechanical properties obtained on bar stock rolled from ingots cast from a number of these heats are compared with established properties of vacuum-melted alloys of similar composition. Results of some exploratory heats involving changes in basic melting stock, as well as some with small additions of third elements, are presented.

Initial attempts to produce sound castings in sand molds are described.

Performance of certain iron-aluminum alloy samples and fabricated articles in service is described.

Ford Motor Company. WADC TR 57-298, Part III. IRON-ALUMINUM ALLOY SYSTEMS, Part III - Welding of Iron-Aluminum Alloys. R. Burthwick, S. Goodman. AF 33(600)-32448. May 1957. ASTIA Document No. AD 130825. PB 137503.

Investigations were conducted to develop fusion welding and resistance welding procedures for three (3) heats of iron-aluminum alloy. Successful fusion welding procedures were developed using the tungsten inert gas shielded arc process. Variables of power type, current, voltage, shielding, travel speed, cleaning, preheat and postheat were investigated. No definite resistance welding schedule was established, although successful welds were realized with several schedules. Three fabrications were made using the fusion welding process in order to evaluate weld and material performance under service condition. The service evaluations had not been completed at the time of writing this report.

University of Michigan. WADC TR 57-58, Part I. NOTCH SENSITIVITY OF AIRCRAFT STRUCTURAL AND ENGINE ALLOYS. Part I. Preliminary Studies With A-286 and 17-7 PH (TH 1050) Alloys. Howard R. Voorhees, James W. Freeman. AF 33(616)-3380. May 1957. ASTIA Document No. AD 118289. PB 131213.

This program was designed to extend previous analyses of the creep-rupture behavior of notched test specimens held under steady axial load. Experimental studies have also been planned and carried out in an effort to clarify the factors controlling rupture life in the presence of a nonuniform complex stress.

Vacuum melted A-286 alloy produced by the consumable electrode process was chosen for this investigation with the expectation that a range of notch sensitivity could be developed by increasing the temperature of solution treatment. Extensive tests had been planned to study changes in smooth-bar properties corresponding to marked differences in notched-bar strength, in hopes of isolating all the major factors associated with notch sensitivity. For solution temperatures ranging from 1650° to 2300°F, the lot of material studied exhibited strong notch strengthening at 1200°F for nearly all specimens tested with K_t's of 1.8, 3.0 and 4.1 despite elongation at fracture as low

as 1% or less for certain solution temperature near 2200°F.

Additional smooth- and notched-specimen data were obtained for 17-7PH (TH 1050) sheet material at test temperatures of 600°, 800° and 900°F, and for two small lots of Waspaloy at 1350°F. The 17-7PH alloy exhibited a high degree of freedom from notch sensitivity. The Waspaloy was notched weakened for nominal stresses where yielding occurred at the notch root during load application. For lower nominal stresses, notch strengthening was indicated.

Results obtained indicate that reduction of an alloy's inherent strength by prior plastic deformation may be prominent factor in notch sensitivity. Examination of all available data discloses no case of notch weakening without accompanying loss of life in smooth bars which are prestrained at the test temperature. Conversely, in no known case of marked notch strengthening has the material been found to be weakened by plastic prestrain.

Prestrain effects alone may not be able to explain all notch behavior, but response of the material to plastic strains appears to be a necessary part of any complete analysis of notch effects.

Cornell Aeronautical Laboratory, Inc. WADC TR 54-270, Part 1.
INVESTIGATION OF THE COMPRESSIVE, BEARING AND SHEAR CREEP-RUPTURE PROPERTIES OF AIRCRAFT STRUCTURAL METALS AND JOINTS AT ELEVATED TEMPERATURES. F. J. Vawter, G. J. Guarnieri, L. A. Yerkovich, G. Derrick. AF 33(616)-190. June 1957. PB 121437. Order from OTS $2.50.

The intent of this investigation is to supplement conventional tensile creep data of several aircraft structural alloys with compression, bearing, and sheer properties as well. While these data alone are of interest, a correlation is being attempted between tensile creep and compression, bearing, and shear creep properties so that the latter type of data may be predicted from tensile creep properties alone.

This report includes descriptions of equipment and fixtures for conducting tensile, compression, bearing, and shear creep tests. Tensile creep properties are reported at several test temperatures for the following alloys: (1) 2024-T3 aluminum sheet, 0.064 and 3/16 inch thick; (2) C-110M titanium sheet; (3) type 321 stainless steel sheet; (4) 2117-T4 aluminum rivet wire; (5) Monel rivet wire; and (6) type 301 stainless steel rivet wire. Bearing and shear creep characteristics are included for the 2024-T3 aluminum alloy.

Westinghouse Research Laboratories. WADC TR 57-309.
JOINING OF MOLYBDENUM. W. N. Platte. AF 33(616)-3524. August 1957. ASTIA Document No. AD 149847. PB 143644. Order from LC, Mi $7.80, Ph $25.80.

An examination of available metallurgical data on molybdenum base alloys indicated that additions of aluminum, titanium, carbon, and zirconium should provide good welding properties. A group of the alloys selected was welded in a chamber containing an atmosphere of argon. Mechanical and metallurgical tests showed that aluminum additions in the range of 0.18 to 0.22% gave welds that could be deflected 120° at room temperature. However, all the alloys containing aluminum showed fine porosity. Titanium additions of 0.5% produce welds which can be deflected 120° at temperatures between 80 and 120°F depending upon the amount of carbon in the alloy. Welds in 0.5% Ti with 0.05% C produced the best results. No porosity was observed in titanium alloys. Carbon in molybdenum-carbon alloys was shown to improve the weld metal ductility by reducing the oxygen content of the base material and by a protective deoxidization action during welding. Molybdenum with 0.06% carbon showed the best bend ductility within the carbon range examined.

The effect of welding speed on the grain size of the weld metal was examined. While it is theoretically possible to reduce the weld metal grain size by increasing the welding speed, experimental difficulties were encountered due to retraction of the weld metal at speeds of 2 cm/sec. Chilling the weld failed to overcome these difficulties.

Post-welding residual stresses were measured and it was found that these stresses could be eliminated by a vacuum heat treatment at 1832°F. The residual stresses found in the welds prior to heat treatment are sufficiently large to produce fracture without additional applied stress. The post-welding stress relief was accomplished before the weld had cooled to the transition range. Crater cracking was effectively prevented by this heat treatment.

Metallwerk Plansee. WADC TR 55-243.
DEVELOPMENT OF ALLOYS HAVING GOOD HIGH TEMPERATURE PROPERTIES THROUGH POWDER METALLURGY TECHNIQUES. Dr. R. Kieffer, Dr. F. Benesovsky. AF 61(514)-741-C. September 1957. ASTIA Document No. AD 131071.

Pure titanium carbide and titanium carbide with molybdenum carbide, vanadium carbide and chromium carbide have been alloyed with refractory binding agents such as iron-aluminum alloys with a content of 8 to 14% of aluminum as well as Fe-Al-Cr, Fe, Cr-Al and Fe-Al-Mo. The carbide phase has been varied from 5 to 35%.

The most promising alloys found out among the 12 series of alloys consisted of a binding agent of iron-aluminum with 12 or 14% of aluminum and of mixed crystals of titanium carbide -- chromium carbide 90/10.

All alloys examined can be shaped to a certain extent at high temperatures.

Crucible Steel Company of America. WADC TR 57-242.
INVESTIGATION OF Fe-Mn-Cr-N-C SYSTEM FOR HEAT RESISTANCE AND OXIDATION RESISTANCE BETWEEN 1200° and 2000°F. Chi-Mei Hsiao, Edward J. Dulis. AF 33(616)-3318. November 1957. ASTIA Document No. AD 142086. PB 131563.

A comprehensive study of wrought Cr-Mn-C-N steels was conducted with the objectives, (1) of developing outstanding elevated-temperature steels that contain a minimum amount of strategic alloying elements, and (2) of advancing the existing knowledge of this new type of steel. Based on the results of the present investigation phase boundaries of the base compositions of stable austenitic Cr-Mn-C-N steels have been established, and the effects of these elements and of V, W, Mo, and Cb on the microstructures, and the room- and elevated-temperature properties of the steels have been evaluated. A nomograph was prepared to facilitate the designing of the base compositions of these steels, and the estimating of their room- and elevated-temperature properties.

These steels are not as resistant to oxidation as are the Cr-Ni steels at comparable Cr levels. However, Cr-Mn-C-N base steels that have properly balanced compositions possess an excellent combination of strength and ductility at both room- and elevated-temperatures. For example a steel containing .6C, 12 Mn, 24 Cr, .7 N, .4 V, 2 W and .9 Cb, had a 185,000 psi tensile strength,

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a 120,000 psi yield strength, a 48% elongation, and a 40% reduction of area. Also, its rupture strength was superior to those of 16 Cr-25 Ni-6 Mo, N155, 19-9DL and A-286 in the temperature range of 1200° to 1400°F.

Crucible Steel Company of America. WADC TR 57-343.

A STUDY OF THE METALLURGICAL PROPERTIES THAT ARE NECESSARY FOR SATISFACTORY BEARING PERFORMANCE AND THE DEVELOPMENT OF IMPROVED BEARING ALLOYS FOR SERVICE UP TO 1000°F. Gopal K. Bhat, Alvin E. Nehrenberg. AF 33 (616)-3318. November 1957. ASTIA Document No. AD 142117. PB 131609.

The use of bearings made from hot work steels and other tool steels in experimental engines has resulted in a few premature engine failures. Unfortunately, very little has been known about the elevated temperature properties such as hot hardness, compressive yield strength, resistance to softening and structural and dimensional stability of these hot work and other tool steels. This report describes the work done to obtain these material properties for 29 steels ranging in type from SAE 52100, its modifications, to hot work and other tool steels. An analysis of the data obtained shows that Halmo, VSM, M50, M10, T1, M2, M1 and two experimental compositions one, Steel B, containing 0.70 carbon, 4.20 chromium, 0.60 vanadium,and 5.30 molybdenum, and the other, Steel G, containing 1.31 carbon, 4.07 chromium, 4.13 vanadium, 5.75 tungsten, and 4.87 molybdenum, are suitable for elevated temperature aircraft bearing application. From a point of view of temperature range of application these steels have been classified as followed:

Room Temperature up to 700°F Halmo 1
Room Temperature up to 800°F VSM, M50, M10 and Steel B
Room Temperature up to 900°F T1, M2, M1 and Steel G

None of the steels investigated appeared suitable for application at 1000°F.

WADC TR 57-717.

INTERMEDIATE PHASES IN THE IRON-TUNGSTEN AND COBALT-TUNGSTEN BINARY SYSTEMS. Edward C. Van Reuth. December 1957. ASTIA Document No. AD 142258. PB 131627.

Fifty-three alloys have been examined in the Fe-W and Co-W alloy systems in an attempt to verify the finding of sigma phases in these systems as reported by Goldschmidt. Selected regions of both vinary equilibrium diagrams were investigated by examining alloys metallographically and by X-ray diffraction techniques. The results of this investigation indicate that neither of these systems contains a sigma (σ) phase. However, they do contain an xi (ξ) phase (M_3W_2), originally labeled (μ) by Arnfelt and Westgren, and Magneli and Westgran (M_7W_6).

It was also found that some portions of the existing equilibrium diagrams should be changed.

Universal-Cyclops Steel Corporation. WADC TR 57-426.

INVESTIGATION OF THE EFFECTS OF INCONGRUOUS ELEMENTS AND THE INTERACTION EFFECTS OF THESE ELEMENTS ON HIGH TEMPERATURE STRENGTH OF FE-CO-NI-CR ALLOYS. J. H. Sye, T. L. Robertshaw, F. M. Richmond. AF 33(616)-2777. December 1957. ASTIA Document No. AD 142237. PB 131614.

An investigation was made of the effect of five combinations of incongruous elements on the high temperature properties of vacuum melted alloys with a base composition of 60 atomic % Ni, 20 atomic % Cr, 10 atomic % Fe, 10 atomic % Co. Balanced experimental designs were used for each group.

In the group of alloys containing additions of Al, Ti and W to the base, a cast alloy had a stress-rupture life of 133.9 hours at 1800°F and 15,000 psi. A forged alloy of this series obtained a life of 149.1 hours at 1750°F and 15,000 psi.

The value and limitations of balanced experimental designs are discussed.

A metallographic study of cast and wrought alloys is appended.

Southern Research Institute. WADC TR 57-649, Part 1.

DETERMINATION OF THE MECHANICAL PROPERTIES OF AIRCRAFT-STRUCTURAL MATERIALS AT VERY HIGH TEMPERATURES AFTER RAPID HEATING. James B. Preston, William P. Roe, J. Robert Kattus. AF 33 (616)-3494. January 1958. ASTIA Document No. AD 142284. PB 131664.

Structural components in high-speed aircraft and in missiles must function for short periods of time at high temperatures and at high stresses. The heating time in conventional tests for design data usually exceed those that apply in high-speed aircraft and in missiles. A study was made, therefore, of the mechanical properties -- tensile, creep, fracture, compression, shear, and bending -- of several structural materials under conditions of moderate to rapid rates of heating and of loading.

The materials involved in this investigation included electrolytic-tough-pitch copper, oxygen-free high-conductivity copper, A-nickel, ingot iron, molybdenum, tantalum, Type GBH graphite, and composite OFHC copper plus 316 stainless steel sheet. The testing temperatures ranged from room temperature to the melting points of the metals and to 5750°F for the graphite. The total heating, holding, and loading times to failure ranged from about three seconds to about 30 minutes. Many of the tests were carried out both in air and in inert atmospheres.

It was found that the strength of the test metals decreased continuously with increasing temperatures. The graphite, however, increased in strength up to 4500°F and then decreased in strength with further increases in temperature. With constant load and constant temperature and with transient load or transient temperature, the inherent load-carrying capacity of the metals increased with decreasing times under load. The inherent strength of the graphite was not significantly affected by variations in time under load.

The mechanical properties of the copper and of the nickel test materials were not significantly affected by exposure to air at temperatures up to their melting points within the time limitations of the various tests. Because of oxidation, the apparent strength of the iron, molybdenum, tantalum, and graphite decreased with increasing exposure times to air at elevated temperatures.

P. R. Mallory & Co., Inc. WADC TR 57-535.

A STUDY OF THE POSSIBILITY OF REINFORCING HIGH-TEMPERATURE ALLOYS BY ADDITION OF REFRACTORY POWDERS. John D. Burney. AF 33(616)-2959. February 1958. ASTIA Document No. AD 150971. PB 131768.

This investigation was concerned with attempting to reinforce 80 Ni-20 Cr alloy by the addition of such refractory oxides as TiO_2 and Al_2O_3.

Several powder metallurgical fabrication techniques were investigated such as (1) pressing and sintering, (2) pressing and sintering followed by various working procedures, (3) internal oxidation and (4) liquid phase sintering.

In the press-sinter method of fabrication, the best stress-rupture characteristics at 1600°F were obtained with additions of 3.0 - 4.5% TiO_2. An addition of 4.5% TiO_2 resulted in a rupture time of 29.6 hours as compared to a rupture time of 1.0 hour obtained on bars containing no TiO_2.

Various hot and cold working procedures did not result in any improved stress-rupture properties.

The liquid phase sintering technique produced the best stress-rupture properties. Stress-rupture tests at 1800°F showed a stress of 6200 psi for an 80 Ni-20 Cr + 1.0% TiO_2+C material which was more than three fold better than wrought Nichrome V. Stress-rupture tests on the same materials at 1975°F showed the liquid phase material to have twice the strength of Nichrome V. The impact strengths varied from 10 to more than 50 inch pounds. Room temperature tensile strengths which were fairly consistent, averaged about 75,000 psi. The thermal shock properties were excellent and the oxidation resistance, although not quite as high as Nichrome V, was considered good.

Westinghouse Electric Corp. WADC TR 57-678. STUDIES AND COMPARISON OF THE PROPERTIES OF HIGH TEMPERATURE ALLOYS MELTED AND PRECISION CAST BOTH IN AIR AND IN VACUUM. Milo J. Stutzman. AF 33(616)-3468. March 1958. ASTIA Document No. AD 151035. PB 131807.

The objective of the research program for USAF Contract No. AF 33(616)-3468 was the investigation of the potentialities for improvement of the mechanical properties at elevated temperature of heat-resisting alloys by varied practices of melting and investment casting. Two cobalt and two nickel base alloys were prepared under the following casting conditions:

1. Stellite 31 was melted and investment cast in air and in vacuum, with an argon atmosphere for pouring.
2. He 1049 was melted and investment cast in air and in vacuum, with an argon atmosphere for pouring.
3. Utimet 500 was melted and investment cast in vacuum with and without additions of zirconium and boron.
4. Guy Alloy was melted and investment cast in vacuum and in argon.

The effects of melting and casting procedures upon the gas (hydrogen, oxygen, oxygen and nitrogen) contents, the tensile properties at room and elevated temperatures, the stress-rupture properties in the range of 1350 to 1800°F, and the oxidation resistance to air at constant temperature of 2000°F and intermittent exposure to air at room temperature and 2000°F were studied.

The data obtained on this program failed to support general conclusions applicable to the four alloys under investigation. Tests of specimens of one alloy failed to substantiate data obtained from other tests for related properties. The data must be evaluated for each alloy with close correlation to service conditions for design applications.

Ford Motor Company. WADC TR 57-298 Part 4. IRON-ALUMINUM ALLOY SYSTEMS. Part 4 - The Activities of Aluminum and Iron in Iron-Aluminum Melts at 1600°C. R. D. Pehlke. AF 33(600)-32448. April 1958. ASTIA Document No. AD 151102.

The activities of the components in molten iron-aluminum alloys at 1600°C have been calculated by utilizing the phase diagram. Consideration was made of the equilibria along the liquidus boundary of the intermetallic compound Fe_2Al_5 and known distribution data for aluminum in iron to determine the activity of aluminum in iron-aluminum melts at 1600°C. The activity of iron was then found by graphically integrating the Gibbs-Duhem relationship.

Ford Motor Company. WADC TR 57-298 Part 5. IRON-ALUMINUM ALLOY SYSTEMS. Part 5 - Some Investigations in Air-Melting and Application of Iron-Aluminum Alloys. William J. Zager. AF 33(600)-32448. April 1958. ASTIA Document No. AD 151103.

During the period April 1957 to September 1957, the Process Development Department of the Manufacturing Engineering Office of Ford Motor Company air-melted a series of heats of iron-aluminum alloys in the 6 to 10% aluminum range. The melting and deoxidation of these heats, as well as properties of the as-cast alloys are listed.

Further attempts to produce sound castings in sand molds are described.

Performance of iron-aluminum alloy fabricated articles in service is described.

Ford Motor Company. WADC TR 57-298 Part 6. IRON-ALUMINUM ALLOY SYSTEMS. Part 6 - Welding of Iron-Aluminum Alloys. R. Burthwick, S. Goodman. AF 33(600)-32448. April 1958. ASTIA Document No. AD 151104.

Supplementing a previous report, WADC Technical Report 57-298 Part 3 (AD-130825), additional mechanical test data were established for the effect of fusion welding on three different analyses of iron-aluminum alloy. The consumable electrode gas shielded arc process was briefly investigated. Additional fabrications were selected for evaluation of iron-aluminum material and work initiated.

Ford Motor Company. WADC TR 57-298 Part 7. IRON-ALUMINUM ALLOY SYSTEMS. Part 7 - Study of Magnetic Properties. H. Sato, A. Arrott. AF 33(600)-32448. April 1958. ASTIA Document No. AD 151105.

From a study of magnetic isotherms over the range of compositions from pure Fe to 40 atomic percent Al in Fe and over temperatures from 4°K to 300°K we have produced strong evidence for the existence of super-exchange interactions between iron atoms separated by aluminum atoms. The iron rich alloys show ferromagnetism in the usual way, but for concentrations approaching 40 atomic percent Al the alloys behave as typical anti-ferromagnetics with Neel points near liquid hydrogen temperatures. There is a transition range of composition in which the stable state just below the Curie temperature is ferromagnetic but at low temperatures is anti-ferromatnetic.

The large changes in magnetic properties occur for the ordered structures and in composition ranges where the number of nearest neighbor iron-iron interactions is changing rapidly with composition, but where the number of next nearest neighbor iron-iron interactions remain practically constant. These considerations lead to a model where only nearest neighbor direct positive exchange interactions between iron atoms and indirect negative exchange interactions between iron atoms separated by an aluminum atom are taken into account. The treatment by a Bragg-Williams type calculation leads to a qualitative understanding of the observed magnetic effects in the aluminum system including the differences between the disordered state and the two types of ordered states. The model also leads to a reinterpretation of neutron diffraction data.

The Babcock & Wilcox Company Research Center. WADC TR 58-28.

THE STRENGTHENING OF AUSTENITIC SOLID SOLUTIONS. F. Eberle, J. H. Hoke, E. J. Rozic, W. E. Leyda. AF 33(616)-2413 and AF 33(616)5208. April 1958. ASTIA Document No. Ad 151161. PB 131992. Order from OTS $1.75.

Among vacuum-melted wrought carbon-free alloys of Fe-Cr-Ni, Fe-Cr-Co, and Fe-Cr-Ni-Co, the compositions 10Fe-20Cr-70Ni and 20Cr-80Co have shown highest 100-hour rupture strength at 1800°F. Strengthening susceptibility sutdies with these two base compositions by solid solution hardening, complex carbide hardening, and intermetallic compound hardening revealed that the nickel base responds most effectively to intermetallic compound hardening with Ti and Al, less so to complex carbide hardening, and not at all to solid solution hardening. The cobalt base responds to all three methods of strengthening; however, it does not attain as high a level of rupture strength at 1800°F as the nickel-base alloys.

Westinghouse Electric Corp. WADC TR 57-344. DEVELOPMENT OF NIOBIUM-BASE ALLOYS. Richard T. Begley. AF 33(616)-3316. May 1958. ASTIA Document No. AD 155583. PB 151004. Order from OTS $2.50.

The flow and fracture characteristics of commercial purity powder metallurgy niobium were investigated in the range 250 to -196°C. Niobium was found to undergo a ductile-brittle transition in the range -125 to -196°C, and the transition temperature range of niobium was found to be less affected by the presence of interstitial impurities than many other body-centered cubic metals.

The creep-rupture properties of powder metallurgy niobium were investigated at 982 and 1093C (1800 and 2000°F), and the 100-hour rupture strength of commercial niobium in vacuum was determined to be significantly greater than unalloyed molybdenum. The creep-rupture results suggest that small quantities of gaseous contaminants may be responsible for the high strength of commercial niobium at elevated temperatures.

The oxidation behavior of niobium was investigated in the temperature range 350 to 700°C. At the higher temperatures, oxidation followed a linear rate law. Between 500 and 625°C, rate of oxidation was found to be nearly independent of temperature.

Oxygen and nitrogen contamination of welding atmospheres was studied to determine its effect on the weld properties of niobium. Nitrogen was established to be very detrimental to the mechanical properties of niobium welds.

High-purity niobium, having a hardness of less than 60 VPN, was produced by cage-zone refining techniques.

Cornell Aeronautical Laboratory, Inc. WADC TR 54-270 Part 3.

INVESTIGATION OF THE COMPRESSIVE, BEARING AND SHEAR CREEP-RUPTURE PROPERTIES OF AIRCRAFT STRUCTURAL METALS AND JOINTS AT ELEVATED TEMPERATURES. Luke A. Yerkovich. AF 33(616)-190. May 1958. ASTIA Document No. AD 151194. PB 131977.

The establishment of high-temperature creep and rupture properties of materials is a prerequisite for efficient design if exposure to elevated temperature in service is expected. These properties, which are generally determined from the conventional creep test, are not necessarily applicable if stress conditions other than tension are encountered. This project was initiated to supplement the usual tensile creep and rupture data. The high-temperature creep strengths of a number of structural aircraft alloys were determined when under the influence of compression, bearing, and shear stresses. Specifically, data of these types are required to formulate high temperature joint design criteria.

This report summarizes three years study on the creep behavior of fifteen sheet, plate, and bar alloys creep tested in tension, compression, bearing, and shear. In addition, correlations of tensile creep and rupture properties with compression, bearing and shear creep-rupture properties have been made and are presented in tabular form.

Cornell Aeronautical Laboratory, Inc. WADC TR 54-270 Part IV.

INVESTIGATION OF THE COMPRESSIVE, BEARING AND SHEAR CREEP-RUPTURE PROPERTIES OF AIRCRAFT STRUCTURAL METALS AND JOINTS AT ELEVATED TEMPERATURES. Luke A. Yerkovich. AF 33(616)-3456. May 1958. ASTIA Document No. AD 155570. PB 151445.

The establishment of the high-temperature mechanical properties of aircraft constructional materials is a prerequisite to efficient design when elevated temperature service is expected. These properties, which normally are determined from the conventional short-time tensile test and the tensile-creep test, are not necessarily applicable if stress conditions other than tension are encountered. The present program was initiated to examine the high-temperature strength characteristics of a number of aircraft structural alloys when subjected to a variety of stresses under both short and long time loading with the specific purpose of applying these high-temperature characteristics to riveted joint configurations.

This report summarizes in tabular form the high-temperature short-time strength properties of a number of test alloys in tension, compression, bearing, and shear. Data representing the bearing creep behavior of 2024-T3 aluminum alloy sheet for a number of variables associated with bearing are also included. The creep behavior of several selected joint designs undergoing bearing and shear creep are illustrated as time-deformation charts, and comparisons between the predicted and experimental performances are made.

Ford Motor Company. WADC TR 57-298 Part 8. IRON-ALUMINUM ALLOY SYSTEMS. Part 8 - Mechanical Properties of Fe-Al-Si Alloys at Room and Elevated Temperatures. D. J. Schmatz, V. F. Zackay. AF 33 (600)-32448. ASTIA Document No. AD 155659.

An investigation of the mechanical properties of iron-rich, iron-aluminum-silicon alloys was conducted. Silicon was used as the independent variable at three aluminum levels; 5, 7 and 9 weight percent. Silicon increased the strength at both room and elevated temperatures. Ductility was markedly decreased by silicon and interstitial elements, such as carbon. Silicon appeared to be more than additive to aluminum in improving the oxidation resistance. Room temperature electrical resistivity measurements on quenched and slow cooled alloys indicated silicon was additive to aluminum in initiating order. Silicon also raised the ordering temperature rapidly. A correlation between ordering temperature, as determined by electrical resistivity and creep rate was noted.

Ford Motor Company. WADC TR 57-298 Part 9. IRON-ALUMINUM ALLOY SYSTEMS. Part 9 - Some Investigations in Air-Melting and Application of Iron-Aluminum Alloy. William J. Zager. AF 33(600)-32448.

June 1958. ASTIA Document No. AD 155660.

During the period October 1, 1957 through March 31, 1958, the Process Development Department of the Manufacturing Engineering Office of Ford Motor Company air-melted several heats of iron-aluminum alloys in the 6 to 10% aluminum range. The melting and deoxidation of these heats, performed in induction furnaces are described. Mechanical properties obtained on cast tensile bars are listed.

Effect of cold deformation (compression) tests on one of the wrought alloys is described.

Performance of iron-aluminum alloy fabricated articles in service is described.

Ford Motor Company. WADC TR 57-298 Part 10. IRON-ALUMINUM ALLOY SYSTEMS. Part 10 - Welding of Iron-ALUMINUM ALLOYS. R. Brooks, A. Volio. AF 33(600)-32448. June 1958. ASTIA Document No. AD 155661.

The work conducted during this reporting period is a continuation of the investigations to develop both fusion and resistance welding processes for iron-aluminum alloy fabrication as reported under Document No. 151104. The limited work conducted using the tungsten arc gas shielded process further verified the need for pre and postweld heat treatment. An investigation was initiated to develop coated manual arc welding electrodes for welding iron-aluminum alloys. With aluminum added to the coating, it was possible to obtain a weld metal deposit with up to 10% aluminum content. Resistance spot and stitch welding schedules were developed for the material thicknesses to be used in fabricating the J-57 engine turbine exhaust cone. Two different approaches in making the cone body blank were tried to eliminate cracking during hot spinning of the cone detail without success. A two part blank welded together after spinning to form the complete cone body is now in process. A large carburizing box was fabricated and put in service. Severe cracking was experienced during welding and in service.

National Bureau of Standards.
WADC TR 58-164 Part I.
OXIDATION OF EXPERIMENTAL ALLOYS.
Joseph C. Richmond, H. Richard Thornton. AF 33(616)-56-19. June 1958. ASTIA Document No. AD 155688. PB 151264. Order from OTS $1.50.

A study was made of the oxidation resistance of ten high-temperature alloys; (1) aluminum-modified Nichrome V, (2) Nichrome V, (3) niobium-modified Nichrome V, (4) an iron-chromium-aluminum alloy, (5) Inconel 702, (6) Hastelloy R235, (7) Hastelloy W, (8) type 316 stainless steel, (9) Inconel X, and (10) Inconel. Tests included the measurement of the average depth of external oxidation and maximum depth of oxide penetration on specimens that had been subjected to stress in air at high temperature for 100 hours, and on specimens that had been subjected to stress in air under fluctuating-temperature conditions for an equivalent length of time, plus continuous recording of the weight change of specimens oxidized in air at high temperatures.

Horizons Inc. WADC TR 58-181.
A RESEARCH PROGRAM ON THE INVESTIGATION OF SEAL MATERIALS FOR HIGH TEMPERATURE APPLICATION. Raymond H. Baskey. AF 33(616)-3891. June 1958. ASTIA Document No. AD 155689. PB 151451.

Novel rotating seal materials for potential aircraft applications at high speed and high temperatures were developed and tested under conditions of no external lubrication. These materials were arrived at after a systematic study of the wear behavior of:

1. Pure refractory hard metals
2. Binary alloys of pure refractory hard metals, bonded with nickel.
3. Ternary alloys of pure refractory hard metals, bonded with nickel and infiltrated with silver.

Tests were conducted to determine the benefit derived from additions of nickel or silver to the refractory hard metals. The nickel acts as a tough, oxidation-resistant matrix. The silver was added to act as a lubricant as it softens at high temperatures. This aids the shearing action between mating surfaces and lowers the surface friction.

Two ternary alloys prepared by powder metallurgy techniques and consisting of high percentages by weight of a refractory hard metal, with nickel and silver additions, show superior wear qualities when run against either tool steel or Inconel. Tests were made at sliding speeds of 30,000 fpm and ambient temperatures up to 1350°F. The best alloys contained either tungsten boride (WB) or chromium nitride (CrN) as the hard refractory metal.

The WB alloy exhibited a constant wear rate at all temperatures against tool steel. The wear rate of the CrN alloy decreased at the higher temperatures.

All ternary alloys run against Inconel displayed a decrease in wear rate at ambient temperatures over 600°F. The wear decreased as a function of temperature for several ternary alloys against tool steel.

An unimpregnated graphite seal coated with an oxidation resistant layer of zirconium carbide showed improved wear properties over a regular graphite impregnated seal at an ambient temperature of 1050°F and sliding speeds of 14,000 fpm against stainless steel type 303.

The finest wear performance was attained when Kentanium K162B ran against Kentanium K162B at sliding speeds of 14,000 fpm in an ambient temperature of 1100°F.

It is hypothesized that the wear process is drastically influenced by the oxidation occuring on the rubbing surfaces and that with proper choice of materials, stable, complex oxides are formed which provide the correct solid lubricating film on each component and markedly lower the wear rate as the temperature is increased.

Crucible Steel Company of America.
WADC TR 57-242 Part II.
INVESTIGATION OF Fe-Mn-Cr-N-C SYSTEM FOR HEAT RESISTANCE AND OXIDATION RESISTANCE BETWEEN 1200°F and 2000°F. J. P. Tarwater, E. J. Dulis. AF 33(616)-3318. October 1958. ASTIA Document No. AD 203523. PB 151358. Order from OTS $2.50.

Austenitic Cr-Mn-C-N steels containing various combinations of V, Nb, W, and /or Mo had, after heat-treating, room- and elevated -temperature tensile properties and creep-rupture strengths that compared favorably with those of such superalloys as A286 and Inco 901.

Proper selection of composition and heat-treatment enabled development of adequate ductility in tension and creep rupture although a marked decrease in impact properties could not be avoided.

Calcium additions improved the creep rupture strength of Cr-Mn-C-N steels that did not contain additions of strengthening elements (V, Nb, Mo, and W). A silicon addition of about 2% to high-chromium Cr-Mn-C-N steels increased the oxidation resistance at 2000 and 2200°F so that these steels compared favorably with Type 310 stainless steel.

Both strengthened and unstrengthened austenitic Cr-Mn-C-N steels could be hot-rolled to sheets without the formation of edge cracks or surface defects, but rolling pressures higher than those for Type 310 stainless steel were required.

The depth of oxidation was found to increase with temperature and with stress. There was little effect of stress at stresses below that required to produce 1% extension in 100 hours. All of the alloys conformed reasonably well to the parabolic rate law in the weight-gain oxidation tests.

Crucible Steel Company of America.
WADC TR 57-343 Part II.
A STUDY OF THE METALLURGICAL PROPERTIES THAT ARE NECESSARY FOR SATISFACTORY BEARING PERFORMANCE AND THE DEVELOPMENT OF IMPROVED BEARING ALLOYS FOR SERVICE UP TO 1000°F. T. V. Philip, A. E. Nehrenberg, G. Steven. AF 33(616)-3318. October 1958. ASTIA Document No. AD 203524. PB 151415.

To develop a bearing steel for operating temperatures up to 1000°F, fifty-one experimental compositions were studied. This report describes the theoretical considerations which formed the basis of the formulation of these steels as well as the annealing cycle, austenitizing temperature survey, and the tempering survey. The results of the tempering studies are plotted as "master tempering curves".

From the study of the temper resistance, eight steels were selected for further evaluation tests. These tests consisted of (1) hot hardness determinations, (2) dimensional stability measurements, (3) compression tests at room temperature and elevated temperature, (4) oxidation resistance, and (5) hot oil corrosion resistance.

As a result of the foregoing tests, a steel of the following composition is recommended for use as a bearing steel for operating temperatures up to 1000°F:

C	Cr	V	W	Mo	Co
1.09	4.2	1.9	6.7	3.7	5.2

The steel should be heat-treated as follows: austenitize at 2225°F, oil quench, and temper for two consecutive 2-hour periods at 1050°F.

This work has not only developed a steel for elevated-temperature bearing applications but it has also clarified the effects of alloying elements on secondary hardness, hardness retention at elevated temperatures, and other properties desired of a high-temperature bearing steel.

Battelle Memorial Institute. WADC TR 58-299.
A STUDY OF REFRACTORY MATERIALS FOR SEAL AND BEARING APPLICATIONS IN AIRCRAFT ACCESSORY UNITS AND ROCKET MOTORS.
L. B. Sibley, C. M. Allen, W. J. Zielenbach, C. L. Peterson, W. H. Goldthwaite. AF 33(616)-3995. October 1958. ASTIA Document No. AD 203787. PB 151483. Order from OTS $1.50.

A number of ceramic, cermet, and high-temperature alloy materials have been evaluated for corrosion resistance and for friction and wear behavior in an oxidizing atmosphere from 1000 to 1800°F. Rubbing-wear experiments were conducted at 200 feet per second sliding speed with about 20-psi load pressure usually applied to the contact surfaces. Several material combinations involving an alumina-chromium [illegible], silicon carbide, alumina, and [illegible] titanium carbide-nickel-molybdenum cermet may be satisfactory for short-life rubbing-seal applications under these conditions. The applications of these materials are limited since they wear rapidly and exhibit erratic frictional behavior. Superficial surface cracking was observed on the wear surfaces of all specimens containing brittle ceramic phases except possibly silicon carbide. A wear-failure mechanism for refractory materials under sliding contact is postulated, and some correlation of the experimental results with conventional thermal-stress-resistance parameters is obtained.

Two rubbing-wear experiments were conducted in a high-temperature reducing atmosphere. The static corrosion resistance of several potential bearing and seal materials was determined in a nitric acid oxidizer used in some rocket-propellant pumps. Experimental ceramic and cermet materials were fabricated, and some of these specimens were evaluated in rubbing-wear experiments in the oxidizing atmosphere to study certain material-property effects on friction and wear behavior.

Stanford Research Institute. WADC TR 58-396.
DEVELOPMENT OF PROCEDURES AND TECHNIQUES FOR PREPARING BONDED DOUBLE LAYER TANTALUM-COPPER COMPOSITE PLATES. R. C. Bertossa, S. Rau. AF 33(616)-5215. November 1958. ASTIA Document No. AD 203901. PB 143623.

The purpose of this project was to develop a process for bonding tantalum sheet to OFHC copper. This was successfully accomplished both by casting molten copper directly into an annealed tantalum cone under controlled vacuum-inert gas atmospheres, and by using intermediate brazing alloys to produce high integrity, vacuum bonded joints between OFHC copper and annealed tantalum.

Metallurgical and microhardness evaluations reveal that sound, continuous bonds can be effected, with ductile interfaces, in joining OFHC copper to annealed tantalum by vacuum-inert gas techniques.

Physical tests on brazed copper tantalum composite plates show excellent bond strengths under multi-axial stresses encountered in 180° flat bends and in cold forming of flat composite plate into nose-cone shapes.

Elevated temperature tests on copper-tantalum clad nose-cones, both uncoated and metal or ceramic coated on the exposed tantalum surfaces, exhibited the ability of selected brazing alloys to maintain intimate contact between the copper and tantalum at temperatures up to the melting point of copper. The high thermal conductivity of the copper layer enabled it to absorb sufficient heat from the tantalum surface to allow the tantalum cone shell to contain the molton copper as long as two minutes at temperatures above 2000°C (measured optically on the exposed tantalum).

Battelle Memorial Institute. WADC TR 58-525.
INVESTIGATION OF THE PROPERTIES OF TANTALUM AND ITS ALLOYS. William D. Klopp, Fred R. Schwartzberg, Frank C. Holden, Chester T. Sims, H. Russell Ogden, Robert I. Jaffee. AF 33(616)-5668. November 1958. ASTIA Document No. AD 206073. PB 151562.

A survey of literature pertaining to tantalum and tantalum alloys has been carried out as part of the study of the properties of tantalum and its alloys. The results of that survey are reported in narrative style with references to published data. A bibliography of available publications is included. Emphasis was placed on subjects pertinent to the research being conducted on this project with a more general coverage of other subjects.

Ford Motor Company. WADC TR 57-298, Part II.
IRON-ALUMINUM ALLOY SYSTEMS. Part II -- Comparative Study of Oxidation Resistance in the Fe-Al System. Richard E. Heimerdinger. AF 33(600)-32448. November 1958. ASTIA Document No. AD 207907.

A study of the oxidation resistance of Fe-Al alloys with varying aluminum content has been made. Visual observations confirm the effect of aluminum (0-16 percent) on the oxidation resistance of Fe-Al alloys at temperatures to

2200°F and for periods of up to 500 hours.

Attempts to quantitatively determine weight gain of Fe-Al alloys were not successful. The lack of reproducible weight gain data is suggested to be a result of oxide seams and inclusions.

The addition of chromium was found to be beneficial only at the 7.50 percent chromium level. The apparent deleterious effects of chromium at levels both higher and lower than 7.50 percent is to be further investigated. An alloy containing 7.50 percent aluminum and 7.50 percent chromium is recommended for further study in view of its good oxidation resistance, corrosion resistance, and ductility.

Ford Motor Company. WADC TR 57-298, Part 12.
IRON-ALUMINUM ALLOY SYSTEMS. PART 12 -- Welding of Iron-Aluminum Alloys. R. Brooks, A. Volio. AF 33(600)-32448. November 1958. ASTIA Document No. AD 207908.

During the period from March 31, 1958, through September 30, 1958, further investigations were conducted for both fusion welding and resistance welding procedures for iron-aluminum alloys. Additional resistance spot and stitch welds were developed for heats E-1005 and E-1006. Fabrication of a J-57 Turbine Exhaust Cone made from iron-aluminum alloys was completed and submitted for testing. The forming and fusion welding processes used for this cone were described in WADC Technical Report 57-298, Part 10. Information on the resistance welding of the stiffener and flange to the cone body are contained herein.

Investigations were continued toward the development of coated iron-aluminum electrodes for manual arc welding to provide a weld metal deposit containing 7-9% aluminum content.

A program for investigating High Temperature Brazing was initiated for two reasons, (1) to develop a high strength brazing joint in iron-aluminum alloys suitable for use at elevated temperatures, and (2) to investigate brazing problems which would be likely in connection with aircraft fabrications.

General Electric Company. WADC TR 58-546.
EVALUATION OF ALLOYS FOR HIGH TEMPERATURE GEAR APPLICATIONS. E. G. Jackson, C. F. Muench, E. H. Rowe, E. H. Scott. AF 33(616)-5266. December 1958. ASTIA Document No. AD 206666. PB 151625.

The selection of materials for use in high mach number aircraft gears requires consideration of high temperature physical, mechanical and chemical properties, and of rubbing compatibility. This program was designed to obtain bench test data on ten different materials for application as gears at operating temperatures to 700°F. Oils representing two important classes, silicone and mineral, were chosen for scoring tests.

High temperature data were obtained on hardness, dimensional stability, tensile, impact and tooth fatigue strengths, corrosion and oxidation, and on surface scoring resistance.

The most fatigue resistant material was Modified M50, Carburized, while Matrix M2 was the best of the through-hardened steels. In score resistance, the best material depended on the oil; L.P.D. (Vacuum), Nitrided was the best with silicone, but M1 rose from second place with silicone to first with mineral oil. Choice of material for any application must depend on whether tooth breakage or scoring is the limiting factor, however, the L.P.D. (Vacuum) Nitrided is the best compromise since it ranked high in all tests.

Cornell Aeronautical Laboratory, Inc. WADC TR 54-270, Part V.
INVESTIGATION OF THE COMPRESSIVE, BEARING, AND SHEAR CREEP-RUPTURE PROPERTIES OF AIRCRAFT STRUCTURAL METAL AND JOINTS AT ELEVATED TEMPERATURES. Luke A. Yerkovich. AF 33(616)-3456. December 1958. ASTIA Document No. AD 206661. PB 151561. Order from OTS $2.00.

The establishment of the high-temperature mechanical properties of aircraft structural materials is a prerequisite to efficient design when elevated temperature service is expected. Normally these properties are determined from the conventional short-time tensile and creep-rupture tests and as such are not necessarily applicable for stress conditions other than tension. The present program was conducted to examine the high temperature strength and deformation characteristics of two high-strength airframe alloys when subjected to a variety of stresses under both short and long time loading. Specifically the object of the program was to determine the high-temperature tension, compression, bearing and shear properties of selected airframe alloys with the ultimate purpose of correlating tension behavior with behavior under the various other types of loads and applying these basic data to predict the behavior of riveted joints undergoing creep deformation in tension, bearing, and shear.

This report summarizes in tabular and chart form the high temperature properties of Ph15-7 Mo stainless steel and 6Al-4V titanium alloy in tension, compression, bearing, and shear. In addition correlations of the tensile creep-rupture properties with corresponding compression, bearing, and shear creep-rupture properties are presented.

The creep-rupture characteristics of doubler type riveted joints, which represent single units of a multiple riveted assembly, prepared from the test alloys are presented herein. Correlations between measured joint creep-rupture and predicted joint creep-rupture are also included.

Firth Sterling, Inc. WADC TR 58-432.
AN INVESTIGATION OF THE MECHANICAL PROPERTIES OF CERMETS AS RELATED TO THE MICROSTRUCTURE. Ira Binder, Robert Steinitz. AF 33(616)-5084. January 1959. ASTIA Document No. AD 208148. PB 151722.

A summary of cermet literature, especially that pertaining to microstructure, was made. Using 60 TiC-40 Ni as the test material, seven different test groups were formulated, comprising changes in original particle size, processing procedure, and controlled binder addition. Each test group was heated in seven different fashions.

Each test batch so obtained was tested for physical properties and its microstructure was investigated. The microstructures were correlated with changes in physical properties.

National Bureau of Standards. WADC TR 58-164. Part II.
OXIDATION OF EXPERIMENTAL ALLOYS. Joseph C. Richmond, H. Richard Thornton. AF 33(616)-56-19. February 1959. ASTIA Document No. AD 209379. PB 151741. Order from OTS $0.50.

A study was made of the oxidation resistance of five newly-developed high-temperature alloys: two nickel-molybdenum-chromium alloys and three iron-chromium-aluminum alloys. Tests included the measurement of the average depth of external oxidation and maximum depth of oxide penetration on specimens that had been subjected to stress in air at high temperature for 100 hours, and on specimens that had been subjected to stress

in air under fluctuating-temperature conditions for an equivalent length of time. The weight change of specimens oxidized in air at high temperatures was continuously recorded for periods up to 100 hours.

The depth of oxide penetration was found to increase with temperature and with stress. There was little effect of stress at stresses below that required to produce one percent extension in 100 hours. All of the alloys conformed reasonably well to the parabolic rate law in the weight-gain oxidation tests.

Westinghouse Electric Corporation. WADC TR 57-344, Part II.
DEVELOPMENT OF NIOBIUM-BASE ALLOYS. Richard T. Begley. AF 33(616)-3316. March 1959. ASTIA Document No. AD 210258. PB 151739. Order from OTS $3.00.

Flow and fracture studies indicate that the ductile to brittle transition of niobium is little affected by oxygen content in the range 0.01 to 0.1% O_2. The impact transition range of electron-beam melted niobium was found to be considerably below that of powder metallurgy niobium of somewhat higher oxygen concentration.

Strain-hardening and strain aging in niobium were studied. An activation energy of 27,100 cal/mol was determined for strain aging in niobium. The recrystallization behavior of electron-beam melted niobium was studied in detail.

Creep-rupture data were obtained on electron-beam melted niobium at (871°C) 1600°F and (982°C) 1800°F. The electron-beam material had much lower rupture strength than powder metallurgy niobium containing 0.6% Zr tested previously. The effect of temperature on the modulus of elasticity of niobium and tantalum was determined in the range 25°C to 900°C.

Studies of the thermodynamics of niobium oxides and the kinetics of Nb-water vapor reaction were carried out. The thermodynamic functions obtained in this study for the formation of Nb_2O_5 from Nb_2O_4 are in excellent agreement with calculated values.

A selection of the most promising weldable niobium-base alloys was made on the basis of available physical and mechanical property data.

Niobium of low oxygen and nitrogen content was produced by cage-zone and floating zone melting techniques. Evaluation of material prepared by electron-beam melting and vacuum arc melting was carried out.

Battelle Memorial Institute. WADC TR 58-682.
DEVELOPMENT OF TESTING PROCEDURES AND EVALUATION OF REFRACTORY MATERIALS. John M. Allen, James L. Harp. AF 33(616)-5301. March 1959. ASTIA Document No. AD 211766.

In this work specimens of several materials, both with and without protective coatings, were subjected to severe heat pulses to determine their capacity as a heat sink. Results were compared on the basis of the amount of heat a specimen could absorb without experiencing surface failure by melting or chemical reaction. The heat pulses were imposed by placing the materials in the throat of an H_2-O_2 rocket motor and operating the motor in such a manner that the heat flux increased to a maximum in 12 seconds and decreased during the next 12 seconds. By suitable calibration, the instantaneous applied heat flux and the integrated heat impulse were determined. It was found that 1-inch-thick copper can absorb an integrated heat impulse of 12,000 Btu/(ft^2) in a 24-second period when the maximum heat flux at the midpoint of the pulse is 822 Btu/(ft^2) (sec). Similarly, nickel can absorb 7500 Btu/(ft^2) with a maximum flux of 570 Btu/(ft^2) (sec). Graphite protected by a coating of silicon carbide formed in place can absorb approximately 9000 Btu/(ft^2) in a strongly oxidizing atmosphere with a peak heat flux of 600 Btu/(ft^2) (sec) before the protective coating is destroyed. More severe exposure caused drastic failure of the surface of each of these materials.

The Carborundum Company. WADC TR 59-10, Part I.
DEVELOPMENT AND EVALUATION OF MATERIALS FOR HIGH TEMPERATURE APPLICATIONS. Charles E. Shulze, Frank A. Saulino, William T. Adams, Gustave E. Mangsen, James C. Andersen, Howard W. Emmons. AF 33(616)-5542. May 1959. ASTIA Document No. AD 214427. PB 151968.

Various preparational techniques have been used to fabricate graphite-based bodies which have potential resistance to high velocity, high temperature air. Materials have been evaluated in a high intensity arc testing facility. Results are reported herein.

At least ten compositions have been developed which are superior to either AGKSP graphite or to a graphite-silicon carbide body on the bases of tests imposed.

Ford Motor Company. WADC TR 57-298, Part 13.
IRON-ALUMINUM ALLOY SYSTEMS. PART 13 -- Development of Creep Resisting Compositions. Roger H. Richman. AF 33(600)-32448. May 1959. ASTIA Document No. AD 213472.

Utilization of oxidation-resistant iron--7 percent aluminum as a basis for creep-resisting high temperature materials was the objective of the research described in this report. Data are presented to illustrate the effects of 1 to 4 percent molybdenum and 1/2 to 1 percent titanium upon the mechanical and metallurgical properties of iron--7 aluminum. All the compositions except those containing a nominal 0.25-percent silicon demonstrated good room temperature mechanical properties, after oil-quenching or air cooling from the recrystallization anneal, but only alloys of 3 and 4 percent molybdenum and 1/2 to 1 percent titanium possessed promising elevated temperature properties. From correlation of age-hardening studies with stress-rupture test results it was concluded that molybdenum and titanium served primarily as solid solution strengtheners. Suggestions for the modification of the composition to improve the high temperature performance are presented.

Ford Motor Company. WADC TR 57-298, Part 14.
IRON-ALUMINUM ALLOY SYSTEMS. PART 14 -- Welding of Iron-Aluminum Alloys. R. Brooks, A. Volio. AF 33(600)-32448. May 1959. ASTIA Document No. Ad 213473.

During the period from October 1, 1958 to January 31, 1959, investigations were continued to develop fusion welding procedures for iron-aluminum alloys. Fabrication of a furnace hearth and heat-treatment box made from iron-aluminum alloys was completed and they were submitted for service. The manufacturing and fusion welding processes used for these applications are described herein.

A J-57 Turbine Exhaust Cone of iron-aluminum alloy that was submitted for testing was returned to the Welding Development Department. This cone was damaged during endurance testing. Metallographic examination and mechanical testing were conducted on material from the damaged cone.

Climax Molybdenum Co. of Michigan. WADC TR 59-280.

DEVELOPMENT OF MOLYBDENUM-BASE ALLOYS. M. Semchyshen, Gordon D. McArdle, Robert Q. Barr. AF 33(616)-5447. October 1959. PB 161413. Order from OTS $2.75.

Some significant advancements have been made in the development of high strength, high recrystallization temperature molybdenum-base alloys. Of the new alloys developed in the course of this investigation, the following four were of particular interest, and represent the strongest known metallic materials for service at 1800° to 2400°F:

1. Mo + 1.45% Nb + 0.25% C
2. Mo + 3.20% Ti + 0.50% C
3. Mo + 1.27% Ti + 0.29% Zr + 0.30% C
4. Mo + 25.1% W + 0.11% Zr + 0.051% C

An ultimate recrystallization temperature in excess of 3200°F was displayed by the alloy designated as No. 3 above. Alloy No. 1 above had ultimate strengths in tension of 155,600 psi at room temperature and 101,300 psi at 1800°F. The tensile strength of Alloy No. 4 at 2400°F was 73,500 psi. The strength values noted here were significantly higher than had previously been recorded for molybdenum-base alloys. Alloy No. 1 had a 100-hour creep-rupture strength of 80,000 psi at 1800°F, and Alloy No. 3 a 32,700 psi at 2400°F.

Nominally 1/16-inch-thick molybdenum-base sheets containing alloy additions of (1) 0.5% titanium, (2) 0.05% zirconium, and (3) 0.5% titanium + 0.07% zirconium exhibited recrystallization temperatures comparable to those of similarly alloyed wrought (1/2 to 5/8 inches diameter) bar stock. Annealing temperatures sufficient to recrystallize the core of the alloy sheets were inadequate to recrystallize the extreme surface layers. The case effect was attributed to an increased level of contamination at the surface of the sheet resulting from rolling and annealing steps. Tensile strengths and elongation values obtained for the alloy sheets tended to be lower than those of wrought bar stock of similar compositions. Bendability of the sheet materials was substantially improved when the extreme surface layers were removed by chemical milling.

National Bureau of Standards. WADC TR 59-314. PREPARATION OF HIGH PURITY W, Mo, Ta, Cb, and Zr. George A. Moore, L. L. Wyman. AF 33(616)-58-11. October 1959. PB 161365. Order from OTS $0.50.

This project has been undertaken to prepare refractory metals of the highest possible purity in massive forms suitable for spectrochemical standards and for other essential applications. Experiments were made leading toward the preparation of purified volatile halides, taking advantage of fractional distillation and ion exchange reactions. An apparatus for the formation of refractory metal rods by the thermal decomposition of purified volatile halides, taking advantage of fractional distillation and ion exchange reactions. An apparatus for the formation of refractory metal rods by the thermal decomposition of purified halides was completed. A large zone melting unit for the final purification of these rods was approaching completion at the end of the project year. In this unit electric bombardment heating is provided for the most refractory metals and an RF heating unit is provided for zone melting larger quantities of the less refractory metals.

Armour Research Foundation. WADC TR 59-483. PHASE RELATIONSHIPS IN SELECTED BINARY AND TERNARY VANADIUM-BASE ALLOYS SYSTEMS. S. A. Komjathy, R. H. Read, W. Rostoker. AF 33(616)-5771. January 1960. PB 161816. Order from OTS $2.00.

In order to obtain information for the development of vanadium-base alloys, binary systems (V-La, V-Ce, V-Pr, V-Nd, V-Gd, V-Sn, V-Hf, V-Th, V-W, and V-Re) and ternary systems (V-Ti-Nb, V-Ti-Mo, V-Ti-Ta, V-Ti-W, V-Ti-O, V-Ti-Si, and V-Ti-Be) were investigated for:

1. The extent of the vanadium-rich solid solution between 800° and 1500°,
2. the character of the second phase in equilibrium with the solid solution, and,
3. the possible melt reaction.

Metallographic examination, X-ray powder diffraction analysis, incipient melting point determination, differential thermal analysis, and micro-hardness tests were used as experimental technique.

The experimental results are illustrated by partial phase diagrams, where it was necessary. In some cases work was done on compositions with less than 50 w/o V to resolve certain problems.

Precipitation kinetics studies were carried out on selected alloy compositions in ternary systems V-Ti-O, V-Ti-Si, and V-Ti-Be. The age hardening of the alloys at 550°, 650° and 750°C was followed by routine Vickers hardness tests.

Westinghouse Electric Corporation. WADC TR 59-575.

OXIDATION OF TUNGSTEN AND TUNGSTEN BASED ALLOYS. E. A. Gulbransen, K. F. Andrew, P. E. Blackburn, T. P. Copan, A. Merlin. AF 33(616)-5770. February 1960. PB 161718. Order from OTS $2.25.

This report describes work on a fundamental study of the oxidation of tungsten and its alloys.

To understand the tungsten-oxygen system thermodynamic measurements were made on the oxides WO_3, $WO_{2.9}$ and $WO_{2.72}$. The W_3O_9 (g) pressure over the solid oxide phases from WO_3 to WO_9 was measured and the homogeneity range of the several oxide phases determined.

Kinetic studies were made on sheet and wire specimens from 500° to 1300°C and for a broad pressure range. Crystal structure studies and photographic studies were made on the oxide scales.

All of the results suggest that the mechanism of oxidation is very complex. At 5000°C and lower the reaction is probably diffusion controlled. Above 600°C localized edge type of reaction adds a complication. Above 1200°C the oxidation reaction is similar to the combustion of graphite. The rate of oxidation is limited by the access of oxygen to the surface.

University of Michigan Research Institute. WADC TR 59-606.

STUDIES OF HEAT-RESISTANT ALLOYS. A. Philip Coldren, Jerry E. White, Ronald K. Bowen, James W. Freeman. AF 33(616)-5466. February 1960. PB 161667. Order from OTS $2.50.

Results are reported for an investigation that was carried out to show the effect of hot working on the structure and creep-rupture properties of heat resistant alloys. Three types of materials were studied a commercially-pure metal ("A" Nickel), a precipitation-strengthened, austenitic alloy (A-286), and a low-alloy, ferritic steel ("17-22-A"V").

In the case of "A" Nickel Rupture tested at 1100°F, near the static recrystallization temperature, the studies indicated that substructures were the major factor affecting strength and that internal lattice strains as measured by hardness were relatively unimportant. At 800°F, the creep resistance of "A" Nickel appeared to depend on internal strains and substructures more or less equally.

For A-286 Alloy the rolling temperature influenced rupture properties at 1200°F and 1350°F more than the amount of reduction. Rough correlations between strength and grain size were found, but it was not clear whether solutions of excess phases or grain size per se was the strength-determining factor. A small effect of G phase in the grain boundaries was observed.

The rolling experiments on "17-22-A"V" Steel demonstrated that beneficial effects on both rupture strength and ductility at 1100°F can be produced by working thoroughly-solutioned austenite at temperatures too low for simultaneous recrystallization. The results suggested that the more severely strained the austenite the greater the improvement in properties.

A second phase of the investigation dealt with the effects of strain-aging-type phenomena on strength at high temperatures. The results of constant-strain-rate tension tests on 1020 carbon steels and A-286 Alloy suggested that forces of chemical attraction between nitrogen atoms and silicon, aluminum, or titanium atoms stabilize Cottrell-type atmospheres, permitting them to be effective at higher-than-normal temperatures.

Battelle Memorial Institute. WADD TR 59-13. INVESTIGATION OF THE PROPERTIES OF TANTALUM AND ITS ALLOYS. F. F. Schmidt, W. D. Klopp, W. M. Albrecht, F. C. Holden, H. R. Ogden, R. I. Jaffee. AF 33(616)-5668. March 1960. PB 161758. Order from OTS $2.50.

The reactions of unalloyed tantalum with air, nitrogen, and oxygen were studied. Vacuum sintering of high-purity and high-impurity-content tantalum powders was investigated to determine the conditions required for purification. The effects of alloying on the oxidation behavior of tantalum were determined. Several alloying elements were found to be effective in reducing both scaling and contamination. Interstitials were studied for their effects on mechanical behavior at low and elevated temperatures. Screening studies were conducted on the effects of substitutional alloying on mechanical properties at room temperature and at 2200°F. References are included.

Armour Research Foundation. WADC TR 59-492. A STUDY OF TERNARY PHASE DIAGRAMS OF TUNGSTEN AND TANTALUM. W. Rostoker. AF 33(616)-5678. March 1960. PB 161709. Order from OTS $2.25.

Phase relationships in 36 ternary refractory metal systems were investigated. Some 28 related binary systems were first established by reviewing the literature and doing whatever supplementary experimental work necessary to assure a valid basis for the ternary studies.

Nearly 200 ternary alloy compositions were prepared from high-purity alloying ingredients by nonconsumable electrode arc-melting procedures. These alloys were studied metallographically in the as-cast condition as well as after annealing at and quenching from 1500° and 1000°C.

The main purpose of this effort was to scan the solid solubility limits of ternary systems based on tungsten and/or tantalum and involving the following metals: molybdenum, niobium, vanadium, chromium, osmium, and rhenium. The diagrams presented herein are intended to serve as a foundation for future alloy development. Because none of the systems presented is completely detailed, definitive work can and should be done as the specific need arises.

Armour Research Foundation. WADC TR 59-19. DEVELOPMENT AND EVALUATION OF HIGH-TEMPERATURE TUNGSTEN ALLOYS. F. C. Holtz, R. J. Van Thyne. AF 33(616)-5218. April 1960.

Alloys containing a minimum of 90 wt % tungsten were developed for use at temperatures up to 2000°F. Compositions based on the W-Ni-Fe system were prepared by cold-pressing blended metal powders, followed by liquid-phase sintering in hydrogen. The sintered products consisted of rounded, tungsten-rich grains in a W-Ni-Fe solid solution matrix. Ternary alloys containing up to 98 wt % tungsten were investigated; materials at the 95 wt % tungsten level were very ductile and could be rolled at room temperature. The hardness of cold-rolled specimens decreased upon annealing at temperatures above 1400°F. Maximum tensile strength values of 34,000 psi at 2000°F occurred at the 97 wt % tungsten level. Tensile elongations decreased above 1000°F, and these ternary materials had a 100-hour rupture life at a stress level of about 2500 psi at 2000°F.

The effects of tungsten contents, nickel-to-iron ratios, and quaternary additions on properties of W-Ni-Fe alloys were studied at room temperature and at 2000°F. Increasing amounts of tungsten resulted in lower ductility and slightly higher strength; a slight improvement in stress rupture life at 2000°F was noted. Optimum nickel-to-iron ratios were dependent on the tungsten levels. Proportionally higher iron contents increased tensile strength and decreased elongation at elevated temperature. The elements Al, Cr, Mo, Nb, Ta, and Ti were added to a 90W-Ni-Fe-base. Room temperature strength and ductility of this base were lowered by 2.5 wt % of Cr, Nb, and Ta, and by 0.5 wt % of Al and Ti. Moderate improvements in elevated temperature properties were noted in alloys containing 2.5 wt % Cr, Mo, and Ta. Oxidation resistance, slightly inferior to that of unalloyed tungsten, was not materially affected by the small quaternary additions.

Blended W-Ni-Fe powders were hot-pressed in graphite dies, using induction heating. The compacts were relatively fine-grained and had a thin carbide layer on the surfaces. Densities were usually above theoretical values due to squeeze-out of the very fluid matrix phase. Ingots of unalloyed tungsten and a 90W-10Nb alloy prepared by consumable-electrode arc-casting had a coarse, columnar grain structure and were not amenable to working.

Westinghouse Lamp Division. WADD TR 60-37. PHYSICAL METALLURGY OF TUNGSTEN AND TUNGSTEN BASE ALLOYS. Russell H. Atkinson. AF 33(616)-5632. May 1960. PB 161978. Order from OTS $1.00.

To establish a comparative standard for evaluation of changes in properties brought about by the addition of alloying constituents to tungsten, base line data and fundamental physical metallurgical information on tungsten of various purity levels were obtained and studied.

Base line data on high purity and commercial powder metallurgy tungsten have revealed differences in tensile transition and recrystallization temperatures believed attributable to variations in metallic impurity concentrations. High temperature (to 2700°F) tensile properties of

these materials are little affected by differences in levels of trace impurities except for the possibility that observed anomalous/and poorly reproducible creep rupture behavior is impurity-induced.

The tensile transition temperature of electron beam zone melted single crystal tungsten lies between -196° and -107°C, the main influence being surface condition. Deformation of these crystals is accompanied by twinning, and observed low temperature yield point phenomena are tentatively attributed to hydrogen.

Ploycrystalline tungsten exhibits several low magnitude internal friction peaks of undetermined origin in the 20°-800°C region. Annealed single crystals show no such behavior.

Purposeful introduction of carbon by carbiding and annealing and of oxygen by either direct gas-metal reaction or soaking in tungstic oxide at elevated temperatures does not produce homogeneous distribution of these elements in concentrations above 20-40 weight parts per million.

Addition of thoria as a dispersed second phase to tungsten raises the recrystallization temperature of tungsten and thus improves the high temperature (2700°F) yield strength by a factor of 2-3.5 and the 100 hour creep rupture strength by a factor of about 2. Significant improvement over these values is indicated for the addition of tantalum carbide in place of the thoria. Other dispersed second phase systems employing ZrO_2, B_4C, NbC, SiO_2, or WB have been studied. Data are incomplete.

Battelle Memorial Institute. WADD TR 60-155.
DEVELOPMENT OF METHODS AND INSTRUMENTS FOR MECHANICAL EVALUATION OF REFRACTORY MATERIALS AT VERY HIGH TEMPERATURES. D. H. Fisher, D. N. Gideon, G. M. McClure, H. J. Grover, R. L. Carlson, G. K. Manning. AF 33(616)-6155. June 1960. PB 171012. Order from OTS $2.25.

A mechanical-testing system has been established which is capable of providing tensile and compressive stress-strain data and shear-strength data up to 4000°F in vacuum. The results of an evaluation of the system using a molybdenum-0.5 percent titanium alloy in the bar form are presented. Tension, compression, shear and bearing tests were conducted at temperatures up to 3500°F. A detailed description of the testing system and specimen designs is presented. An optical strain-measurement system which permits displacements to be measured directly in the gage section is described.

The state-of-the-art report on the development of equipment and techniques of mechanical-properties measurement is based on a survey of literature and discussions with researchers at Battelle and other laboratories. With the objective of developing equipment and techniques for use to 6000°F or higher, problems to be solved in order that accurate data will be obtained are considered. Recommendations are made concerning furnace design, temperature and strain measurement, and specimen, grip, and loading design. In particular, the probable advantages of graphite heaters coated with tantalum carbide are pointed out; plans for experimental investigations and a description of apparatus built to study carbide coatings are presented. The further investigation of two-color pyrometry was also recommended.

Brush Beryllium Company. WADC TR 59-29, Part II.
AN INVESTIGATION OF INTERMETALLIC COMPOUNDS FOR VERY HIGH TEMPERATURE APPLICATIONS. Robert M. Paine, A. James Stonehouse, Wallace W. Beaver. AF 33(616)-56-12. July 1960. PB 161683. Order from OTS $3.50.

The preparation, fabrication, and properties of intermetallic compounds under development for service in the temperature range of 2300° to 3000°F are described. The compounds, principally beryllides, have exhibited strengths as measured by the modulus-of-rupture test of up to 40,000 psi at 2700°F and resistance to oxidation for 100 hours to 2800°F and have indicated good thermal conductivity.

The compounds were prepared by solid-state reactions and fabricated chiefly by hot-pressing techniques. Oxidation tests were made in dry air at 2500° to 3000°F and in moist air (57°F dew point) at 2300° to 2500°F. Transverse-rupture tests were carried out at 2300°, 2500° and 2750°F. Room temperature hardness data and melting points are reported for some compounds. Thermal-expansion and thermal-conductivity data for selected compounds are also presented.

The intermetallic compounds included in the investigations were $NbAl_3$, $TaAl_3$, $CrBe_2$, $MoBe_{12}$, Nb_2Be_{17}, $NbBe_{12}$, $TaBe_2$, Ta_2Be_{17}, $TaBe_{12}$, $TiBe_2$, $TiBe_{12}$, $ZrBe_{13}$, Cr_3Si, Ti_5Si_3, TiSi, and $TiSi_2$, with $CrBe_2$, $TaBe_2$, $TiBe_2$ and the silicides receiving only a very limited effort (chiefly oxidation tests). The more promising intermetallic compounds for high-temperature applications are concluded to be $ZrBe_{13}$, Nb_2Be_{17}, $NbBe_{12}$, Ta_2Be_{17}, and $TaBe_{12}$.

Curtiss-Wright Corporation. WADD TR 60-240.
RESEARCH ON PROPERTIES OF HIGH STRENGTH MATERIALS SUITABLE FOR HIGH TEMPERATURE APPLICATIONS. Harold N. Cummings, Foster B. Stulen, William C. Schulte. AF 33(616)-6552. July 1960. PB 171060. Order from OTS $1.75.

1. Bars of iron-molybdenum alloys, intended for study as to suitability for ball bearings at temperatures up to 1000°F, were found to be so non-homogeneous and brittle that specimens could not be machined from them.

2. High temperature torsion tests of relaxation were made to study the suitability of two alloys, M-1 tool steel and Inconel X, for springs at 1000°F, and of two alloys, Waspalloy and Udimet 500, for springs at 1500°F. Alternating torsion of ± 10 ksi, superimposed on an initial steady stress of 25 ksi, was applied to specimens of each alloy. Inconel X lost about 10 to 15 percent of the steady load in about 100 hours. The three other alloys relaxed two to three times as much when tested at the respective temperature mentioned above.

Manufacturing Laboratories, Inc. WADD TR 60-343.
INVESTIGATION OF DIFFUSION BARRIERS FOR REFRACTORY METALS. E. M. Passmore, J. E. Boyd, L. P. Neal, C. A. Andersson, B. S. Lemment. AF 33(616)-6354. August 1960. ASTIA Document No. AD 246559. PB 171400. Order from OTS $1.50.

Twenty-three base-barrier metal combinations were screened by annealing diffusion couples at 1700 ± 15°C. Measurements indicating the extent of interdiffusion between barrier and base metals were made by metallographic examination, microhardness tests, and electron microbeam analysis.

From these measurements, it is concluded that Hf and Ir are the most promising barriers for W at 1700°C, with V as a third choice. It appears to be the best choice for Ta and Mo. No recommendations could be made for Cb base metal, primarily because of melting at the screening temperature.

A comparison of interdiffusion behavior with base-barrier phase relations indicates no correlation with either

solid solubility or presence of intermediate phases. The melting points of both barrier and base appear to be the most important factors affecting the extent of interdiffusion.

Manufacturing Laboratories, Inc. WADD TR 60-566.

SURFACE TENSION OF REFRACTORY METALS. S Victor Radcliffe, H. Udin. AF 33(616)-6269. December 1960. ASTIA Document No. AD 258567. Order from OTS $1.25.

An electron-bombardment heating technique has been applied to the measurement of the surface tension of solid niobium by a modification of the Udin method. The value obtained is 2100 dynes. cm^{-1} at 2250°C in a vacuum of approximately 10^{-5} mm. This result is consistent with a theoretical estimate and provides support for Taylor's estimates of the surface tension of the refractory metals.

From measurements of the dihedral angle of grain boundaries formed in niobium by thermal etching, the interfacial energy (γ') was computed to be 0.36 of the surface energy.

An electrical-resistance heating technique has been applied to the measurement of the surface tension of solid tungsten, molybdenum and niobium by a modification of the Udin method. The method in this form was found to be unsuitable for these metals.

General Electric Company. WADD TR 60-646, Part I.

CARBONIZATION OF PLASTICS AND REFRACTORY MATERIALS RESEARCH, PART I. J. A. Coffman, G. M. Kibler, T. R. Riethof, A. A. Watts. AF 33(616)-6841. February 1961. ASTIA Document No. AD 257747. Order from OTS $2.25.

This report presents details of the conduct of, and the results obtained from, experimental investigations concerned with (1) the rate and mechanism of the carbonization of plastics; (2) the measurement of the vapor pressure of refractory materials; and (3) the determination of the spectral emissivities of refractory materials. These investigations are continuing.

Three complementary experimental techniques were, and are being used to study the carbonization of plastics. These are (1) thermogravimetric analysis, which gives an over-all picture of reaction rates under slow heating; (2) fluid-bed carbonization of powdered resin, which provides insight into the mechanism; and (3) arc image furnace experiments in which the plastics are exposed to temperatures approaching those of actual ablation applications.

Vapor pressure studies are being conducted using the Langmuir evaporation technologies. In addition matrix-isolation techniques and resonance-line absorption spectroscopy are being applied to the study of vapor species. Materials studied are tantalum carbide, zirconium carbide and thoria.

Normal spectral emissivities of tungsten, tantalum carbide and zirconium carbide have been measured between 1800°K and 3000°K using an emissometer designed and built for this purpose.

Results of these studies are given and discussed.

Crucible Steel Company of America. WADD TR 60-405.

A STUDY ON CONTROLLING HIGH TEMPERATURE OXIDATION OF VANADIUM BASE ALLOYS. James D. Klicker, Howard B. Bomberger. AF 33(616)-6113. February 1961. ASTIA Document No. AD 258378. Order from OTS $2.25.

The primary objective of this contract was to study means for inhibiting the rapid oxidation of vanadium alloys.

The characteristics of 240 oxide mixtures were studied first to determine those elements whose oxides form refractory oxides with vanadium oxide. Melting points of these mixtures contained one or more of the following oxides: Al_2O_3, Cb_2O_3, CrO_3, NiO_2, TiO_2, Y_2O_3, ZrO_2.

Twenty-eight of the more refractory oxides were then applied to vanadium sheet but they appeared to offer little protection as coatings.

Seventy laboratory-size alloy ingots were cast, processed, and tested for oxidation resistance. Vanadium alloys containing titanium, aluminum and nickel yielded the most refractory scales; and, oxide melting points as high as 1950°F were observed. Some of the more promising compositions could be hot worked by rolling and extruding.

This study indicates that useful oxidation resistance may be possible through further alloy development.

Nonferrous (Aluminum) Metals

TR 4531.

PROPERTIES OF X74S ALUMINUM ALLOY SHEET. James W. Poynter. May 1940.

TR 4547.

EFFECTS OF ARTIFICIAL AGING ON THE PROPERTIES OF ALUMINUM ALLOY TUBING. D. A. Shinn, W. G. Ramke. June 1940.

TR 4551.

STUDY OF THE MECHANICAL PROPERTIES AND BEHAVIOR ON HEAT-TREATMENT OF A HIGH STRENGTH ALUMINUM BASE FORGING ALLOY. James W. Poynter. July 1940.

TR 4560.

MECHANICAL PROPERTIES OF VARIOUS ALUMINUM ALLOY FORGINGS. N. B. Frank, Ture T. Oberg. August 1940.

TR 4566.

DETERMINATION OF INTERNAL STRESSES IN ALUMINUM ALLOY PLATES. R. T. Schwartz. October 1940.

TR 4902.

PROPERTIES OF XA75S-T ALUMINUM ALLOY. S. J. Broderick. April 1944.

TR 4902. Addendum.

PROPERTIES OF XA75S-T ALUMINUM ALLOY. S. J. Broderick, Ture T. Oberg. November 1943.

TR 5103.

EFFECT OF ARTIFICIAL AGING AND PRE-STRETCHING ON THE PROPERTIES OF ALCLAD 24S-T ALUMINUM ALLOY. S. J. Broderick. March 1944.

TR 5111.
INVESTIGATION OF R-301 CLAD ALUMINUM ALLOY SHEET. Baxter C. Madden, Major. May 1944.

TR 5129.
PROPERTIES OF ALCLAD XB75S SHEET. Richard R. Kennedy. July 1944.

TR 5187.
EXAMINATION OF PARALUMIN "2" ALLOY. William Courter, Pvt. January 1945.

TR 5228.
ALUMINUM ALLOY AIRCRAFT EXTRUSIONS. Donald A. Shinn, Ture T. Oberg. March 1945.

TR 5357.
PROPERTIES OF R303 ALUMINUM ALLOY SHEET. Gustavus S. Simpson, Jr., 1/Lt. November 1945.

TR 5589.
AGE HARDENING HEAT TREATMENT OF 75S-T AND R303 ALUMINUM ALLOY SHEET MATERIALS. James J. Niehaus. June 1947.

TR 5603.
DEVELOPMENT OF ML ALUMINUM ALLOY FOR ELEVATED TEMPERATURE SERVICE. J. C. McGee. June 1947.

TR 5696.
DETERMINATION OF HARDENABILITY OF LOW ALLOY STEELS. James W. Poynter. April 1948.

Pennsylvania State College. TR 5694.
TENSION-COMPRESSION BIAXIAL PLASTIC STRESS-STRAIN RELATIONS FOR ALCOA 24S-T. Joseph Marin, J. H. Faupel, V. L. Dutton. W33-038-ac-15934. April 1948.

TR 5701.
STATIC AND FATIGUE TESTS OF CAST AND FORGED I-BEAMS OF ALUMINUM AND MAGNESIUM ALLOYS. Ture T. Oberg, R. J. Rooney. May 1948.

TR 5945.
75S-T6 ALUMINUM ALLOY -- SOME BENDING AND DISTORTION CHARACTERISTICS. J. C. McGee. May 1950.

TR 6513.
FATIGUE PROPERTIES OF TUNGSTEN-ARC BUTT-WELDED WROUGHT ALUMINUM ALLOYS. Ture T. Oberg, Edward J. Ward, Captain, USAF. October 1951.

A study has been made of the fatigue characteristics in reversed bending of argon gas shielded tungsten arc butt welded joints in 14S-T and 75S-T sheet. The effects of post weld heat treating and filler material were investigated. The Aluminum Company of America furnished the specimens for the investigation which was conducted by the Materials Laboratory, Research Division, Wright Air Development Center.

The static tensile strengths of the welded joints varied from 46 to 69% of the tensile strengths of the sheet materials. The fatigue strengths were from 39 to 65% of the fatigue strength of the sheet material, depending upon the sheet material, the filler material and post weld heat treatment. The argon gas shielded tungsten arc welding process therefore does not produce joints in 75S-T or 14S-T alloys of satisfactory fatigue strength.

Aeorprojects, Incorporated. TR 6675.
ULTRASONICS APPLIED TO SOLIDIFICATION AND SOLID-STATE TRANSFORMATION. J. Byron Jones. AF 33(038)-11208. October 1951.

Ultrasonic energy was applied to 24S and 75S aluminum alloys after solution heat treatment, and the effect on age hardening evaluated, primarily by means of Rockwell hardness readings, and tensile tests. The effects were small and indicate that high intensity elastic energy retards precipitation hardening.

Ultrasonic energy was applied to eighteen cubic inch melts of pure magnesium, pure zinc, a 3.5 percent magnesium-zinc alloy, and magnesium-zirconium alloys. Grain refinement was accomplished and sounder ingots resulted from the use of ultrasonics. Zirconium sponge was alloyed with pure magnesium by the application of ultrasonics.

University of Minnesota. TR 52-307, Part 1.
FATIGUE PROPERTIES OF ALUMINUM ALLOYS AT VARIOUS DIRECT STRESS RATIOS, Part I -- ROLLED ALLOYS. B. J. Lazan, A. A. Blatherwick. AF 33(038)-20840. December 1952. PB 116164. Order from LC, Mi $4.00, Ph $11.50.

Newly developed equipment for axial stress fatigue testing in the tension-compression range is described. Fatigue data on 14S-T6, 24S-T4, and 75S-T6 aluminum alloys are presented as S - N curves and stress range diagrams to illustrate and analyze the effects of: (a) stress ratios in the range from static tension to reversed axial stress, (b) stress magnitude which causes failure in the range from 10^3 to 10^7 cycles, and (c) severity of circumferential notches having four different theoretical stress concentration factors in the range between 1.0 and 3.4. The extreme flatness of the stress range diagrams for severely notched specimens at long life is discussed in terms of the large reduction in mean load carrying capacity resulting from the addition of relatively small alternating stress. Unitless stress range diagrams are presented which indicate how material, life, and specimen type affect the combinations of alternating and mean stress which cause failure in a specified number of cycles. Data on the reduction in fatigue strength caused by notches are diagrammed to clarify the significance of mean stress, alternating stress, stress ratio, and cycles to failure as factors in fatigue notch sensitivity. The fatigue properties determined in this program are compared with prior work. The low fatigue strengths observed for 75S-T6 are briefly discussed.

University of Minnesota. TR 52-307, Part 2.
FATIGUE PROPERTIES OF ALUMINUM ALLOYS AT VARIOUS DIRECT STRESS RATIOS, Part II -- EXTRUDED ALLOYS. B. J. Lazan, A. A. Blatherwick. AF 33(038)-20840. December 1952. PB 116165. Order from LC, Mi $2.50, Ph $5.25.

Axial-stress fatigue tests were performed at various stress ratios on extruded aluminum alloys 14S-T6, 24S-T4, and 75S-T6 using one unnotched and one notched type of round specimen. The data are presented in the form of S-N curves and stress-range diagrams to analyze

the effect of: (a) stress ratio, ranging from static tension to reversed axial stress, (b) stress magnitudes which cause failure in the range from 10^3 to 10^7 cycles, and (c) stress concentration resulting from a circumferential V-notch. The notch-sensitivity data are further analyzed by diagrams which display the importance of stress ratio, stress level, and life on fatigue strength reduction. The fatigue properties of the three extruded alloys are compared both with each other and with rolled aluminum alloys.

National Bureau of Standards, U. S. Department of Commerce. TR 53-40.
EFFECT OF MEAN STRESS ON THE FATIGUE LIFE OF ALCLAD 24S-T3 AND ALCLAD 75S-T6 ALUMINUM ALLOY. I. Edward Wilks, Darnley M. Howard. AF 33(038)-51-4061, Part A. February 1953. PB 116 821. Order from LC, Mi $2.50, Ph $5.25.

An investigation has been conducted to determine the effect of mean stress on the axial loading fatigue life of two alclad aluminum alloys in sheet form with stress concentrations. The alloys were 24S-T3 and 75S-T6. The specimens were rectangular, 0.8 inches wide and 6.5 inches long with a 0.125 inch diameter hole drilled at the center. Tests were conducted using a lever type machine.

The range of mean stress was from 20,000 lb/in^2 tension to 20,000 lb/in^2 compression and the alternating stress ranged from zero to $\pm$ 30,000 lb/in^2. Some of the specimens tested in all compression stress range failed with axial as well as transverse cracks emanating from the edge of the hole.

The results showed that: (1) The effect of mean stress on 24S-T3 and 75S-T6 was the same, (2) At any alternating stress amplitude fatigue life increases as the mean stress decreases, (3) The effect of mean stress on fatigue life is increased as the alternating stress amplitude is decreased and (4) As the tensile mean stress decreases the allowable amplitude of alternating stress increases, in the compression range the allowable amplitude of alternating stress also increases as the compressive mean stress increases, at least as far as the range of this test.

Cornell Aeronautical Laboratory, Inc. TR 53-151.
STUDY OF HARD COATING FOR ALUMINUM ALLOYS. F. G. Gillig. AF 18(600)-98. May 1953. PB 111320. Order from OTS $2.00.

A study has been made of the effects of hard oxide coatings produced by the MHC process on the properties of five wrought and two cast aluminum alloys. Coating thicknesses ranging from 0.0005 inch to 0.005 inch were studied. Of the many properties that were studied, the abrasion resistance of the coatings and their effect on the fatigue strength of the parent metal are the most significant. The abrasion resistance of the hard coatings is far in excess of that of coatings produced by standard anodizing treatments and has been demonstrated to be equal to or better than that of thin cyanide coatings on steel. In addition to this, the coatings impart increased corrosion resistance to the aluminum alloy surface. The abrasion resistance decreases with exposure to humidity and atmospheric conditions but proper post-treatments, other than boiling water which is used for sealing regular anodized coatings, will undoubtedly prevent this. The most serious shortcoming of the coatings has been found to be their drastic lowering of the fatigue strength of the coated alloy. Decreases as much as 65% in the base metal fatigue strength have been found. The effect is not proportional to coating thickness and coatings of 0.001 inch produce practically the same effect as 0.005 inch coatings.

Cornell Aeronautical Laboratory, Inc. TR 53-151. Suppl. I.
STUDY OF HARD COATING FOR ALUMINUM ALLOYS. F. J. Gillig. AF 18(600)-98. October 1953. PB 111320s. Order from OTS $0.75.

The program for the study of the effects of hard oxide coatings (produced by the MHC Process) on the properties of aluminum and its alloys was extended in order to provide additional data. The corrosion resistance in three environments was evaluated up to 11 months. The abrasion resistance showed another small decrease with the five-month additional exposure to atmospheric and high humidity conditions. Two treatments that were given the coatings of 61S and 75S alloys appear to alleviate the drastic reduction in fatigue strength brought about by the coatings. Attempts at retaining the abrasion resistance in a humid atmosphere were only moderately successful. Oil was found to have a detrimental effect on the resistance to an erosion type of wear.

Aeroprojects, Incorporated. TR 53-527.
INVESTIGATION OF THE DEGASSING EFFECT OF ULTRASONICS ON ALUMINUM ALLOY. J. Byron Jones, John G. Thomas. AF 18(600)-32. February 1954. ASTIA Document No. AD 31527. PB 123091. Order from LC, Mi $3.90, Ph $10.80.

In this study an attempt has been made to degas molten aluminum by causing gas to collect and rise to the surface from the nodes of sound waves introduced into a melt. The apparatus for effecting this phenomena has been successfully built and demonstrated. In this work certain problems remain unanswered however, since the degassing was erratic and accompanied by serious contamination of the melt by erosion of the transducer coupler material. Efforts to solve the problems will be made in future work.

Univ. of California. WADC TR 54-104.
THE EFFECT OF TEMPERATURE, FREQUENCY, AND GRAIN SIZE ON THE FATIGUE PROPERTIES OF PURE ALUMINUM. N. H. G. Daniels, John E. Dorn. AF 33 (038)-22608. October 1954.

A comparison of the fatigue strength of coarse and fine grain high purity aluminum was made at room temperature and at 250°C. The superiority of the fine grain material as evidenced at room temperature was not maintained at the higher temperature where no difference was found between the fatigue properties of the two grain sizes.

A preliminary investigation of the effects of temperature on the fatigue behavior of pure aluminum was also carried out at frequencies of 25 and 1440 cycles per minute. The results suggest that high temperature fatigue of aluminum might be controlled by an activation process with an activation energy of approximately 34 K cals/gm atom, a value closely similar to that found for self-diffusion, creep, stress-rupture, recovery, and grain boundary relaxation. The change of fatigue properties with temperature and frequency is explained in terms of recovery. Supporting metallographic studies showing the corresponding change in structure with particular reference to polygonization have been made.

Other metallographic studies relative to the mode of failure and crack propagation are also described.

Aeroprojects Incorporated. WADC TR 54-490.
THE EFFECT OF ULTRASONICS ON MOLTEN METALS. J. Byron Jones, John G. Thomas, Carmine F. DePrisco.

AF 33(616)-2050. January 1955. PB 121403. Order from OTS $3.25.

The application of ultrasonics at a frequency of about 15 kc to small melts of 195 aluminum alloy resulted in accelerated degassing. Grain refinement was observed only when ultrasonic energy was introduced at or above the liquidus temperature. A microphone to appraise elastic energy levels in liquid melts was devised. Extensive investigations of the problems incident to the transmission of elastic vibratory energy into the molten metals were carried out, and the requirements therefore are reported.

Thompson Products, Inc. WADC TR 54-595.
RESEARCH INVESTIGATION OF THE FORGING CHARACTERISTICS OF ALUMINUM POWDER METALLURGY PRODUCTS. G. D. Dolch, F. E. McBride, J. P. Long, R. A. Paetz. AF 33(616)-2091. March 1955.

Three aluminum powder metallurgy products were forged into gas turbine compressor rotor blades to determine if these materials were amenable to such processing.

Forgeability was evaluated through a series of temperatures under various types of working, including: rolling, flat forging, up-setting, extruding, blocking, coining, and trimming.

The effect of forging temperatures on stress-rupture, tensile and fatigue strengths was evaluated by means of specimens prepared from flat forgings. Also, stress-rupture, tensile, and fatigue strengths, along with impact data were obtained on the as-received bar stock for a comparison base line.

Using strength data as a means of appraisal, one material and its optimum forging temperature were selected. Finished compressor blades were processed in accordance with optimum material-forging conditions, and subjected to limited fatigue and metallographic evaluation.

It was found that these materials could be processed into finished compressor rotor blades by substituting extrusion for rolling, and that processing is possible in the temperature range of 500-1000°F.

The two materials with oxide contents in the vicinity of 14% were found to have room temperature strengths comparable to a heat treated wrought aluminum alloy currently used for compressor rotor blades. However, the elongation and impact properties at all temperatures tested were inferior to the wrought aluminum alloy.

WADC TR 55-49.
THE EFFECT OF PRIOR CREEP ON THE MECHANICAL PROPERTIES OF ALCLAD 2024-T3. Clark E. Beck. September 1955.

Tensile tests at room temperature and at 500°F were conducted on Alclad 2024-T3 (formerly designated as clad 24S-T3) aluminum alloy sheet specimens after they had been subjected to various amounts of creep deformation. This prior creep deformation was obtained by stressing the specimens at constant loads in tension for periods of approximately 20 hours and 90 hours. The amounts of prior deformation varied from 0% in 2 inches for no applied stress to 2% in 2 inches with a stress of 15,500 psi applied.

At room temperature the tests showed a decrease in ultimate tensile strength properties as the amount of prior creep deformation increased toward the maximum values considered in this investigation. However, at 500°F the tensile yield strengths were much nearer the ultimate tensile strengths on a percentage basis than was shown for the room temperature tensile tests. There was a notable decrease in ultimate tensile and tensile yield strength values as the amount of prior creep deformation increased. This was particularly true for those specimens in which the prior deformation had been obtained in 90 hours.

Kaiser Aluminum and Chemical Corp. WADC TR 54-590.
POWDER FABRICATION OF ALUMINUM ALLOYS. J. B. Hess, R. S. Mateer. AF 33(616)-2296. September 1955. PB 121138. Order from OTS $0.75.

A fabrication method, combining powder metallurgy techniques and conventional extrusion, was developed for the purpose of alloying aluminum with refractory compounds and other unusual constituents. Additions of B_4C of Al_2O_3, TiC, SiC, ZrO_2, WC, TiAl, $MnAl_6$, $FeAl_3$, Mo, Cr, Si and Cu were made to a base of commercial atomized aluminum powders, and the resulting alloy properties were determined.

Outstanding improvements in Youngs' modulus were achieved by means of the SiC and B_4C additions; however, accompanying tensile properties were only mediocre.

Current attempts to improve these tensile properties by utilizing prealloyed powders as bases for the refractory additions are still in preliminary stages of study.

WADC TR 54-119.
PROPERTIES OF XA78S ALUMINUM ALLOY SHEET, PLATE, AND EXTRUSIONS. H. W. Zoeller, R. E. Wittman. November 1955. ASTIA Document No. AD 91224. PB 130350. Order from LC, Mi $6.00, Ph $18.30.

The mechanical and metallurgical properties of XA78S aluminum alloy clad sheet, plate and extrusions have been evaluated in this work. Long time tests will be reported in a supplement to this work. The results when compared with the evaluation by other investigators, may lead to the acceptance of this alloy as an aircraft material. The XA78S aluminum alloy has approximately 10 percent greater strength than other high strength aluminum alloys. It has corrosion resistance, fatigue strength, microstructural characteristics, response to heat treatment and spot welding properties comparable to other commercial aluminum base alloys containing zinc, magnesium, and copper. This alloy offers greater resistance to bending therefore, the forming will be more difficult. As a result of this evaluation three specifications have been issued. The designations for XA78S, 75S and 24S aluminum alloys have been recently changed to X7178, 7075, and 2024, respectively.

WADC TR 56-99.
MECHANICAL PROPERTY, CORROSION AND WELDING STUDIES ON 6066 ALUMINUM ALLOY. John D. Wood, 1/Lt. June 1956. PB 121497. Order from OTS $1.00.

The mechanical properties and stress corrosion characteristics of 6066-T6 extrusion were studied. The effect of salt spray exposure on 6066-T6 sheet was determined and found to approach 2014-T6 alloy. In addition, the welding characteristics of 6066 were investigated and it was found that 6066 alloy can be arc welded using 6066, 716 and 195 alloy filler metals.

Kaiser Aluminum & Chemical Corp. WADC TR 56-481.
POWDER FABRICATION OF ALUMINUM ALLOYS. S. G. Roberts. AF 33(616)-2296. April 1957. ASTIA Document No. AD 118192.

A new approach to aluminum alloy development, involving the use of powder metallurgy techniques to produce "superalloys" of aluminum, is described; and the preliminary results obtained in an evaluation survey of aluminum alloy systems using this approach are given. These results show that, by this approach, excellent prospects exist for the development of aluminum base alloys having improved mechanical properties at elevated temperatures. The alloy systems found to show promise as a base for alloy development were Al-Mn, Al-Fe, Al-Ti and Al-Cr.

Mixtures of aluminum alloy powders and low density refractory carbides having high moduli of elasticity were fabricated by hot compaction and extrusion, and evaluated by tensile testing at room and elevated temperature (600°F) and by microscopic examination. These fabrications were found to have high moduli of elasticity but were generally inferior in tensile properties to fabrications of the base aluminum alloys without carbide additions.

Extrusions were also prepared from Al-Cu-Mn, Al-Cu-Mg, Al-Mg and Al-Zn-Cu-Mg type alloy powders and evaluated. Significant improvements in the tensile yield strengths at 600°F were found to be effected by powder metallurgy fabrication as compared to conventional wrought fabrication for the Al-Cu-Mn and Al-Cu-Mg type alloys, but no benefits were obtained with the Al-Mg and Al-Zn-Cu-Mg type alloys.

The H_2O present in the surface film of aluminum alloy powder particles was measured and its effects on the characteristics of subsequent extrusions are discussed.

Northrop Aircraft, Inc. WADC TR 56-585, Part 1.
EFFECTS OF TEMPERATURE-TIME-STRESS HISTORIES ON THE MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL METALLIC MATERIALS. Part I. Temperature-Time Studies for 2024-T3 and 7075-T6 Alclad Sheet. Robert E. Fortney, Charles H. Avery. AF 33(616)-3028. September 1957. ASTIA Document No. AD 142007. PB 131520.

In order to establish realistic design criteria applicable to aerodynamically heated materials and their complex combinations of temperature, time and stress exposure and inspection criteria for materials after exposure to complex service conditions, the tensile properties of 2024-T3 alclad and 7075-T6 alcald sheet were determined at room temperature, 200°, 300° and 400°F after single and sequential multiple exposure in the range 250° through 600°F. In addition, the Rockwell hardness properties at room temperature after the above exposure conditions were determined to provide a basis for inspection of aircraft after service exposure to aerodynamic or engine heating.

Five tensile properties were determined for each exposure and test condition. Three of these, the proportional limit, modulus of elasticity, and percent elongation were tabulated and graphed in a non-dimentional form to generalize the data with respect to test material variability. Since the yield and ultimate strengths determine the load carrying ability, these tensile properties were analyzed carefully and generalizations with respect to exposure temperature and time and testing temperatures were accomplished. Statistical calculations were made to determine the accuracy of the various analyses. The conclusion was reached that the yield and ultimate strength analysis is adequate for establishing design criteria in the range room temperature through 400°F, after complex exposures to times from 1.0 to 1000 hours at temperatures from 250° to 600°F.

Material, equipment, specimens and procedures are described in detail. Test results are presented in the form of tables and curves to illustrate the effect of the exposure and test conditions on the materials under investigation and the effect of normalization analyses on the generalization of the data.

WADC TN 58-57.
METALLURGICAL INVESTIGATION OF ALUMINUM ALLOY X2219-T6. Paul L. Hendricks. June 1958. ASTIA Document No. AD 155672. PB 151235. Order from OTS $0.75.

This investigation was undertaken to determine some design properties of aluminum alloy X2219-T6 at room and elevated temperatures. The investigation including the determination of tensile properties, fatigue properties, stress rupture properties and stress corrosion characteristics of aluminum alloy X2219-T6 under various temperature conditions up to 600°F.

Nonferrous (Magnesium) Metals

TR 4514.
MAGNESIUM ALLOY EXTRUDED SECTION -- (ALUMINUM ZINC MAGNESIUM A.M.C.-57S). D. M. Warner. February 1940.

TR 4556.
CORROSION RESISTANCE OF MAGNESIUM ALLOY SHEET IN CONTACT WITH DISSIMILAR METALS. M. R. Whitmore. July 1940.

TR 4725.
MECHANICAL PROPERTIES OF MAGNESIUM ALLOY TUBING. G. M. Martell. January 1942.

TR 4780.
PROPERTIES OF MAGNESIUM CASTING ALLOYS. J. L. Gregg, S. J. Broderick. May 1942.

TR 5156.
MAGNESIUM ALLOYS. PROPERTIES AND AIRCRAFT APPLICATIONS. J. B. Cahan, P. W. Bakarian. October 1944.

TR 5246.
FOUNDRY PROCEDURES AND TEST DATA OF THE WRIGHT FIELD MAGNESIUM FOUNDRY. John J. Casey, Capt. July 1945.

TR 5349.
SUSCEPTIBILITY OF FOUR MAGNESIUM CASTING ALLOYS TO MICROPOROSITY AND ITS EFFECT ON THE MECHANICAL PROPERTIES. Jay R. Burns, S/Sgt. November 1945.

TR 5517.
SOME COMPARATIVE FOUNDING AND CORROSION PROPERTIES OF AZ63, AZ 92, A8 and AZ91 MAGNESIUM CASTING ALLOYS. Jay R. Burns, J. J. Casey, Wilford Dent. August 1946.

TR 5755
MAGNESIUM-ZINC BASE CASTING ALLOYS. Jay R. Burns. November 1948.

Rensselaer Polytechnic Institute. TR 5734. NEW MAGNESIUM ALLOYS. A. Jones, R. D. Malin, and R. R. Nash. W33-038-ac-16541(17401). November 1948.

TR 6173.
MAGNESIUM ALLOYS CONTAINING ZIRCONIUM. G. W. Orton, 1/Lt. June 1950.

Rensselaer Polytechnic Institute. TR 6174. MAGNESIUM ALLOY RESEARCH. TERNARY MAGNESIUM-LITHIUM BASE CONSTITUTION DIAGRAMS. MAGNESIUM ALLOYS OF LOW ALLOY ADDITIONS. PREPARATION OF SINGLE CRYSTALS OF MAGNESIUM ALLOYS. A. Jones, R. R. Nash, R. G. Brown, and W. P. James. W33-038-ac-22542. June 1950.

TR 6594.
CLADDING MAGNESIUM. George W. Orton, Captain, USAF, and A. David Gibson, Captain, USAF. July 1951.

Results of experiments to clad a strong magnesium alloy with aluminum alloys and anodic magnesium alloys are reported. It was demonstrated that magnesium alloys are easily clad with other magnesium alloys but cladding with aluminum is more difficult.

TR 5926.
MECHANICAL PROPERTIES OF EXTRUDED MAGNESIUM ALLOYS - ZK60 AND AZ80. W. J. Trapp. November 1950.

Rensselaer Polytechnic Institute. TR 52-169. MAGNESIUM ALLOY RESEARCH STUDIES. A. Jones, J. H. Lennon, R. R. Nash, W. H. Chang and E. G. MacPeek. W-33-038-ac-22542. September 1952. PB 111762. Order from OTS $3.50.

Diagrams are presented to show the constitution at 500° and 700°F for the magnesium-lithium-aluminum and magnesium-lithium-zinc alloy systems. These diagrams were determined mainly by microscopic examination of alloys quenched from elevated temperatures. Corrosion and loss of lithium prevented the successful use of x-ray diffraction and electrical resistivity measurements at elevated temperatures. The solubility for aluminum and for zinc was less at 500° than at 700°F but the solubility for lithium was almost the same at these two temperatures.

The study of dilute magnesium alloys found a good combination of properties for magnesium-1.0% zinc-0.4% cerium. The addition of cerium refined the grain structure and introduced a small amount of a second constituent. The best properties were obtained with a fine equiaxed grain structure produced by warm rolling and annealing just above the recrystallization range. These conditions of structure and properties were not obtainable by a single cycle of cold rolling and annealing.

Single crystals of high purity magnesium were grown by controlled solidification in a gradient furnace. The furnace and the mold remained stationary and only the temperature gradient moved. The conditions of growth were found to be more critical for single crystals of magnesium-aluminum and magnesium-zinc alloys. No alloy single crystals were produced but favorable conditions for their growth were approached. Causes and remedies are discussed for the failure to produce magnesium alloy single crystals.

TR 53-18, Part 1.
POWDER-FABRICATED MAGNESIUM ALLOYS, Part 1. DEVELOPMENT OF HIGH-STRENGTH SHEET FROM POWDER-FABRICATED Mg ALLOYS CONTAINING Zn, Zr, AND Al. H. A. Johnson, 2d Lt, USAF, ed. January 1953.

High strength sheet has been produced through development of rolling techniques for ZK + A20 powder extrusions. Excellent properties and rollability have been obtained by rapidly heating ZK60 powder extrusions to the rolling temperature, hot rolling in one heat, and warm rolling to the desired gage. High quality ZK60 powder fabrications which have corrosion rates equal to those of billet material have been produced. Illustrative properties of ZK60 sheet hot rolled on the 8" mill under the above technique are compared below with typical values of FS-H24, our present high strength commercial sheet alloy:

	Hot		1000 psi				Corrosion
Alloy	Rollability	%E	TYS	CYS	TS	NBE	Rate (mcd)
ZK60	450-950F	6	51	43	59	7	0.6-1.0
FS-H24	500-900F	16	32	27	42	11	0.4-0.6

As development work has been completed on laboratory equipment, we are now ready to make preliminary evaluation of powder fabricated ZK60 sheet on production equipment. Further improvement of the properties of ZK60 by small melt additions of Ca, Ce, Th, Cd, and Ag will be tried on the 3" mill.

Dow Chemical Company. TR 53-18, Part 2. POWDER-FABRICATED MAGNESIUM ALLOYS, Part 2. LARGE-SCALE EXTRUSION OF ZK60A ALLOY POWDER. H. A. Johnson, 2d Lt, USAF, ed. W33(038)ac-19884(19479). January 1953.

Approximately 7,000 pounds of ZK60A alloy powder have been extruded into various shapes on production equipment.

A kiln type powder heater, a portable powder loader, and a small extrusion container extension have been constructed and operated. Other equipment necessary for production extrusion of ZK60A alloy powder has been studied.

It has been demonstrated that high purity ZK60A alloy powder possesses a corrosion rate similar to that of billet extrusions.

It has been demonstrated that blisters are caused by flux and rust inclusions. The best method of eliminating blisters is screening the powder to -20 mesh, and heating it to 650°F prior to extrusion.

Typical properties and properties which 95% or more of the powder extrusions will meet based on the thickness of the extrusion are as follows:

Thickness	Typical Properties				95% Equal or Over			
Inches	1000 psi				1000 psi			
	%El	TYS	CYS	UTS	%El	TYS	CYS	UTS
Less than 0.2	17	38	37	48	7	32	33	42
0.2 and Greater	17	39.5	41	50	10	36	36	47

WADC TR 53-405.
WADC MAGNESIUM SYMPOSIUM-HELD ON 24 FEBRUARY 1953. Henry A. Johnson. March 1953. PB 119044. Order from LC, Mi $4.50, Ph $12.30.

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University of Minnesota. TR 53-181. FATIGUE PROPERTIES OF EXTRUDED MAGNESIUM ALLOY ZK 60 UNDER VARIOUS COMBINATIONS OF ALTERNATING AND MEAN AXIAL STRESSES. A. A. Blatherwick and B. J. Lazan. AF 33(038)-20840. August 1953. ASTIA Document No. AD 18117.

Axial-stress fatigue tests were conducted on specimens of extruded magnesium alloy ZK60A-T5 under five selected ratios of alternating to mean stress. The data thus obtained are presented in the form of S-N diagrams, one curve for each stress ratio. The effects of varying the combinations of alternating and mean stresses are demonstrated by a series of stress-range diagrams. Three types of specimens (one unnotched and two notched) provide data for analysis of the fatigue strength reduction due to stress concentration. These data are illustrated by two types of charts showing the fatigue strength-reduction factor as a function of stress ratio, life, and stress magnitude.

Rensselaer Polytechnic Institute. TR 53-113. MAGNESIUM ALLOY RESEARCH. A. Jones and R. R. Nash. W33-038-ac-22542. October 1953. ASTIA Document No. AD 24386.

A tentative constitutional diagram is presented for the magnesium-rich portion of the magnesium-thorium alloy system. This diagram shows limited solid solubility and a eutectic. The maximum solid solubility is estimated as 10 percent at the eutectic temperature of 1105°F. The eutectic composition contains about 35 percent thorium and the eutectic structure consists of an alpha solid solution and an intermediate phase containing about 55 percent thorium.

Tentative diagrams are given to show the effect of zirconium on the phase boundaries for magnesium-zinc-zirconium alloys containing four and six percent zinc, respectively. These diagrams are from data obtained by thermal analysis, microscopic examination and, to a limited extent, x-ray diffraction of a small number of alloys. The liquidus temperature increases slightly with an increase of zirconium in the four percent zinc alloys but remains nearly constant in the six percent zinc alloys. Very little data are given for the solidus and solvus temperatures.

Mechanical property data are given showing a good combination of strength and elongation in magnesium-1% zinc-0.4% cerium, magnesium-1% zinc-0.5% calcium, magnesium-1% zinc-0.4% cerium-0.001% lithium and magnesium-1% zinc-0.4% cerium-0.005% calcium. Cerium refines the grain structure and introduces a second constituent. The best properties are associated with a fine equiaxed grain structure.

Conditions and procedures are given for the controlled solidification method which produced magnesium and binary magnesium alloy single crystals with a nominal diameter of 1/2 inch and a length of about 8 inches. These crystals contain up to 5 percent indium, 5 percent cadmium, 0.5 percent zinc, 0.5 percent silver and 0.3 percent aluminum, respectively. Procedures are given for stressing the crystals in tension to establish the strengthening effects of the individual solutes. Work is reported up to the actual experimental stressing of the single crystals.

TR 54-83, Part 1. EXPERIMENTAL MAGNESIUM ALLOYS. PART 1 - FURTHER DEVELOPMENT OF PELLET FABRICATED MAGNESIUM ALLOYS. H. A. Johnson, 1st Lt, USAF. AF (600)-19147. June 1954. ASTIA Document AD 41427. PB 130394. Order from LC, Mi $3.90, Ph $10.80.

The effect of melt and Mg-Al pellet additions on the properties of Mg-Zn-Zr pellet and/or ingot fabrications was investigated in an attempt to develop higher strength sheet and extrusion alloys. The best combination of properties of Mg-Zn-Zr + Mg-Al pellet extrusions is obtained with ZK60; addition of Mg-Al decreases the sensitivity of the extrusion to annealing but does not improve the overall properties of the base alloy. Certain melt additions, however, do result in significant improvements in the properties of Mg-Zn-Zr. In extrusions, QZ66 containing 2% MM offers much higher strength, particularly CYS, than ZK60, while in sheet ZK40 containing 1%MM and 1%Th has properties vastly superior to AZ31. The following shows this comparison on the basis of laboratory work.

Extrusions* - T5					Sheet* - H26				
		1000 psi					1000 psi		
ALLOY	%E	TYS	CYS	TS	ALLOY	%E	TYS	CYS	TS
ZK60	9	45	38	53	AZ31	8	36	31	43
QZ66-2MM	6	49	50	58	ZE41-1Th	8	40	40	49

*Fabricated from ingots

Comparative properties of extrusions made from pellets and ingot clearly indicate a very marked superiority of the pellet fabrications. Melt additions, however, have smaller effects in pellet than in ingot fabrications.

TR 54-83, Part 2. EXPERIMENTAL MAGNESIUM ALLOYS. PART 2 - WROUGHT ALLOY SURVEY OF MINOR ADDITIONS TO SELECTED Mg- BASE ALLOYS. H. A. Johnson, 1st Lt, USAF. AF (600)-19147. June 1954. ASTIA Document No. AD 41425. PB 130395. Order from LC, Mi $3.00, Ph $6.30.

The effect of 1% additions of Ba, Cb, Cr, Hg, Li, MM, Mo, Pd, Se, Ta, Te, Th, Ti, Tl, V, and W on the mechanical properties, workability, formability, corrosion resistance, and microstructure of Mg-3Zn and Mg-5Al was investigated. Th greatly improves the strength of Mg-3Zn in both extrusions and rolled strip without an appreciable loss of toughness (NBE) as illustrated below:

	Extrusion-T5					Rolled Strip-H24			
		1000 psi					1000 psi		
Alloy	%E	TYS	CYS	TS	NBE	%E	TYS	CYS	TS
Mg-3Zn	17	22	12	35	25	13	24	18	33
Mg-3Zn-1Th	14	31	20	40	23	16	32	24	39

Smaller strength increases are obtained by the addition of Li, Ba, and Pd to both base alloys and with Hg and Cr in Mg-3Zn. Sizeable strength increases, through the addition of Th, Li, Pd, or Ba, are achieved only with an appreciable loss in workability, toughness, formability, and/or corrosion resistance. The other additions, Cb, MM, Mo, Se, Ta, Te, Ti, Tl, V, and W, have negligible effects.

TR 54-83, Part 3. EXPERIMENTAL MAGNESIUM ALLOYS. PART 3 - THERMAL AND ELECTRICAL PROPERTIES OF MAGNESIUM BASE ALLOYS. H. A. Johnson, 1st Lt, USAF. AF (600)-19147. June 1954. ASTIA Document No. AD 41426. PB 130809. Order from LC, Mi $3.00 Ph $6.30.

The effect of crystal orientation on the electrical resistivity of high-purity magnesium was determined. The results, at 24C, may be represented by the equation:

$$\rho(\Phi) = 4.60 - 0.75 \cos^2 \Phi ,$$

where Φ is the angle between the hexagonal axis and the direction of current flow.

The effect of temperature on the electrical resistivity for varying orientations was investigated, with temperature coefficients of .00390 and .00408/C obtained perpendicular and parallel to the hexagonal axis respectively for the temperature range of 24 to 200°C.

Extruded high-purity magnesium and ZK60A-T5 alloy were stressed in tension to various levels and the effect of this prestressing operation on the electrical resistivity recorded. There was no significant change in electrical resistivity at the levels investigated for either alloy.

Apparatus for the measurement of thermal conductivity, electrical conductivity and linear thermal expansion have been purchased or designed but are still in either the construction or calibration stage.

The advantages and disadvantages of electric resistance strain gages as a method of measuring the coefficient of linear thermal expansion are discussed.

TR 54-83, Part 4.
EXPERIMENTAL MAGNESIUM ALLOYS. PART 4 - EVALUATION OF HK31. H. A. Johnson, 1st Lt, USAF. AF (600)-19147. June 1954. ASTIA Document No. AD 45830. PB 135340. Order from LC, Mi $2.40, Ph $3.30.

This phase of work on contract No. AF (600)-19147 includes the testing and evaluation of production rolled HK31XA sheet. The results are preliminary insofar as the overall production development of this alloy is concerned but do indicate the good combination of room and elevated temperature properties previously obtained in the laboratory can be produced under plant-scale rolling conditions. Continued development work and customer evaluation is warranted.

TR 54-83, Part 5.
EXPERIMENTAL MAGNESIUM ALLOYS. PART 5 - WELDING TESTS ON HK31A SHEET. H. A. Johnson, 1st Lt, USAF. AF (600)-19147. June 1954. ASTIA Document No. AD 41424. PB 131956. Order from LC, Mi $3.30, Ph $7.80.

The arc, gas and spot welding characteristics of HK31A sheet and plate were investigated. The alloy was very weldable by the arc and spot methods but difficulty was encountered in gas welding. Heavy plate, multiple pass arc welds were subject to oxide inclusions, the cause of which has not been definitely established. Weld strengths could be improved by aging heat treatments. The welds were subject to germination when solution heat treated at the recommended 1050°F temperature. The short time high temperature properties of the welds were not significantly different from the unwelded material. Spot welding techniques and properties appeared to be similar to those obtained on AZ31A sheet. Suggestions for continuing the evaluation of the welding characteristics of HK31A were included.

Rensselaer Polytechnic Institute. WADC TR 54-347.
MAGNESIUM SHEET ALLOYS. R. R. Nash, H. K. Adams, Jr., A. E. Bibb, Jr., E. J. Tulloch, M. C. Huffstutler. AF 33(616)-439. November 1954.

Tentative optimum procedures to add chromium, antimony, strontium, barium, calcium and manganese to AZ31 base alloy are described. Relative recoveries of additions of chromium, strontium and barium were extremely low. Recoveries of antimony, calcium and manganese approached the intended additions. These additions were made singly and in combination to determine their influence on the mechanical properties and resistance to corrosion of AZ31 alloy.

The most promising combination of mechanical strength and ductility was associated with alloys containing single additions of chromium and additions of chromium and manganese together in the as-fabricated by hot rolling condition and after cold rolling a controlled amount followed by a stress relieving heat treatment. Compared with AZ31 alloy on the basis of the same conditions of mechanical working and heat treatment, this combination of mechanical properties was a small but definite improvement.

The relative resistance to corrosion of AZ31 alloy was lowered by additions of chromium, barium, strontium and antimony and improved by manganese additions above a nominal 0.3%. This behavior was related, tentatively, to the iron content of the experimental alloys.

Dow Chemical Company. WADC TR 55-160, Part 1.
INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES. PART 1 - Improved Mg Wrought Alloys. J. D. Wood, 1/Lt., USAF. AF 33(616)-2337. October 1955. ASTIA Document No. AD 90583. PB 135202. Order from LC, Mi $3.90, Ph $10.80.

The work on this contract was directed toward the improvement of high strength wrought Mg alloys through alloy survey based on Mg-Zn and evaluation of promising compositions in pellet fabrications. A Mg sheet alloy containing 4-5% Zn, 1-2% MM, and 1-2% Mn with an excellent combination of workability, strength, and ductility was developed. A typical yield strength of 35,000 psi or higher should be realized through proper fabrication of these alloys. Further study of the Mg-Ag-Zn-Zr system indicated that high strength sheet can also be obtained by work hardening Mg-Ag-Zn-Zr alloys such as QZ66 and QZ63. Additional work is necessary to determine if higher strength can be obtained in these alloys than in the Mg-Zn-MM-Mn (or Zr) system. Considerable promise is also shown by HM31 pellet extrusions. Pellet fabrication of this alloy substantially increases static strength, particularly CYS at temperatures up to 600°F. It has the highest static strength in the range of 300-600 F of any Mg extrusions yet developed.

Dow Chemical Company. WADC TR 55-160, Part 2.
INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES. PART 2 - Physical Properties of Mg Base Alloys. J. D. Wood, 1/Lt., USAF. AF 33(616)-2337. October 1955. ASTIA Document No. AD 90584. PB 135201. Order from LC, Mi $2.70, Ph $4.80.

The coefficients of linear thermal expansion have been determined for pure magnesium, AZ31A, EZ33A, HK31XA, and HZ32XA using a high precision dilatometer. The electrical conductivities of AZ31A, AZ31B, AZ63A, AZ92A, cast ZK60A, and pellet extruded ZK60A have been determined using a Kelvin Bridge. Values obtained compare favorably with previous determinations. Thermal conductivities of the alloys were calculated from the electrical conductivity data using the Powell and Bungardt equations. The heat capacity and heat fusion of pure magnesium are also reported.

The effects of heat treatment, composition, mechanical history, and working direction on electrical conductivity were noted. There was no significant effect of these variables on thermal expansion.

Dow Chemical Company. WADC TR 55-160, Part 3.
INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES. PART 3. J. D. Wood. AF 33(616)-2337. November 1955.

Inhomogeneous deformation has been studied in high-purity magnesium and magnesium-calcium alloys. Metallographic examination of cold-rolled sheet disclosed that narrow bands of preferential recrystallization had occurred in the alloys containing less than 0.055 percent calcium. These bands rotated toward the rolling plane with increasing reduction while newly formed bands subtended an angle of 30°. Room temperature compression tests on hot-rolled materials showed that the magnesium showed banding whereas a Mg-0.5 Ca alloy did not, in agreement with the observations on sheet. The homogeneity of deformation also showed an isotrophy in the compression tests. In both compression and rolling the bands became eventual crack loci.

A large amount of information on preferred orientation of flat magnesium alloy extrusions has been obtained. As in the case of rolled magnesium alloy sheet, various deviations from the ideal orientation occur. These deviations are primarily rotations of the maximum basal pole density about the transverse direction as an axis. In addition, marked variation of preferred orientation with depth below the surface has been found. Profile plots of the rotation as a function of depth fall into two broad groups: (1) those which show a double orientation at the center and (2) those which show a single orientation at the center. A theory is outlined in an attempt to explain these general categories of sheet and flat extrusions. Basal slip and twinning are assumed of primary importance. Compressive strains are modified by shear strain in such a way as to explain many of the experimental observations in a consistent manner.

WADC TR 56-409.
AUTORADIOGRAPHIC STUDY FOR THE DISTRIBUTION OF THORIUM IN MAGNESIUM ALLOYS. Kurt Wolfsbert, 1/Lt, George John. November 1956. ASTIA Document No. AD 110571. PB 121975. Order from OTS $1.25.

An autoradiographic study of magnesium alloys containing 3% and 13% thorium has been performed. The stripping film method was slightly modified because of the extreme reacitivity of magnesium. The results show qualitatively that a large percentage of the thorium is located in or very near to the grain boundaries.

Dow Chemical Company. WADC TR 56-88.
INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES. G. S. Foerster, S. L. Couling, H. Baker, R. Johnson. AF 33(616)-2337. November 1956. ASTIA Document No. AD 110541. PB 121801.

The work reported under this contract is divided into three sections. Section 1 deals with the development of improved Mg wrought alloys. Two Mg-MM-Th-Zn-Zr wrought alloys--ZE11 (.6Th-.6Zr) in sheet and ZE31 (1Th-.6Zr) in extrusions--have excellent properties at moderate temperatures, unobtainable in any conventional Mg alloy yet developed. Excellent properties at moderate temperatures can also be obtained by pellet extrusion of high temperature Mg alloys such as HZ32XA. High strength Mg-4Zn-1.5MM-1.5Mn sheet has been developed for room temperature service but is slightly inferior to ZE41XA, a new experimental sheet alloy.

In Section 2, recent measurements of the thermal and electrical properties of several Mg alloys--AZ31A, HK31XA, HZ32XA, EK30A, EZ33A and HM21XA--are reported. Electrical resistivity varies from 5.3×10^{-6} for HM21XA-H24 to 9.1×10^{-6} ohms/cm^3 for cast AZ31A-F at room temperature and from 9.2×10^{-6} to 12.9×10^{-3} for the same alloys at 500F. The temperature coefficient of electrical resistivity is essentially constant at 0.90×10^{-8} ohms/cm^3/°F. The thermal conductivity of these alloys varies in the reverse order of their electrical resistivity and increases with temperature.

The plastic deformation and preferred orientation of wrought Mg alloys are discussed in Section 3. Mechanical twinning is significant (15-20 volume per cent) in the cold rolling of AZ31A and AZ31B. The attempt to measure indirectly twin volume was only partly successful. Bend tests of Mg-2Al sheet indicate that twin volume decreases with increasing temperature but is still appreciable as high as 815F. The role of heterogeneous deformation or banding in compressive working of Mg alloys is also discussed.

Battelle Memorial Institute. WADC TR 56-405.
A STUDY OF THE FEASIBILITY OF COATING MAGNESIUM WITH HIGH-PURITH ALUMINUM. C. F. Powell, I. E. Cambell. AF 33(616)-3309. Nov. 1956. ASTIA Document No. AD 110512. PB 121 860.

The feasibility of vapor plating magnesium and magnesium-base alloys with aluminum is evaluated on the basis of information available in the literature on volatile aluminum compounds with the stability range required for vapor-plating "atmospheres".

Pyrolysis of aluminum alkyls and of aluminum hydride and its derivatives is considered of sufficient interest to warrant experimental investigation.

Reduction of aluminum halides with magnesium and disproportionation of aluminum monohalides are considered technically feasible but of lesser interest because the conditions for procuring satisfactory coatings are critical.

WADC TN 56-461.
THE DETERMINATION OF CALCIUM IN MAGNESIUM BASE ALLOYS. Lois A. Keyser, Charles D. Houston. Jan. 1957. ASTIA Document No. AD 110706.

A gravimetric procedure suitable for the determination of trace amounts of calcium in magnesium-base alloys has been developed in this laboratory.

The sample is dissolved in hydrochloric acid and the magnesium is removed by a double precipitation as the phosphate. The calcium is then

precipitated at a pH of six as the oxalate and the washed precipitate is titrated with potassium permanganate.

Massachusetts Institute of Technology. WADC TR 56-453.

FACTORS AFFECTING THE FLUIDITY AND HOT CRACKING OF MAGNESIUM ALLOYS. Howard F. Taylor, Merton C. Flemings, Jr., AF 33(616)-2958. January 1957. ASTIA Document No. AD 118011. PB 131045. Order from OTS $2.50.

A vacuum fluidity apparatus was used to determine the fluidities of magnesium alloys. The alloy systems studied were:

1. Magnesium-aluminum binary system from 0 to 20% aluminum.
2. Magnesium-zinc binary system from 0 to 20% zinc.
3. Magnesium-aluminum-zinc ternary system up to 20% alloy content.
4. Magnesium-zinc-zirconium system from 0 to 20% zinc.
5. Magnesium-thorium-zirconium system from 0 to 20% thorium.

Fluidity was determined as a function of temperature for each alloy. Then fluidity at 1400°F was plotted as a function of alloy content. Fluidity at 100°F superheat was plotted for those alloy systems for which the liquidus temperature had been established. Comparison of commercial alloys with the experimental alloys was good. Fluidity curves as a function of alloy content were found to vary as the inverse of computed, non-equilibrium freezing range curves.

A hot tear pattern was adapted for studying the effect of geometry on the hot tearing of magnesium alloys, and for determining the susceptibility of various magnesium alloys to hot tearing; the pattern is stressed in simple tension during solidification. The portion of the test casting susceptible to hot tearing is well fed. Hot tearing in Mg-4% Al alloys was shown to increase with decreasing fillet radius, increasing casting length, increasing section size change, and decreasing gas content.

An experimental apparatus was developed for the measurement of alloy rupture stress and ductility at temperatures above and below the solidus. The apparatus permits stress rupture measurements without first cooling the cast alloy to room temperature.

Rensselaer Polytechnic Institute. WADC TR 55-207.

MAGNESIUM ALLOYS WITH HIGH MELTING POINT ADDITIONS. R. R. Nash, H. K. Adams, Jr., A. E. Bibb, Jr., M. C. Huffstutler, E. J. Tulloch. AF 33(616)-2312. March 1957. ASTIA Document No. AD 118122. PB 131198. Order from OTS $5.00.

An exploratory investigation was made to determine the alloying characteristics and the influence on microstructures, mechanical properties and resistance to corrosion of small additions of titanium, hafnium, tantalum, nickel, cobalt, vanadium, boron, chromium, yttrium, rhenium, niobium, tungsten and molybdenum to magnesium and to selected magnesium-base alloys.

A potentially attractive method for the pronounced grain refinement of as-cast structure of magnesium-base alloys was found. Additions of small amounts of titanium dioxide and several forms of vanadium produced a moderate refinement in un-alloyed magnesium. These additions were particularly effective when made to magnesium alloys with one or more soluble components. The mechanism of this action was believed to be extensive grain nucleation by stable, foreign nuclei in constitutionally super-cooled liquid.

The solubility of titanium, hafnium, vanadium, boron, chromium, niobium, tungsten and molybdenum in magnesium was believed to be extremely limited. Additions of these elements to magnesium and to selected magnesium-base alloys did not influence significantly the mechanical properties or resistance to corrosion of hot rolled sheet. A consistent increase in tensile mechanical properties without loss in ductility in the as-extruded condition was associated with increased chromium additions to AZ31 composition reaching an optimum threshold at approximately 0.3 to 0.5% chromium, intended additions. The retention of rhenium and tantalum in magnesium and magnesium alloys was also believed to be of low magnitude and no improvement in mechanical properties or general resistance to corrosion was observed. Exceptionally high resistance to corrosion was exhibited by two alloys with additions of rhenium and two alloys with additions of tantalum to unalloyed magnesium in the form of hot rolled sheet. The result was considered significant but the specific cause of the benefit was not isolated.

Nickel and cobalt exhibited limited solid solubility in magnesium and magnesium alloys. Nickel additions produced a small improvement in room and elevated temperature mechanical properties of hot rolled sheet but lowered sharply the resistance to corrosion. Cobalt additions were less effective in improving mechanical properties and also lowered resistance to corrosion.

Microscopic examination indicated that the solid solubility of yttrium in magnesium was at least 0.5%. Yttrium additions to unalloyed magnesium produced an increase in tensile yield and ultimate strengths of 36 and 26%, respectively, in the form of hot rolled sheet at room temperature.

Polytechnic Institute of Brooklyn. WADC TR 57-241, Part I.

DEVELOPMENT OF A CORROSION RESISTANT MAGNESIUM ALLOY. Part I - Development of Magnesium Alloys for Better Corrosion Resistance. M. Balicki, C. D'Antonio, A. Kravic. AF 33(616)-2917. August 1957. ASTIA Document No. AD 131018. PB 131443.

A novel way for finding alloys of magnesium with better corrosion resistance has been evolved. This scheme relies upon alloying magnesium with elements which, owing to their lower surface tension, are expected to concentrate on the surface and thus alter the corrosion behavior. From a number of elements having a lower surface tension than magnesium the following: Hg, Pb, Ge, In, Bi, Sn, Cd, Ca have been added to it to form very pure, dilute, binary alloys. Since this was a feasibility study, insufficient data was obtained to allow a statistical analysis of results. The preliminary results obtained in general, however, indicate that magnesium alloys containing Sn, Sb, Ge, Cd, and In compare favorably with pure magnesium in the QQ-M-151A Salt Spray Test.

Polytechnic Institute of Brooklyn. WADC TR 57-241, Part II.

DEVELOPMENT OF A CORROSION RESISTANT MAGNESIUM ALLOY. Part II - Surface Tension Data of Elements. V. P. Siuta, M. Balicki. AF 33(616)-2917. August 1957. ASTIA Document No. AD 131010. PB 131444.

Experimental and estimated values of surface tension of 75 elements have been gathered. This unique collection was of value in elucidating the magnesium alloying program and should be of use to workers in other fields.

Correlations for estimating surface tension values of elements have been scrutinized, augmented and used. Extensive bibliography on the subject of surface tension is presented.

WADC TR 56-415.
DEVELOPMENT OF ZM41 MAGNESIUM SHEET ALLOY. H. A. Johnson, R. D. Masteller, 1/Lt. September 1957. ASTIA Document No. AD 131042. PB 131417.

A very promising magnesium alloy system was developed which appears superior to AZ31 sheet alloy. This alloy has been designated as ZM 41 and has a nominal composition of 4 zinc 1.0 Manganese 0.7 mischmetal balance Magnesium. Some of the features of ZM41 alloy compared to AZ31 are higher strength, more uniform longitudinal and transverse tensile properties, and a much greater tolerance for iron without the corrosion resistance of the alloy being adversely affected.

Dow Chemical Company. WADC TR 57-194, Pt. II.
INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES. Part II - Thermal and Electrical Properties of Magnesium Base Alloys. H. Baker. AF 33(616)-2337. September 1957. ASTIA Document No. AD 131034. PB 131436.

The electrical resistivities for cast AZ31A & B, AZ63A, AZ81A, AZ91C, AZ92A, AM100A, EK30A, EK41A, EZ33A, HK31A, rolled HK31A and HM21XA, and cast HZ32A were determined at room temperature and in some cases up to 500F. The values at 68F ranged from 17.2 microhm/cm^3 for AM100A-T4 to 9.2 microhm/cm^3 for AZ31A & B -F & -T4 and from 7.7 microhm/cm^3 for cast HK31A-T6 to 5.0 microhm/cm^3 for rolled HM21XA-T8. At 500F the values ranged from 11.6 microhm/cm^3 for EK41A-T6 to 9.1 microhm/cm^3 for HM21XA-T8 for the RE and Th containing alloys. Room temperature resistivity measurements on cast EM31 and EK31 ranged between 7.8 and 5.5 microhm/cm^3 for the -T6 and -T7 tempers respectively. The temperature coefficients of electrical resistivity were fairly constant ranging from 0.8 to 1.0 microhm/cm^3-^{o}F. Calculations of thermal conductivity from electrical data are reported for AZ31A & B, AZ63A, AZ81A, AZ91C, AZ92A, AM100A, EK30A, EK41A, EZ33A, HK31A, HM21XA, and HZ32A. The temperature coefficients of thermal conductivity were all positive and decreased with increasing temperature. Enthalpy, specific heat and heat of fusion were measured for AZ31B, HK31A, HM21XA, HM31XA, and ZK60A. The heat of fusion seemed to decrease with increasing alloy content from 82 cal/gm for HM21XA to 76 cal/gm for ZK60A. At temperatures around 500F the specific heats of the alloys were about the same as that of pure magnesium except the specific heat of AZ31B which was somewhat higher.

Dow Chemical Company. WADC TR 57-194, Part III.
INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES. Part III - Development of Preferred Orientation in Wrought Magnesium Alloys. S. L. Couling. AF 33(616)-2337. September 1957. ASTIA Document No. AD 131035. PB 131437.

A polarized-light metallographic technique capable of supplying information on the orientation of individual grains in a polycrystalline magnesium aggregate has been developed and used as a tool in several orientation studies. The distribution of preferentially oriented grains in various extruded alloys has been examined and it was found that elongated clusters of grains of one orientation alternate with clusters of another orientation throughout much of the extrusion thickness. Inhomogeneous flow or "banding" has been found to be an important deformation mechanism during the cold rolling of certain alloys and the hot compression of alloy cylinders. The bands are believed to be formed by a fracture-glide mechanism. Banding may be operative during hot rolling with large reductions per pass and could account for the observed double peak in the preferred orientation of most rolled alloys. Polycrystalline material with a preferred orientation close to the ideal can be twinned almost completely by the application of a sufficient compressive stress parallel to the sheet surface; on annealing, the heavily twinned metal recrystallizes in a preferred orientation markedly different from the initial ideal orientation.

Cornell University. WADC TR 56-478.
LITERATURE SURVEY OF THE CORROSION OF MAGNESIUM AND MAGNESIUM ALLOYS. Henri S. Sack, Bryon P. Roe. AF 33(616)-3032. September 1957. ASTIA Document No. AD 142008.

This report represents a literature survey of the corrosion of magnesium and magnesium alloys, and emphasizes papers published since 1940. In view of the complexity of the phenomena and controversial or insufficiently documented experimental results only very few generalizations concerning the corrosion process can be made.

Cornell University. WADC TR 57-576.
A BASIC STUDY OF CORROSION OF MAGNESIUM. Richard R. Addiss, Mitchell S. Cohen, Robert I. Frank, Herbert Hollister, Henri S. Sack, Karl Scharf. AF 33 (616)-3032. December 1957. ASTIA Document No. AD 142209. PB 131662.

This report constitutes the final report of the work done under the present contract. Attempts to obtain a perfectly clean surface of magnesium are described. Due to limitations in time no definite results on the oxidation of Mg were obtained; however, some preliminary results on the patterns obtained from pure Mg in a field emission microscope, and the oxidation at low temperatures are reported.

Dow Chemical Company. WADC TR 57-518.
DETERMINATION OF THE FORGING CHARACTERISTICS OF NEW AND HIGH TEMPERATURE MAGNESIUM ALLOYS. R. E. Hook, J. F. Pashak. AF 33(616)-3578. December 1957. ASTIA Document No. AD 142197.

This laboratory forging program was established for the purpose of selecting the best magnesium alloy or alloys for elevated temperature service. The alloys investigated were three Mg-Th alloys (HM21XA, HK31A, HZ32A) and four Mg-RE alloys (EK31(MM), EZ33A, EK31(Di), EM41XA); two commercial alloys (AZ80A, ZK60A) were included for comparative purposes. Both cast ingot and extruded billet were used as forging stock. Static properties and creep resistance of the alloys were determined on forged discs of 90% reduction. The data, therefore, cannot be considered as representative of die forgings.

Of the alloys investigated, HM21XA-T5 exhibits the best combination of properties in the range of 500 to 700F while EK31XA-T6 presents the optimum values at 70 to 500F. Pre-forging cast ingot has no significant effect on the properties of forgings when the final forging reduction is as great as 75%; however, increasing preforging temperature has a deleterious effect on the final forged properties.

Ohio State University Research Foundation. TR 5732.
INVESTIGATION OF CHROMIUM-TITANIUM BINARY ALLOYS. Dr. M. G. Fontana. W33-038-ac-16368. November 1948.

TR 5935.
MECHANICAL PROPERTIES, WELDABILITY AND CORROSION OF COMMERCIALLY PURE TITANIUM. Dr. H. K. Adenstedt. February 1950.

Ohio State University Research Foundation. TR 5946.
TITANIUM-CHROMIUM BINARY ALLOYS. Dr. M. G. Fontana. W33-038-ac-21339(20377). May 1950.

Battelle Memorial Institute. TR 6218, Part 2.
RESEARCH AND DEVELOPMENT ON TITANIUM ALLOYS. L. W. Eastwood and C. H. Lorig. AF 33(038)-3736. June 1950.

Battelle Memorial Institute. TR 6623.
DEVELOPMENT OF TITANIUM-BASE ALLOYS. J. W. P. Rengstorff, C. T. Greenidge, L. W. Eastwood, C. H. Lorig, J. O. Brittain, H. A. Pray, P. D. Frost, W. H. Kearns, R. D. Williams, C. B. Voldrich, W. F. Fink, R. F. Peoples, W. F. Simmons, and H. C. Cross. AF 33(038)-3736. June 1951.

Battelle Memorial Institute. TR 6516, Part I.
THE TITANIUM-MANGANESE, TITANIUM-TUNGSTEN, AND TITANIUM-TANTALUM PHASE DIAGRAMS. R. I. Joffee, L. W. Eastwood, and D. J. Maykuth. AF 33(038)-8544. June 1951.

New York University. TR 6596, Part 1.
TITANIUM-NICKEL PHASE DIAGRAM. John P. Nielsen, Harold Margolin. AF 33(038)-8725. October 1951.

Covering the period of September 30, 1949 to December 1, 1950, this report presents the data obtained by the Research Division of New York University. The titanium-nickel phase diagram has been investigated up to 90 percent nickel, and the features up to 40 percent nickel have been established. An eutectic horizontal is found between 950° and 965°C, and an eutectoid transformation between 750° and 775°C. Little solubility of nickel in alpha titanium is indicated. Preliminary experiments on high nickel alloys indicate a possible error in the Wallbaum diagram in the range 55-60 percent nickel. Because of possible contamination, further work is necessary to determine whether any difference actually exists. Also included are accounts of alloy preparations, heat treatment, polishing and X-ray techniques employed in this investigation.

New York University. TR 6596, Part 2.
THE TITANIUM-NICKEL PHASE DIAGRAM. H. Margolin, E. Ence and J. P. Nielsen. AF 33(038)-8725. October 1951.

This summary report presents the data obtained by the Research Division of New York University for Part 2 of the investigation of the Titanium-Nickel Phase diagram during the period 1 December 1950 to 30 September 1951.

The complete diagram from 0-100% nickel and a comparison of the phase diagrams for Process A sponge and iodide titanium-base nickel alloys up to 15% nickel are presented. Comparisons of results have been made with other work described in the literature. Copper contamination encountered in high-nickel alloys was almost entirely eliminated by using smaller charges and shorter melting times.

Summaries of procedures for alloy preparation, melting, heat treatment, metallographic and X-ray investigation are included.

Massachusetts Institute of Technology. TR 6595, Part 1.
STUDIES AND EXPERIMENTAL INVESTIGATIONS FOR THE DEVELOPMENT OF PHASE DIAGRAMS OF THE TITANIUM-CHROMIUM AND TITANIUM-COPPER ALLOY SYSTEMS. Frank B. Cuff, Jr., Arnold S. Joukainen, Lee S. Richardson, John C. Nicholls. AF 33(038)-8754. November 1951.

The titanium-chromium binary system has been partially completed in the range from 0 to 63 percent chromium, using high purity (99.7 percent) sponge titanium and electrolytic chromium.

A preliminary diagram has been drawn of the titanium-copper system from X-ray and metallographic investigations.

An investigation is in process for determining a reliable method for analysing the oxygen and nitrogen content of titanium by the use of vacuum fusion.

University of Notre Dame. TR 6597, Part 1.
THE TITANIUM-IRON PHASE DIAGRAM. W. J. Fretague, C. S. Barker and E. A. Peretti. AF 33(038)-8495. November 1951.

This project was concerned with the development of a phase diagram of the titanium-iron system, with special emphasis on that region of the diagram between 0 and 50 percent iron and from room temperature up to and including the solidus of the system. An arc melting furnace, employing a water cooled tungsten electrode and copper crucible, was constructed and operated with an argon atmosphere. Approximately seventy-seven arc melted alloys were prepared covering the composition range from 0 to 71 percent iron. Hardness data on the as-cast alloys were collected and metallographic examination of all as-cast alloys performed. A tentative diagram was constructed from the information obtained from metallographic examination of the arc melted alloys, and from published information appearing in the literature. The eutectic composition of the system was estimated to be at approximately 32 percent iron as determined by examination of the as-cast structures. The eutectic temperature of the Process A titanium -- 32 percent iron alloy was determined to be 1094° ± 1°C. Thermal analysis of a 10 percent iron alloy established an arrest at approximately 550°C. At the present time, this is believed to be an eutectoid reaction although typical eutectoid microstructures have not been obtained.

Massachusetts Institute of Technology. TR 6595, Part 2.
STUDIES AND EXPERIMENTAL INVESTIGATIONS FOR THE DEVELOPMENT OF PHASE DIAGRAMS OF THE TITANIUM-CHROMIUM AND TITANIUM-COPPER ALLOY SYSTEMS, Part 2. THE TITANIUM-COPPER AND THE TITANIUM-CHROMIUM PHASE DIAGRAMS. Arnold S. Joukainen, Frank B. Cuff. AF 33(038)-8754. December 1951.

In view of the recognition of the potentialities of titanium and its alloys as important structural materials, there has arisen a need for a systematic investigation of various binary diagrams. Of these, the titanium-copper, and the titanium-chromium systems were investigated under this contract.

The titanium-copper system was found to contain four inter-metallic compounds: Ti_2Cu, TiCu, Ti_2Cu_3 and $TiCu_3$. It is an eutectoid type system offering heat treatment possibilities due to a suppression of the alpha to beta transformation.

The titanium-chromium system was of particular interest because of the improved properties imparted to titanium by small chromium additions. It was found that a continuous series of solid solutions existed between titanium and chromium. An intermetallic compound, $TiCr_2$, which decomposes upon heating, was found at 60 to 65 weight percent chromium. At the titanium end of the diagram there is an eutectoid type of reaction at 15 weight percent chromium.

University of Notre Dame. TR 6597, Part 2. THE TITANIUM-IRON PHASE DIAGRAM. W. J. Fretague, C. S. Barker, E. A. Peretti. AF 33(038)-8495. March 1952.

Battelle Memorial Institute. TR 52-249. DEVELOPMENT OF TITANIUM-BASE ALLOYS. C. H. Lorig, et al. AF 33(038)-3736. June 1952. ASTIA Document No. AD 6453. PB 126910. Order from LC, Mi $9.60, Ph $33.30.

Selection and Heat Treatment of Promising Alloys.

Experimental work conducted under this contract prior to May, 1951, had shown that the beta-stabilizing elements, chromium, iron, manganese, molybdenum and vanadium, offered the most promise in the development of titanium alloys. The alpha-stabilizing, interstitial elements, carbon, oxygen, and nitrogen, were potent strengtheners, but decreased ductility disproportionately to their strengthening ability.

Based on these observations, most of the exploratory alloys melted and tested during the past contract year contained one or more of the five metallic elements mentioned above. Several ternary alloys showed much promise as potential high-strength alloys. The Ti-Mn-Cr, Ti-Mn-Mo, and Ti-Fe-Mo systems were of interest in that high-strength properties were obtained over a wide range of compositions. Several complex alloys containing small amounts of all five beta-stabilizing elements also had outstanding properties.

Although there were a number of other compositions which might be of equal interest, twelve alloys were selected for more detailed studies of the effects of heat treatments on tensile properties. Tensile and bend properties of hot-rolled sheet from exploratory heats of these alloys are summarized in Figure I. All of the alloys selected except the Ti-15%Cr alloy had tensile strengths of the order of 200,000 psi. The current heat-treatment program is expected to produce improvements in the ductility of these alloys without excessive loss of strength. The Ti-15%Cr alloy was selected because of its excellent bend ductility at the 150,000 psi strength level.

Several of these alloys have been produced as five- and ten-pound ingots and fabricated into sheet and bar stock for testing. During the next year, ingots weighing 20 or more pounds of the most interesting alloys of this group will be melted, fabricated, and evaluated.

The evaluation of the selected alloys was started shortly before the close of the past contract year. Only a limited amount of data is available at this time. The following seven compositions were melted as duplicate one-pound ingots to test the reproducibility of mechanical properties from heat to heat:

Ti-3.5%Cr-3.5%V	Ti-5%Mn-2%Fe
Ti-3.5%Cr-3%Mn	Ti-5%Mo-4%Fe
Ti-2.5%Cr-5%Mn	Ti-5%Cr-1.5%Fe
Ti-5%Mn-2%Mo	

In the as-hot-rolled condition, the mechanical properties of the two heats of each alloy varied considerably. Other specimens from the same heats will be tested in various heat-treated conditions to determine whether such treatments will produce more consistent properties.

Four of the selected alloys, having the nominal compositions listed below, were melted as five-pound ingots, fabricated to sheet, and tested in the as-hot-rolled and annealed conditions.

Ti-3.5%Cr-3.5%V
Ti-2.5%Cr-5%Mn
Ti-1%Cr-1%Fe-3%Mn-1%Mo-1%V
Ti-1%Cr-4%Fe-1%Mn-1%Mo-1%V

In general, the properties of all four heats were within the high strength range in the as-hot-rolled condition, but considerable variations were found in specimens taken from different sections of the same heat. Chemical analyses of these specimens revealed relatively large differences in composition, in some cases. Annealing at 1300°-1400°F followed by air cooling produced much more consistent properties within individual heats. Average properties of the four alloys after annealing at these temperatures are given in Figure II. Specimens annealed at 1300°F had excellent ductility at strength levels of 137,000 to 150,000 psi. Annealing at 1400°F resulted in somewhat higher strengths and much lower ductility. A 1500°F anneal produced relatively poor properties in all of the alloys and erratic test results in two individual alloys.

Heat Treatment of Exploratory Alloys.

While ingots of the selected alloys were being prepared, several groups of exploratory alloys for which sheet material was already available were heat treated and tested to determine the general effects of different thermal cycles on tensile properties.

Sheet specimens annealed at 1300°F and air cooled generally had excellent ductility but relatively low strength. Increasing the annealing temperature to 1400°-1500°F resulted in higher strengths and lower ductility. In some cases, strengths comparable with those obtained in the as-hot-rolled condition were produced by the latter treatments. Solution treatments in the alpha-beta- or beta-phase fields (1300°-1600°F) followed by quenching and subsequent aging in the temperature range 600°-825°F produced high strengths in many alloys, but, in general, ductility was very low. However, some of the alloys, as solution treated, had excellent intermediate strength properties. For example, the Ti-3.5%Cr-3.5%Mn alloy solution treated at 1450°F and water quenched had an ultimate tensile strength of 145,800 psi with an elongation of 24 percent in one inch.

In conjunction with this exploratory heat-treatment program, the effects of quenching media and low-temperature (200°-300°F) aging on the hardness of binary Ti-Cr and Ti-Mn alloys were investigated. The hardness, as quenched, of heats containing 5 to 8 percent of alloying element increased sharply with decreasing cooling rate. It was discovered that the highest hardness values were obtained in what appeared to be single-phase beta structures. The hardness was a function of alloy content and quenching rate. As a result of these observations, the hypothesis was proposed that the beta phase, in this type of alloy, is susceptible to a hardening phenomenon involving a submicroscopic precipitation of the alpha phase. This

"coherency" hardening may occur during cooling at critical rates from the alpha-beta- or beta-phase fields, as well as during artificial aging treatments.

In connection with the above work, it was found that aging occurred at relatively low temperatures in certain of the binary Ti-Cr and Ti-Mn alloys cooled at various rates from the beta field. Hardness increases up to 100 VHN resulted from aging treatments of 45 minutes at 212°F. Similar hardness increases were produced by the heating involved in mounting metallographic specimens in Bakelite. Alloys in the as-hot-rolled condition did not harden appreciably at these low temperatures.

Recrystallization Experiments.

Beta-stabilized titanium alloys develop undesirably large grain sizes when heated in the beta-phase field either during fabrication or subsequent heat treatment. Grain refinement of such alloys by cold working and annealing is desirable from a physical property standpoint. Experiments with a series of binary Ti-Cr alloys indicated that those containing 5 percent or less of chromium could be recrystallized into a fine-grained structure by suitable thermal-mechanical treatment. However, heats containing 10 to 30 percent chromium could not be recrystallized by conventional cold rolling and annealing techniques.

Isothermal-Transformation Studies.

A study of the isothermal transformation characteristics of three binary Ti-Cr alloys was carried out to provide basic information which has contributed to the proper understanding of the heat-treatment reactions.

Time-temperature-transformation curves were established for three binary Ti-Cr alloys containing 2.35, 4.64, and 7.54 percent chromium, respectively. The initiation of proeutectoid alpha separation was very rapid in all of the alloys. This phase was detected at transformation times of the order of 10 to 30 seconds at all temperatures investigated. The compound $TiCr_2$ appeared in all of the alloys in about 1 hour at temperatures of 1022-1112°F (550°-600°C). At 1292°F, initiation of this reaction was prolonged to very long times (more than 100 hours). M_s temperature ranges were established for the 2.35 and 4.64 percent chromium alloys at 1004°-1040°F (540°-560°C) and 932°-968°F (500°-520°C), respectively. The 7.54 percent Cr alloy did not transform to martensite. Hardnesses of isothermally transformed specimens, in general, decreased with increasing transformation temperature and with increasing time at a given temperature.

Welding Studies.

One of the most promising alloys reported early in last year's work was a binary which contained 15 percent chromium. In the first attempt to weld thin sheets of this alloy, it was found that a single-pass heliarc weld could be bent in the as-welded condition over a radius of 3T. This success led to the welding of several other intermediate and high-strength alloys, all of which could be bent either in the as-welded condition or after a heat treatment. The welding studies are being continued on some of the selected alloys mentioned above.

Extrusion Studies.

The contract between Wright Field and Metal Trims, Inc., providing for extrusion studies of titanium was delayed and, therefore, extrusion ingots were not melted at Battelle until late in this contract year. The first shipment of extrusion billets, 4-7/16 inches in diameter by about 6 inches long, has been made.

Battelle Memorial Institute. TR 52-251, Part 1. INVESTIGATION OF COMPRESSIVE-CREEP PROPERTIES OF ALUMINUM COLUMNS AT ELEVATED TEMPERATURES. R. L. Carlson, A. D. Schwope. AF 33 (038)-9542. September 1952. PB 122199. Order from LC, Mi $4.50, Ph $13.80.

An experimental investigation of the behavior of 24S-T4 (stabilized) aluminum columns at three elevated temperatures has been conducted. Tests were performed on long- and short-hinged-end columns of five slenderness ratios. By using an adjustable end eccentricity, it was possible to fix the eccentricity for a column of a given slenderness ratio and thereby obtain comparable results for different loads. Deflection measurements were taken at the mid-point throughout the duration of each test, and curves of deflection versus time with load as the parameter were obtained for each slenderness ratio. Test results indicate that, for a column of a given slenderness ratio and eccentricity, there is a limiting load below which collapse due to creep will not occur. It is concluded that this lower limit should be considered the limiting or allowable load. It is suggested that an approximate method of the type introduced in the report should be employed to determine this load.

Massachusetts Institute of Technology. TR 52-255. TITANIUM RICH TITANIUM-CHROMIUM-OXYGEN TERNARY SYSTEM. Chih-Chung Wang, Nicholas J. Grant, Carl F. Floe. AF 33(038)-8754. November 1952.

The existence of a ternary phase, Ti_3Cr_3O, has been confirmed. It has a face-centered cubic structure with a lattice constant of 13.80 Kx. This phase does not form directly from the liquid phase. The temperature range in which Ti_3Cr_3O is stable has not been determined, but it is possible that it may transform to alpha or other phases at higher temperatures.

Massachusetts Institute of Technology intends to continue independently the investigation of this system. Inquiries concerning further publications should be referred to Massachusetts Institute of Technology.

Battelle Memorial Institute. TR 52-334. DEVELOPMENT OF TITANIUM-BASE ALLOYS. C. H. Lorig, et al. AF 33(038)-3736. December 1952. PB 112227. Order from LC, Mi $6.00, Ph $20.00.

Suitable heat treatments for the high strength alloys have been developed. By varying the heat treatment it has been possible to obtain tensile strengths of 150,000 psi with an elongation of 25% in one inch. Solution treating at higher temperatures generally increases the strength but with a corresponding loss in ductility. It now seems commercially probable to solution treat, machine, and subsequently age, thus producing alloys with a high strength level.

A new phase, called omega, has been discovered by X-ray diffraction studies. This omega phase seems to be responsible for the loss in ductility, or embrittlement which accompanies an increase in tensile strength upon heat treatment. Results indicate that the omega phase vanishes after a certain time at elevated temperatures. Therefore, the omega phase may be a transition product from beta to alpha. The omega phase is pseudocubic in nature and localized increased concentrations of alloying elements indicate that the omega phase is lower in alloy content than the original alpha. The omega phase definitely appears to be connected with the high hardness characteristics of beta stabilized alloys.

A significant development has been the production of ductile arc welds in alpha-beta alloys; varying degrees of ductility have been obtained in alpha-beta alloys by annealing or tempering after welding.

A program has been initiated whereby industrial concerns will evaluate the newly developed alloys of titanium for large scale usage.

Armour Research Foundation, Illinois Institute of Technology. TR 52-16.

PHASE DIAGRAMS OF THE TITANIUM-ALUMINUM, TITANIUM-CHROMIUM-IRON, AND TITANIUM-OXYGEN ALLOY SYSTEMS. R. J. Van Thyne, E. S. Bumps, H. D. Kessler, M. Hansen. AF 33(038)-8708. December 1952. PB 111327. Order from OTS $2.00.

Partial phase diagrams are presented for the systems titanium-aluminum, titanium-chromium, titanium-iron, titanium-chromium-iron, and titanium-oxygen. All studies are completed except for the titanium-chromium-iron system, which requires further confirmatory work in certain areas. The results are outlined in Section IV of this report (page 78).

TR 52-202.

A PRELIMINARY INVESTIGATION ON THE EFFECTS OF SURFACE TREATMENTS ON THE FATIGUE STRENGTH OF TITANIUM ALLOYS Ti-150A AND RC-130B. Heinrich K. Adenstedt, Frank E. Binns, Lt, USAF, Robert J. Rooney. February 1953.

The evaluation of the effects of various treatments on the fatigue properties of titanium bar stock alloys Ti-150A and RC-130B was made. The various treatments of Ti-150A and their corresponding fatigue endurance limits are as follows:

1. Machined and polished - 68,000 psi
2. Ground - 63,000 to 70,000 psi
3. Ten percent permanently stretched and ground - 54,000 psi (wide scatter of data)
4. Ground and scaled - 56,000 psi
5. Machined notched - 40,000 psi
6. Ground and notched - 21,000 psi

The fatigue strength varied from about 35 to 45 percent of the tensile ultimate strength for the different treatments, except for the notched condition as would be expected. RC-130B gave endurance limits of about 67,000 psi (approximately 45 percent of tensile ultimate strength) for the ground, unnotched condition, and about 24,000 psi for the ground notched material. The wide range of values for the ground Ti-150B alloy and for the 10% stretched and ground Ti-150A alloy may have been due to various degrees of surface cold work, and surface discontinuities, caused by grinding and cold work. In addition, radiography identified tungsten inclusions which were probably a contributing factor. In general, the surface treatment has a marked effect upon the fatigue strength of titanium and its alloys. For the conditions tested, a machined and polished surface produced the optimum fatigue properties.

Curtiss-Wright Corporation, Wright Aeronautical Division, Woodridge, New Jersey. TR 53-26.

INVESTIGATION OF HEAT TREATMENT OF COMMERCIAL TITANIUM BASE ALLOYS. L. Luini. AF 33(038)-23302. April 1953. ASTIA Document No. AD 13773. PB 133695. Order from LC, Mi $4.50, Ph $12.30.

An exploratory survey of the heat treatment response of commercial titanium alloys (Ti-150A, RC-130B, RC-130A and MST-3Al-5Cr alloys) shows a wide range of possible hardness and microstructural characteristics. The hardening is primarily dependent on the solid state transformation of the beta phase. An age hardening reaction which is apparently associated with beta decomposition and precipitation has been shown. Brittleness and notch sensitivity appear to be characteristic of age hardening to high hardness. Low ductility is shown by the MST-3Al-5Cr alloy and the Ti-150A alloy exhibits notch sensitivity. Tensile strengths up to 191,000 psi for Ti-150A, 194,000 psi for RC-130B, and 192,000 psi for MST-3Al-5Cr have been demonstrated. Elongations in the range of 2 to 8% where shown for these high strengths. Stresses imposed by the machining operation appear to lower the observed endurance limit.

Battelle Memorial Institute. TR 53-101.

TITANIUM ALLOYS FOR ANALYTICAL STANDARDS. H. W. Lownie, Jr., D. L. Chase, W. M. Henry. AF 33(600)-6850. May 1953. PB 121107. Order from OTS $0.75.

The purpose of this work was to prepare titanium alloys for tentative chemical standards. Titanium alloys have been melted, chipped, and analyzed to serve as tentative chemical standards. The chemical and spectrographic procedures used are described in detail. Analytical results are close to the intended compositions and also show the individual samples to be uniform within the limits of accuracy of the analytical techniques.

This report covers the melting and the chemical and spectrographic analyses of sixteen ten-pound titanium ingots as specified and listed in Table 1. Analytical methods and results are presented in detail.

Armour Research Foundation, Illinois Institute of Technology. TR 52-245.

TITANIUM ALLOYS FOR ELEVATED TEMPERATURE APPLICATION. William F. Carew, Frank A. Crossley, Harold D. Kessler, Max Hansen. AF 33(038)-22806. May 1953. ASTIA Document No. AD 14003. PB 125211. Order from LC, Mi $6.90, Ph $21.30.

The theoretical aspects of the development of titanium alloys for use at elevated temperatures are presented. The results of a preliminary tensile test survey of binary alloys are given. These results indicate that aluminum is one of the most promising strengtheners of titanium at elevated temperatures. Therefore, titanium-aluminum alloys were used as a base for ternary alloys which were surveyed by means of the hot hardness test. From the results of this survey twelve ternary alloys and two binary titanium-aluminum alloys were selected for further differentiation by creep rupture testing. Some creep rupture results at 425° and 550° are reported and give considerable promise for the development of alloys having equivalent if not better properties than Type 403 stainless steel (13 Cr, balance Fe).

Concurrently with the hot hardness survey programs, stabilization studies were made as an aid in developing heat treatments for these alloys.

Oxidation studies were made on some of the alloys and are reported. The results show that generally the oxidation rate decreases with time. Also, the results indicate that at 550° and 675°C the oxidation resistance of some of the alloys may be as good as that of Type 403 stainless steel.

TR 53-109.

THE TENTATIVE TITANIUM-SILVER BINARY SYSTEM. Henry K. Adenstedt and William R. Freeman, Jr., 1/Lt, USAF. July 1953. ASTIA Document No. AD 18321.

Silver is a beta former when alloyed in small amounts with titanium. There is a large range of solubility of silver in alpha titanium and it reaches a maximum of 9 ± 1 wt.% silver at 1560 ± 7°F, at which temperature an eutectoid reaction, $\beta \rightleftharpoons \alpha$ + TiAg, occurs at 17 ± 0.5 wt.% silver. The beta solid solubility increases to 30.5 ± 1 wt.% silver at 1903 ± 5°F.

Contrary to general opinion, the existence of the compound TiAg (69.25 wt.% Ag) has been established. It is formed at 1903 ± 5°F by the following peritectic reaction: β + Liq. $\rightleftharpoons$ TiAg. The composition limits of the compound

have, as yet, not been determined; however, the compound field appears to be narrow from preliminary observations.

Titanium is soluble in silver in an amount of less than 0.5 wt.% as two phases are present in 99.5 wt.% silver as-cast alloys.

Armour Research Foundation of Illinois Institute of Technology. TR 53-177.
TITANIUM ALLOYS FOR ELEVATED TEMPERATURE APPLICATION. W. F. Carew, F. A. Crossley, H. D. Kessler, M. Hansen. AF 33(038)-22806. August 1953.

Results on the following phases of the investigation are presented: (1) homogenity of the double melted eight-pound ingots, (2) density determinations, (3) structural stability studies, (4) creep rupture testing, (5) room and elevated temperature tensile testing and (6) an evaluation of the effect of exposure to air at elevated temperatures on stress rupture properties. Alloys for which experimental data are reported are: 2% Al, 4% Al, 6% Al, 8% Al, 6% Al-1% Si, 8% Al-1% Si, 8% Al-4% Mo, 6% Al-1, 3 and 5% Ta, 8% Al-1, 3 and 5% Ta and 30% Mo.

Homogeneity of the eight-pound ingots was generally good. The structures developed by the heat treatments used appeared to be stable under the conditions of creep rupture testing. At 550°C for a service life of approximately 1000 hours, the results show that the alloys 6% Al-1% Si, 8% Al-1% Si and 8% Al-4% Mo are superior in creep resistance to stainless steel, Type 410 (12.5% Cr-bal. Fe). Also, at 425°C and probably 550°C for 1000 hours service life, the 6% Al alloy is shown to have superior creep resistance to Type 410. Alloys 6% Al-1, 3, 5% Ta and 8% Al-1, 3, 5% Ta offer little, if any, advantage in rupture strength over their 6 and 8% Al binary counterparts at 550°C and are inferior to the 6% Al-1% Si alloy. Exposure to air at elevated temperatures for 100 hours appears to have little or no effect on the creep rupture properties of the titanium-aluminum-base alloys.

Armour Research Foundation Illinois Institute of Technology. TR 52-335.
TITANIUM PHASE DIAGRAMS. Harold D. Kessler, William Rostoker and Robert J. Van Thyne. AF 33(038)-8708. November 1953.

Using high purity arc melted alloys and micrographic analysis of annealed samples as the principle method of investigation, titanium-rich, partial phase diagrams were determined for the following systems:

Ti-Cr-Fe: The titanium-rich portion of the diagram was studied in detail to 70 weight % titanium. The isotherm at 800°C was determined for the section bounded by Ti, $TiFe_2$, and $TiCr_2$. The solubility of chromium and iron in alpha titanium is less than 1% total alloy content. There is a continuous space of ternary beta solid solution between the Ti-Cr and Ti-Fe systems. A ternary eutectoid, $\beta \rightleftharpoons \alpha + TiCr_2 + TiFe$, occurs at approximately 8% Cr-13%Fe and about 540°C. The beta phase of alloys lying on the low titanium side of a tie line between 7% chromium and 4% iron is retained upon water quenching from the beta space. Hardness data are presented, illustrating the marked effect of heat treatment.

Ti-Al-O and Ti-Al-N: Titanium-rich corners of the systems from 0 to 10% aluminum and 0 to 1% oxygen or nitrogen were investigated. As aluminum, oxygen and nitrogen are alpha-stabilizers, the ternary alpha spaces extend to temperatures well above the transformation temperature of titanium (885°C). Nitrogen is more effective than oxygen in raising the $\beta/\alpha+\beta$ space boundary of the Ti-Al system. Upon water quenching the alloys, the beta phase transforms to alpha prime. Oxygen and nitrogen additions increase the hardness of the Ti-Al alloys.

Ti-Al-C: The phase diagram was determined using alloys containing 0 to 10% aluminum and 0 to 1% carbon. A study of several as-cast carbon master alloys permitted an outline of the Ti-C diagram to be constructed. A high melting compound, TiC, appears to exist over a range of compositions. A eutectic occurs at about 30% carbon between TiC and carbon.

Aluminum raises the temperature of the peritectoid reaction of the Ti-C system, $\beta \rightleftharpoons \alpha + TiC$ (920°C). The maximum solubility of carbon in alpha titanium is increased by aluminum additions from about 0.5% in the binary Ti-C system to 1% at 10% aluminum. Aluminum and carbon increase the hardness of the alpha solid solution.

Ternary Oxide Phases: A family of ternary oxide phases ismorphous with Fe_3W_3C was discovered in which titanium was associated with one of the elements of the first transition series. The phase relationships between these ternary phases were investigated for the Ti-Cr-O, Ti-Fe-O and Ti-Ni-O systems. An isothermal section for the Ti-Mo-O system was constructed.

Armour Research Foundation. TR 53-230.
STUDY OF EFFECTS OF ALLOYING ELEMENTS ON THE WELDABILITY OF TITANIUM SHEET. Herbert M. Meyer, William Rostoker. AF 33(616)-206. May 1954. PB 111885. Order from OTS $2.75.

The reactions of three basic categories of titanium alloys to heliarc welding have been studied. The report covers work performed during the period 15 June 1952 to 15 July 1953. Studies included the influence of preheat-and-postheat-treatments on the mechanical properties of welded specimens. Mechanical properties included bend ductility, tensile strength and elongation, and hardness. Microstructures were examined extensively with a view to recording their relationships to weld behavior. A large number of alloys proved extremely brittle in the as-welded state. In almost every case, a large measure of ductility could be restored by postheat-treatment. Preheat-treatment did not generally improve the ductility of the as-welded state. No single postheat-treatment procedure was found applicable to all alloys. A number of commercial alloys were examined. In particular, the RC-130A alloy was studied in some detail, and brittle welds could be rendered ductile by postheat-treatment.

Battelle Memorial Institute. AFTR 6516, Part 2.
THE TITANIUM-MANGANESE, TITANIUM-TUNGSTEN, AND TITANIUM-TANTALUM PHASE DIAGRAMS. D. J. Maykuth, H. R. Ogden, R. I. Jaffee, J. W. Holladay, J. G. Kura. AF 33(038)-8544. July 1954.

Phase diagrams for the binary systems Ti-Mn, Ti-W and Ti-Ta were determined using both high-purity and commercial titanium.

Manganese shows appreciable solubility in beta titanium, and the beta phase field terminates in a very sluggish eutectoid reaction. Two compounds were found over the alloy range of the Ti-Mn system investigated.

The Ti-W system is characterized, at high temperatures, by a broad beta-plus-tungsten field which originates from a peritectic reaction. At lower temperatures, an alpha-plus-tungsten phase field extends across the diagram as a result of eutectoidal decomposition of the beta phase.

Beta titanium and tantalum are completely isomorphous. Tantalum shows appreciable solubility in alpha titanium. An extensive alpha-plus-beta field exists at low temperatures.

Investigation disclosed that additions at the contaminant level of oxygen or nitrogen to high-purity Ti-Mn alloys have a profound effect on alloy structure.

Data were also obtained on the density, hardness, hot-air oxidation resistance, and mechanical properties of binary titanium-rich manganese, tungsten, and tantalum alloys prepared using iodide titanium.

Battelle Memorial Institute. WADC TR 54-205. DEVELOPMENT OF IMPROVED TITANIUM-BASE ALLOYS. Herbert A. Robinson, Paul D. Frost, Walter M. Parris. AF 33(616)-384. August 1954. ASTIA Document No. AD 53704. PB 130407. Order from LC, Mi $6.90, Ph $21.30.

The research program during the past year was centered around six alloys which had shown considerable promise in earlier work as potential aircraft structural materials. The tensile properties of the alloys, as affected by variations in hot working procedures and heat treatments, were evaluated.

Outstanding properties were obtained in two alloys:

Ti-3Mn-1Cr-1Fe-1Mo-1V
Ti-5Mn-2.5Cr

Several ingots of the Ti-3Mn-complex alloy were prepared. These ingots have been forged and rolled to bar stock for evaluation by several industrial organizations.

Cornell Aeronautical Laboratory, Inc. WADC TR 54-404. EVALUATION OF TITANIUM AIRCRAFT PARTS SEMI-FINISHED PRODUCTS. F. J. Gillig, L. W. Smith. AF 33(616)-471. August 1954. PB 122462. Order from LC, Mi $4.50, Ph $12.30.

A contact survey of the major commercial producers of aircraft engines and aircraft in the United States was conducted for the purpose of determining the difficulties and problems that have been encountered in the fabrication and processing of titanium and titanium alloys for aircraft applications. Thirty-four companies were contacted, some of which were visited several times.

Upon the completion of the contact survey, the results were assembled and analyzed for trends and outstanding difficulties. Three specific problem areas were selected for further evaluation. In order to obtain additional pertinent information on these problems, questionnaires were sent out to the aircraft companies contacted previously who had reported encountering one or more of the problems. These questionnaires were returned together with test data and reports that substantiated the answers contained therein.

All of the above data have been summarized and are presented in a generalized form in this report.

Avco Manufacturing Corp. WADC TR 54-305 Part 1. HANDBOOK ON TITANIUM. Heinrich K. Adenstedt. AF 33(616)-2222. August 1954. PB 111631. Order from OTS $3.00.

Information has been collected on specific properties and procedures of titanium by literature survey and by personal contact with the proper persons and institutions. The data have been evaluated and are condensed in this report. The three major sections of titanium technology covered are: production, physical metallurgy and properties.

University of Michigan. WADC TR 54-112. INTERMEDIATE TEMPERATURE CREEP AND RUPTURE BEHAVIOR OF TITANIUM AND TITANIUM-BASE ALLOYS. Jeremy V. Gluck, James W. Freeman. AF 33(616)-244. September 1954. PB 119041. Order from LC, Mi $6.00, Ph $18.30.

This report summarizes the work performed under Contract No. AF 33(616)-244 during the period from 1 July 1952 to 30 September 1953. The object of the investigation was to determine the relationship between typical structural conditions of representative titanium-base alloys and their mechanical properties in the range from 600° to 1000°F. In order to accomplish this purpose, tensile, short time rupture, and creep properties were determined. The materials studied were: commercially pure titanium, Ti 75A; a commercial alpha-beta alloy, Ti 150A; an experimental stable alpha alloy, 6% Al-94% Ti; and an experimental stable beta alloy, 30% Mo-70% Ti. In addition, hardness, X-ray, and metallographic studies were made.

The results indicate that the single phase α or β alloys possess the best combination of properties at 1000°F. Commercially pure titanium, Ti 75A, exhibited lower properties than the alloys at all test temperatures. Wide ranges of strength variation through heat treatment were found for Ti 150A at 600° and 800°F; however, at 1000°F no essential difference between heat treatments was evident, and the absolute level of strength was quite low. The β alloy, 30% Mo, showed little or no response to prior treatment, but had the best combination of creep and rupture properties at 1000°F of the four materials tested. Both Ti 75A and the α alloy, 6% Al, showed improvement in strengths at 600°F from small amounts of cold working. At 1000°F, however, an optimum amount of cold work, less than 10%, existed, above which properties were lowered through metallurgical instability.

Armour Research Foundation. WADC TR 54-101. STUDIES OF PHASE RELATIONSHIPS AND TRANSFORMATION PROCESSES OF TITANIUM-ALLOY SYSTEMS. Donald J. McPherson, William Rostoker. AF 33(038)-8708. October 1954. ASTIA Document No. AD 56084. PB 135195. Order from LC, Mi $6.90, Ph $21.30.

Time-temperature-transformation charts have been determined for the following titanium-base alloys, which are of interest for high temperature application: 8% Al-6% Mo, 8% Al-4% Mo, 6% Al-6% Fe, 6% Al-6% Mn and 8% Al-4% Cr.

Alloys in the titanium-rich corner of the Ti-Mo-V system were investigated. The locus of minimum total compositions for the complete retention of beta phase on quenching was determined. Aging studies showed that none of the alloys investigated are permanently β stable. The alloys are evaluated from the standpoint of probable ability to retain ductility under elevated temperature service conditions.

Because of the promising behavior of Ti-Al-V alloys at elevated temperatures, the phase relationships in the titanium-rich corner of this system were determined. The investigation covered the space bounded by Ti-22% Al, Ti-11% V, and 600° to 1200°C. Seven isothermal sections and three selected vertical sections are presented and the phase relationships are discussed.

Phase relationships in the titanium-rich corner of the Ti-Al-Si system were studied for similar reasons. The space bounded by Ti-8% Al, Ti-2% Si and 600° to 1200°C was covered. The diagram is represented by six isothermal and four selected vertical sections.

As a prerequisite for the study of possible age hardening titanium base systems, the solubility limits of silicon, boron, beryllium and carbon in the base compositions Ti-4% V, Ti-3% Mo and Ti-2% Cr were investigated. The carbon-containing alloys were not completed.

Information on the other systems is presented in the form of nine partial vertical sections. From this information, selection of the amounts of third component additions and solution treatment temperatures for aging studies will be made.

The formation of two separate and reproducible martensites (both based on a hexagonal lattice) upon quenching binary alloys containing 7 and 9% molybdenum was discovered. No such dual martensitic phases were produced upon similar treatment of Ti-Cr and Ti-Mn alloys.

The method of integrated X-ray diffraction line intensities was proved to be unsuitable for determining the relative amounts of β and α' phases in titanium alloys.

New York University. WADC TR 54-280.
A METHOD FOR RETAINING BETA PHASE IN THE CORE OF PLATES AND RODS OF TITANIUM ALLOYS. P. Herasymenko, J. Winter. AF 33(616)-2259. November 1954.

Twelve 1-kg ingots were prepared having the following nominal compositions: Ti-(6, 8, 10, 12%) Cr, Ti-3%Al-(6, 8, 10%) Cr and Ti-(6, 9, 12%) Mn. The ingots were forged to 1/2-inch and 3/4-inch square bars, which were heat treated using two-step quenching. Microstructures and hardness on the cross-section of bars were investigated.

Evidence is presented that soft beta can be retained in the center of bars and plates by step-quenching in alloys containing 12% Cr in the Ti-Cr series, 10% Cr in the Ti-3%Al-Cr series and 12% Mn in the Ti-Mn series. The outer layers of bars are considerably harder than the core after step-quenching.

Battelle Memorial Institute. WADC TR 54-355.
PRECIPITATION HARDENING AND EMBRITTLEMENT OF HIGH-STRENGTH TITANIUM ALLOYS. H. A. Robinson, M. W. Parris, A. E. Austin, C. M. Schwartz, P. C. Frost. AF 33(616)-445. December 1954. PB 130869. Order from LC, Mi $4.80, Ph $13.80.

Representative alloys of the Ti-Cr, Ti-Fe, and Ti-Mo systems were quenched to retain the beta phase and aged at temperatures in the range of 500° to 1000°F for periods up to 1000 hours. The changes which occurred in the alloys were followed by means of X-ray diffraction, hardness, and electrical resistivity. From this data it was concluded that the decomposition of the retained beta phase occurred in three major stages:

(1) Precipitation of a metastable transition phase, omega, which apparently causes age hardening. At the same time the beta phase seems to become locally enriched in alloy. Hardness increased to very high values during this stage.
(2) The omega phase disappears and the alpha phase makes its appearance. The remaining beta phase becomes highly enriched in alloy. Both hardness and resistivity decrease sharply during this stage.
(3) In the eutectoid systems (Ti-Cr and Ti-Fe), the highly enriched beta decomposes into alpha plus compound. In the Ti-Fe alloys, this stage was accompanied by a further drop in hardness and resistivity. In the Ti-Cr system there is little change in these properties.

The structure of the omega phase has not yet been determined. However, it was shown to be common to at least the majority of beta-stabilized titanium alloy systems, and was apparently not affected by the interstitial impurity content of the alloys.

WADC TR 54-616 Part 1.
HYDROGEN CONTAMINATION IN TITANIUM AND TITANIUM ALLOYS. Ralph J. Kotfila, Harris M. Burte. January 1955. PB 111620. Order from OTS $4.75.

Hydrogen contamination in alpha-beta titanium alloys can cause low ductility in slow strain rate, room temperature tensile tests, and premature brittle fracture in room temperature rupture tests. The combination of stress concentrations and hydrogen contamination can lead to a drastic decrease in the load carrying capacity of these alloys. Another result of hydrogen contamination is increased susceptibility to embrittlement as a result of exposure to stress and elevated temperature.

At the present time, a maximum hydrogen content of 125 parts per million is suggested as a tolerance for aircraft quality alpha-beta titanium forging alloys. Data are presented which show that in the future it will be possible to produce alloys which have much higher tolerances for hydrogen.

Armour Research Foundation. WADC TR 53-182 Part 2.
INVESTIGATION OF THE METALLURGICAL CHARACTERISTICS OF THE 36% ALUMINUM TITANIUM-BASE ALLOY. J. B. McAndrew, D. J. McPherson. AF 33(616)-196. January 1955. PB 121005. Order from OTS $1.00.

The second year of work on a project directed toward an assessment of the potentialities of 36% aluminum titanium-base alloys as aircraft structural materials is reported. Experimental data are given concerning the stress-rupture strength of Ti-36% Al at 800°, 900°, 950° and 1000°C; the room temperature and 1000°C tensile strength of Ti-36% Al; the impact strength of the same alloy at room temperature, 700° and 1000°C; and the oxidation resistance of this material at 1000° and 1200°C in still air environments.

Less extensive data are reported concerning the effects of ternary additions, especially niobium and tantalum, on the properties of the base alloy.

Casting and powder metallurgy techniques applicable to this type of alloy are discussed. Hammer forging was successfully used to prepare material of wrought structure, although this method of forming did not appear useful for the fabrication of finished shapes.

It is concluded that additional research effort directed toward the possibility of modifying the properties of Ti-36% Al to meet the particular demands of various high temperature applications is warranted. Immediate application on other than an experimental basis undoubtedly would be restricted by insufficient development of fabrication processes.

Battelle Memorial Institute. WADC TR 54-487.
THE EFFECT OF GRAIN SIZE ON THE MECHANICAL PROPERTIES OF TITANIUM AND ITS ALLOYS. H. R. Ogden, F. C. Holden, R. I. Jaffee. AF 33(616)-412. March 1955. PB 111881. Order from OTS $3.75.

A study has been made of the effects of grain size on the mechanical properties of commercial-purity titanium, an alpha-beta-titanium alloy, and a metastable beta-titanium alloy. The mechanical properties studied in this research were tensile, notched-tensile, hardness, bend, impact, and fatigue endurance.

For unalloyed titanium, grain size or shape has only a minor effect on the mechanical properties. Yield strengths are decreased slightly for specimens annealed in the alpha-beta range, because of the partition of impurities to the nonmatrix alpha. Impact resistance is highest for the smallest alpha grain sizes. Bend ductility and tensile ductility are excellent for all grain sizes. The fatigue endurance limit at 10^7 cycles is 79 percent of ultimate

strength for unnotched and 42 percent for notched specimens.

The grain size for the metastable beta alloy (Ti-7.5 Cr-7.5Mo) also has only a minor effect on the mechanical properties so long as the alloy is single phase. The presence of alpha in the structure lowered the bend ductility, tensile ductility, and impact resistance. Notched fatigue properties appear to be relatively unaffected by changes in grain size or microstructural condition. The plates of alpha phase in the beta matrix act as stress raisers similar to notches and lower the unnotched endurance limit.

The alpha-beta alloy (Ti-2.5Cr-2.5Mo) was tested in the stabilized condition so that the only variables would be grain size and grain shape. Only minor changes in properties occurred as a result of changing the alpha-beta grain size. Strengths were unaffected by changing grain shape, although the equiaxed alpha-beta specimens had higher tensile ductilities and impact resistance than the acicular alpha-beta specimens. The fatigue endurance limits generally were unaffected by grain size or shape. The unnotched fatigue strength was 34 percent of the ultimate tensile strength, and the notched fatigue strength was 20 percent of the ultimate tensile strength.

Vacuum annealing to remove hydrogen had little effect on the properties because these alloys contained only 30 to 60 ppm hydrogen originally.

Battelle Memorial Institute. WADC TR 54-555. THE EXTRUSION OF TITANIUM. Alvin M. Sabroff, W. Maxwell Parris, Paul D. Frost. AF 33(038)-3736. March 1955. PB 111696. Order from OTS $2.00.

Extrusion tests were conducted on unalloyed titanium and the Ti-3Mn-complex alloy to study the effects of extrusion temperature and die design and to evaluate various lubricants and die materials. Optimum mechanical properties were attained at extrusion temperatures in the alpha-phase region for unalloyed titanium and in the alpha-beta region for the Ti-3Mn-complex alloy.

The surface finish of round bars extruded with flat-face dies was very poor. Improved metal flow and surface finish were obtained with conical dies. The optimum die angle appeared to be about 130 degrees.

Lubricants containing graphite, molybdenum disulfide, and mica produced acceptable surface finishes. The best results were obtained with these materials suspended in a Bentone grease. Titanium carbide, chromium carbide, and cobalt-base alloys showed promise as die materials. The carbides exhibited the least wear and less tendency toward seizing by the titanium. High-quality bars were extruded with these die materials and the Bentone lubricant mixture.

University of Kentucky. WADC TR 54-109. SCALING OF TITANIUM AND TITANIUM ALLOYS. H. W. Maynor, Jr., B. R. Barrett, R. E. Swift. AF 18(606)-60. March 1955. PB 111731. Order from OTS $3.50.

A preliminary study of the scaling characteristics in air of experimentally produced titanium and titanium-base alloys, and commercially produced titanium and titanium-base alloys was conducted at temperatures of 1200°, 1400°, 1600°, and 1800°F (650°, 760°, 870°, and 980°C) in the time range of approximately four to three hundred hours. A total of forty-three titanium-base alloys, one commercial grade of titanium (RS-70), and Type 302 stainless steel were scaled at each of these temperatures; two additional alloys were employed at temperatures of 1200° and 1600°F. Scales formed on a 4.02% Al-Ti alloy were studied in detail and a scaling mechanism was suggested; scales formed on a 4.03% Cr-Ti alloy and a 2.95% W-Ti alloy were studied in less detail. Scaling propensity of titanium-base alloys, relative to titanium and stainless steel, was evaluated on the basis of weight gain with time. Attempts to evaluate scaling propensity on the basis of weight loss with time, through the application of various descaling processes, were unsuccessful; however, results of essentially the same nature were obtained in terms of inches penetration of oxide scale. Isothermal transitions in the parabolic scaling rate were observed for experimentally-produced titanium at 1200°F; transitions were observed, but not studied in detail, for 3.96% Mo-Ti at 1400°F, 1.19% Mo-Ti at 1600°F, and 0.91% Ni-Ti at 1600°F; transitions were indicated, but not studied in detail, for experimentally-produced titanium at 1800°F.

Battelle Memorial Institute. WADC TR 54-609. SPOT-WELDED JOINTS IN TITANIUM ALLOYS AND THEIR BEHAVIOR IN FATIGUE. William H. Kearns, Walter S. Hyler, David C. Martin. AF 33(616)-2005. March 1955. ASTIA Document No. AD 63964. PB 128518. Order from LC, Mi $3.00, Ph $6.30.

A study of the spot welding of titanium and titanium alloys was made to determine the behavior of six-spot weldments under fatigue loading and to compare the fatigue behavior of similar weldments in titanium aluminum, and stainless steel. Commercially pure titanium sheet, commercial titanium -- 7 percent manganese alloy sheet, experimental unalloyed titanium sheet, and Type 321 stainless steel sheet, all of 0.040-inch thickness, were used. Static tension-shear and cross-tension tests were made on single-spot welds in the materials and the tension-to-shear ratios were calculated. For equal gages and spot spacing, six-spot joints in the stainless steel sheet were slightly better in fatigue than similar joints in the titanium materials under similar loading conditions. The joints in the titanium materials were significantly better than similar joints in clad 24S-T and 75S-T aluminum alloys. (The data on the aluminum alloys were developed in previous work.) Under static tension-shear loading, the joints in the titanium materials were stronger than similar joints in the stainless steel and the aluminum alloys.

Armour Research Foundation. WADC TR 54-101, Part 2. STUDIES OF PHASE RELATIONSHIPS AND TRANSFORMATION PROCESSES IN TITANIUM ALLOY SYSTEMS. D. J. McPherson, W. Rostoker. AF 33(616)-2351. April 1955. ASTIA Document No. AD 75186. PB 142056. Order from LC, Mi $4.50, Ph $12.30.

Case Institute of Technology. WADC TR 55-5. TENSILE PROPERTIES AND RHEOTROPIC BEHAVIOR OF TITANIUM ALLOYS AND MOLYBDENUM. E. J. Ripling. AF 33(616)-2223. May 1955. PB 111898. Order from OTS $3.50.

The unnotched and notched tensile properties are described as a function of testing temperature for a series of titanium-nitrogen, and titanium-manganese binary alloys as well as for the commercial alloy, Ti 140 A, and the experimental 3 Mn-complex alloy.

It was shown that the nitrogen embrittles alpha titanium by elevating its transition temperature. This brittleness can be partially eliminated by taking advantage of a rheotropic recovery.

The Ti 140A alloy in the "as-received" condition was high in hydrogen so that a brief investigation of the effects of hydrogen in this alloy was also conducted.

Recrystallization embrittlement in commercial unalloyed molybdenum was found to be a manifestation of rheotropic brittlement.

Battelle Memorial Institute. WADC TR 54-205, Part 2.

DEVELOPMENT OF IMPROVED TITANIUM-BASE ALLOYS. Herbert A. Robinson, W. Maxwell Parris, Paul D. Frost. AF 33(616)-384. June 1955. PB 111988. Order from OTS $2.75.

The heat-treated properties of 24 alpha-beta titanium alloys have been evaluated. Manganese, molybdenum, and vanadium were the principal alloying additions. The strength-ductility relationships of these alloys increased with increasing alloy content up to about 7 percent alloy addition. The Ti-3Mn-complex alloy had room-temperature properties slightly better than those of any of the other alloys tested. However, a Ti-5Mn-2V alloy exhibited excellent elevated-temperature stability at 650°F. A binary Ti-8V alloy was very interesting in that it could be quenched from the beta-phase region and aged to give very good properties. Most alloys are embrittled as a result of being heated into the beta-phase region.

An evaluation of elevated-temperature properties of the Ti-3Mn-complex alloy indicated that the short-time tensile and 100-hour stress-rupture strengths were improved by heat treating initially to a tensile strength of 180,000 psi, instead of annealing to a level of 135,000 psi. However, there was no difference in the 1000-hour stress-rupture strengths for specimens initially heat treated to 135,000- or 180,000 psi strengths.

It was found that pickling to remove the air-contaminated surface of alloy & sheet added large amounts of hydrogen. A process was worked out for descaling and pickling titanium-alloy sheet without appreciable hydrogen pickup. Using this process, the Ti-3Mn-complex alloy sheet has been heat treated to 180,000 psi tensile strength with over 10 percent elongation in 1 inch.

Increasing the hydrogen content of the Ti-3Mn-complex alloy to 260 ppm decreased its tensile ductility and caused premature stress-rupture failures of notched specimens at room temperature. The hydrogen level that caused embrittlement in this alloy is a function of the heat-treated strength of the alloy. As the strength is increased through heat treatment, the hydrogen content required to cause embrittlement is decreased.

Battelle Memorial Institute. WADC TR 54-355, Part 2.

PRECIPITATION HARDENING AND EMBRITTLEMENT OF HIGH-STRENGTH TITANIUM ALLOYS. W. M. Parris, C. M. Schwartz, P. D. Frost. AF 33(616)-445. June 1955. PB 111900. Order from OTS $2.00.

The structure of the metastable transition phase, omega, which is involved in the age hardening of titanium alloys, was determined. It is a complex body-centered-cubic cell of the gamma-brass type containing 54 atoms. The phase contains 3.7 atomic percent of the alloying element. The (100) directions of the omega phase and the parent beta phase are parallel. A tentative mechanism for the age hardening of titanium alloys is proposed.

The formation of the omega phase during aging was found to be inhibited by increasing alloy content. Leaner alloys which transformed to alpha prime upon quenching exhibited some degree of age hardening.

Mechanical-property tests on quenched and aged Ti-4Fe and Ti-8Cr alloys confirmed the severe embrittling effect of the omega phase. Further aging to convert omega to alpha resulted in high strengths with recovery of some ductility.

Attempts to detect the omega phase by metallography or autoradiography were not successful.

Armour Research Foundation. WADC TR 53-230, Part 2.

STUDY OF EFFECTS OF ALLOYING ELEMENTS ON THE WELDABILITY OF TITANIUM SHEET. Herbert M. Meyer. AF 33(616)-206. June 1955. PB 121006. Order from OTS $4.00.

Three interstitial elements, carbon, nitrogen and oxygen, impair the weldability of titanium alloy sheet. To quantitatively evaluate these influences three typical base alloys containing small interstitial additions were prepared and welded. These alloys were:

a binary α alloy: 5 Al-Ti;

a ternary alloy containing an α and a β strengthening element: 7 Al-3 Mo-Ti; and

a binary β alloy: 25 V-Ti.

Weldability was to be determined essentially by bend ductility, while other mechanical properties of unwelded and welded sheet were also investigated. Typical heat treatment cycles were to be selected to establish trends for restoring ductility, or strength, or a combination of "optimum" properties.

In the 5 Al-Ti (an α strengthening alloy), tolerance limits proved to be 0.20% for carbon, 0.15% for nitrogen and 0.25% for oxygen. These interstitial contents strengthened the weldment to various degrees.

No general tolerance pattern emerged for the 7 Al-3 Mo-Ti an alloy containing both an α and a β strengthener. Carbon and nitrogen produced brittle welds under certain specific conditions, but none under others. Oxygen, at the 0.15% level, yields a strong, ductile structure after either a short time, step quench type or a long time, low-temperature anneal.

The 25 V-Ti (not quite a true metastable β alloy) showed little effect from the carbon additions. Nitrogen, which strengthens the weld, did produce erratic ductility results. Oxygen additions displayed no deleterious effect on weldability; as-welded samples were strong and ductile. Oxygen proved to be an excellent weld strengthener and the 0.4% level does not appear to be the limit.

Interesting side developments were the production of a workable and weldable 8 Al-Ti, whereas the 7 Al-Ti was indicated as the forgeable limit previously by many investigators. Also the 15 V-Ti displayed an unorthodox, but promising response to low-temperature isothermal anneals.

Microstructures were methodically used to explain the mechanical properties of the welded zones, and several structural phenomena of major interest were uncovered.

Hardness was not a satisfactory indicator of brittleness in weld zones; it was however, put to good use for controlling the contamination of the α alloy during preparation from the melt.

It may be concluded that interstitial elements in titanium alloy weldments are harmful under some conditions. Where possible, limits of utility have been described. Under some conditions, however, the interstitial elements carbon, nitrogen and oxygen show promise as strengtheners at not too high a cost of ductility, and may be valuable as inexpensive alloying additions already present in the titanium sponge. This fundamental study should help to enlarge and clarify the range of weldable titanium-alloy compositions. At the same time, heat treatments that will restore ductility after embrittlement by the welding cycle are described in terms of the individual phase relationships.

Armour Research Foundation of Illinois Institute of Technology. WADC TR 54-278, Part 1.

TITANIUM ALLOYS FOR ELEVATED TEMPERATURE APPLICATION. W. F. Carew, D. J. McPherson, F. A. Crossley. AF 33(038)-22806. June 1955. PB 119008. Order from LC, Mi $11.10, Ph $49.85.

Results on the following phases of the investigation are presented: (1) effect of carbon, nitrogen and oxygen on the tensile, impact and creep-rupture properties of Ti-6% Al alloy; (2) tensile test evaluation of experimental alloys including binary alloys of stabilizing additions to titanium; (3) effect of vacuum annealing on tensile ductility of alloys containing 8% or more of aluminum; (4) creep-rupture evaluation of alloys; (5) creep evaluation of alloys; (6) evaluation of stability of room temperature tensile properties upon exposure to creep at elevated temperatures; and (7) evaluation of the rolling and welding characteristics of the alloys: 6% Al-0.5% Si, 6% Al-4% V and 7% Al-3% Mo.

The additions carbon, nitrogen and oxygen were found to improve tensile strength of the 6% Al alloy at temperatures to 500°C, the highest test temperature. Additions of 0.3% nitrogen and 0.5% oxygen produce complete embrittlement at room temperature. All impact values were low and it is believed that this was due to hydrogen contamination. The additions improved creep and rupture performance of the 6% Al alloy at 425°C, but at 550°C their contributions were less significant.

In general, the tensile ductility of alloys containing 8% or more aluminum was not improved by vacuum annealing to remove hydrogen.

Of the alloys having better than 10% tensile elongation as heat treated to a stable condition, the 7% Al-3% Mo alloy showed the best creep and rupture strength properties. Also, specimens of this alloy were found to be ductile after exposure to elevated temperature creep conditions for 1000 hours. Specimens of the 6% Al-2% V and 4% V alloys were also ductile after exposure to creep conditions for 1000 hours. The 6% Al-0.5% Si and 1% Si alloys were found to embrittle upon exposure to creep conditions.

The three alloys 6% Al-0.5% Si, 6% Al-4% V and 7% Al-3% Mo were readily rolled. Tensile and bend tests on weldments of these alloys were somewhat disappointing. However, the results indicated that further study of the factors influencing ductility in unwelded and welded sheet should prove fruitful.

Armour Research Foundation of Illinois Institute of Technology. WADC TR 54-278, Part 2. TITANIUM ALLOYS FOR ELEVATED TEMPERATURE APPLICATION. W. F. Carew, F. A. Crossley, D. J. McPherson. AF 33(038)-22806. July 1955. PB 119009. Order from LC, Mi $6.00, Ph $18.30.

The investigations directed toward development of superior titanium-base alloys for elevated temperature applications during the contract period June 1, 1954 to December 31, 1954 are reported.

Testing was completed on a series of binary and ternary alloys not completely evaluated during the previous contract period. None of these alloys provided combinations of properties (creep and creep-rupture, room temperature ductility, and stability upon stress-aging) which were superior to previously developed alloys, particularly Ti-7% Al-3% Mo.

Room and elevated temperature tensile, creep-rupture and stability tests were made for a number of Ti-Al and Ti-Al-Mo alloys containing B, Be or C as dispersion or precipitation strengtheners and for the new ternary compositions Ti-5% Al-5% Mo, Ti-6% Al-4% Mo and Ti-6% Al-6% Mo. Again, none of the alloys demonstrated better combinations of strength, ductility and stability than Ti-7% Al-3% Mo. Boron was apparently added in insufficient quantity (0.04%) because, while ductility and stability were unaffected, negligible strengthening was obtained. Beryllium additions were apparently too high (0.5%) and caused embrittlement.

Alloys containing 5% Al-5% Mo, 6% Al-4% Mo and 7% Al-3% Mo were quite successfully fabricated to 0.060 in. sheet. Good bend properties appeared to depend upon pickling prior to heat treatment. Tensile properties for the three sheet alloys in both the welded and unwelded states are tabulated.

Variations in the previously recommended heat treatment for the Ti-7% Al-3% Mo alloy were studied with the aim of increasing commercial practicability. Solution treating must be accomplished at 800°C or higher to maintain adequate ductility. Four hours are adequate at 850°C, but longer periods of time are necessary as the temperature is lowered.

The notched specimen fatigue endurance limit of Ti-7% Al-3% Mo ($K_t \cong 1.8$) was determined to be 43,000-45,000 psi.

Studies of the effects of sponge quality, alloy content variations, and forging practice on the properties of Ti-7% Al-3% Mo were made. Increasing the sponge hardness incrementally from 103 to 180 BHN resulted in a steady decrease of room temperature ductility in the alloy. Varying the alloy contents by ± 1% of each addition appeared to have little effect upon the creep and creep-rupture properties of Ti-7% Al-3% Mo. Forging practice proved to be extremely important in controlling the alloy's tensile properties. Specimens were forged initially at 2000°F to varying degrees of reduction and finished in the field at 1650° to 1800°F. There was an excellent correlation between the amount of reduction at the finishing temperatures and the tensile reduction in area of heat treated specimens. However, there was no correlation of microstructures with ductility. A need to consider the entire thermal history was indicated.

Avco Manufacturing Corporation. WADC TR 54-305, Part 2. HANDBOOK ON TITANIUM. Heinrich K. Adenstedt. AF 33(616)-2222. September 1955. PB 111873. Order from OTS $4.25.

Information has been collected on specific properties of titanium and procedures for the treatment and utilization of titanium and its alloys by literature survey and by personal contact with the proper persons and institutions. The data have been evaluated and are condensed in this report. The two major sections of titanium technology covered are: laboratory procedures and fabrication.

Armour Research Foundation. WADC TR 55-147. INCREASING THE RATIO OF MODULUS OF ELASTICITY TO THE DENSITY OF TITANIUM ALLOYS. William H. Graft, William Rostoker, David W. Levinson. AF 33(616)-2355. November 1955. PB 121151. Order from OTS $2.00.

Titanium and its alloys, although possessing many attractive properties, exhibit a somewhat lower elastic moduli to density ratio than other competitive engineering metals. It was the purpose of this investigation to examine alloy additions and structural conditions which might introduce improvement in the elastic modulus to density ratio.

Elastic modulus values were measured by means of a dynamic method employing electrostatic excitation and detection. The effects of variables on the elastic properties were examined at room temperature, 500° and 750°F, and the results evaluated using the elastic modulus (E) and the elastic modulus to density ration (E/ρ) as the definitive criteria.

Unalloyed titanium was found to exhibit an E of 16.2×10^6 psi for the magnesium reduced variety, and a slightly higher value for the iodide type.

The alpha stabilizing elements oxygen and nitrogen

were found to affect the E and E/ρ ratio but slightly at low concentrations. The addition of aluminum, up to 8%, was found to result in marked improvement in E and E/ρ ratio.

The presence of intermediate phases on the E and E/ρ ratio was quite beneficial in some cases. TiC and TiB in their respective binary systems resulted in greatly improved elastic properties. Intermediate phases Ti_5Si_3 and TiBe in their respective binary alloys exhibited only a minor effect. Ti_2Cu produced by eutectoidal decomposition in Ti-Cu alloys up to 11.2% Cu produced no significant increase in E or E/ρ.

A survey of the E and E/ρ ratio for various crystallographic directions in the base plane of a-Ti in a state of preferred orientation was made. The results showed that E and E/ρ were essentially constant for various directions, and the value of E, 15.2×10^6 psi, was somewhat lower than that for randomly oriented material.

The effect of heat treatment was found to influence the elastic modulus simply in terms of the ratios of proportions of alpha and beta.

University of Kentucky. WADC TR 54-109, Part 2.
SCALING OF TITANIUM AND TITANIUM ALLOYS. H. J. Siegel, R. C. Duncan, Jr., R. E. Swift. AF 18 (600)-60. November 1955. PB 121219. Order from OTS $2.75.

A preliminary study of the scaling characteristics in air of experimentally produced titanium and titanium-base alloys was conducted at temperatures of 1200°, 1400°, 1600°, and 1800°F (650°, 760°, 870°, 980°C) in the time range of approximately four to three hundred hours. A total of thirty-six titanium-base alloys were scaled at each of these temperatures. Scales formed on all of the alloys at 1600°F were studied using X-ray diffraction. Scaling propensity of titanium-base alloys relative to unalloyed titanium were evaluated on the basis of weight gain with time. Isothermal transitions were noted for many of the alloys but were not studied in detail. An investigation of gaseous penetration in the commercial materials RC-70, RC-130A, and RC-130B was made at 1600° and 1800°F.

Armour Research Foundation of Illinois Institute of Technology. WADC TR 54-547.
EVALUATION OF HIGH STRENGTH WELDABLE TITANIUM BASE ALLOYS. C. Robert Lillie. AF 33(616)-2321. December 1955. PB 121069. Order from OTS $1.75.

Six alloy compositions were investigated to determine their suitability as high strength weldable sheet to be used at elevated temperature. These compositions were: Ti-4 Al-2 V, Ti-4 Al-4 V, Ti-6 Al-2 V, Ti-6 Al-4 V, Ti-7 Al-4V and Ti-6 Al. Tensile tests were performed on these alloys, after suitable heat treatment, both at room temperature and at 700° and 900°F, and in both welded and unwelded conditions. Bend tests were employed as a measure of the formability of the material. All bend tests were carried out at room temperature on heat treated material in both welded and unwelded conditions. Metallographic structure of the alloys after various heat treatments was correlated with the mechanical properties.

All of the Ti-Al-V alloys were found to provide easily rollable sheet material capable of being heat treated to a strength level competitive with stainless steel at elevated temperatures, on a strength-weight basis. The alloys were weldable, producing ductile welds which required no post-welding heat treatment. By proper heat treatment, a low ratio of yield strength to ultimate strength was obtained, indicative of good formability; subsequent aging raised the strength to a high level.

Good properties were retained in these alloys when prepared of commercial quality sponge and when subjected to commercial heat treatments.

The optimum compositions were found to lie in the composition range of Ti-4 Al-4 V to Ti-6 Al-4 V.

Battelle Memorial Institute. WADC TR 55-310.
THE EFFECT OF GRAIN SIZE AND STRUCTURAL VARIABLES ON THE STABILIZATION OF TITANIUM ALLOYS. Franck C. Holden, Horance R. Ogden, Robert I. Jaffee. AF 33(616)-412. January 1956. PB 121156. Order from OTS $1.50.

Studies have been made on the effects of alloy composition and heat treatment on the thermal stability of titanium alloys. Additions of molybdenum increase thermal stability of an alpha-beta alloy, whereas chromium decreases stability. Eutectoid decomposition products were observed in the microstructures of a Ti-5Cr alloy after 200-hour aging at 800° or 1000°F. Oxygen additives increase strength and lower ductility, without a pronounced effect on thermal stability.

Heat treatments to produce a thermally stable condition are most effective when the alloy has an acicular-type structure. This is most effectively accomplished by starting the stabilizing heat treatment in the beta field, although some improvement was observed when the alloy was originally worked in the beta field and stabilized in the alpha-beta.

Exposure to a stress of 25,000 psi during aging at 600°F did not affect thermal stability.

Stress-rupture tests did not indicate a strain-aging process in the conditions tested.

Cornell Aeronautical Laboratory, Inc. WADC TR 55-164.
A STUDY OF THE TENSILE AND CREEP-RUPTURE PROPERTIES OF FIFTEEN HEATS OF C-110M TITANIUM ALLOY SHEET. Frank J. Gillig, Glen J. Guarnieri. AF 33(616)-2342. January 1956. ASTIA Document No. AD 91903. PB 138990. Order from LC, Mi $4.50, Ph $12.30.

Fifteen heats of titanium alloy RC-130-A (C-110M) have been sampled and tested at room and elevated temperatures. These data are analyzed for reproducibility and relationships between the room temperature and high temperature properties at 500°F and 700°F. The results indicate that a correlation exists between the room temperature strength properties and the yield and ultimate strengths at 500° and 700°F. However, the creep and rupture properties appear to be independent of the short time tensile strength results even at 700°F, which was the temperature used for creep testing.

Battelle Memorial Institute. WADC TR 55-362.
THE COLD EXTRUSION OF TITANIUM. Alvin M. Sabroff, Paul D. Frost. AF 33(616)-2446. February 1956. PB 121267. Order from OTS $1.25.

Cold-extrusion studies were conducted on two grades of unalloyed titanium - AMS 4900 and AMS 4921 - to evaluate the effects of die design and extrusion reduction. By utilizing a fluoride coating and a conventional oil-graphite-molybdenum disulfide lubricant, forward extrusions with reductions of 40, 50, and 60 percent were successfully accomplished on 1-1/2-inch-diameter by 3-inch billets. Conical dies with included angles of 90, 120, and 150 degrees were used for each reduction. Working pressures were comparable to those required for cold extruding steel. For a given die angle, the extrusion pressure and average work of extrusion increased in an

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approximately linear relationship with the percentage reduction.

Metal-flow studies were conducted on split billets inscribed with grids. Deformation by compression and elongation was uniform over the cross sections of the bars, with maximum shear deformation occurring in the outer fibers. The amount of shear deformation increased as the die angle and percent reduction increased.

Work hardening during extrusion was nearly uniform within the bars, the overall hardness variation from center to surface being within 20 Bhn. Increases in strength varying from about 25 to 60 percent were produced under the various extrusion conditions. However, adequate ductility (elongation greater than 10 percent in 1 inch) was maintained.

The extruded bars had a smooth surface finish, with few traces of galling. No measurable die wear occurred, indicating the fluoride coating functioned satisfactorily. Dimensional tolerances on the extruded bars were maintained within 0.001 inch.

Armour Research Foundation of Illinois Institute of Technology. WADC TR 54-546.
PILOT PRODUCTION OF PROMISING ELEVATED TEMPERATURE TITANIUM-BASE ALLOYS. Donald J. McPherson. AF 33(616)-2060. February 1956. ASTIA Document No. AD 91223. PB 136558. Order from LC, Mi $4.50, Ph $12.30.

Melting procedures and equipment to produce ingots weighing up to 100 pounds of promising titanium-base alloys developed under Air Force Contract No. AF 33(038)-22806 are described. In order to furnish ingots of this size, it was necessary to expand the existing arc melting facilities substantially. A non-consumable electrode arc furnace of 20-pound capacity employing a magnetic field to direct the arc was designed and fabricated. The capacity of the consumable electrode furnace previously developed at Armour Research Foundation was increased to a theoretical maximum of 300 pounds of titanium by the incorporation of a continuous casting mechanism and increasing the diameter of the mold. Ingots weighing up to 113 pounds and up to 6 inches in diameter were successfully cast. Titanium-base alloy stock of the following compositions was supplied to the Air Force's contractors: (1) 6% aluminum, (2) 6% aluminum-4% vanadium, (3) 6% aluminum-0.5% silicon, and (4) 7% aluminum-3% molybdenum. It was recommended that the 6% Al-0.5 Si alloy be dropped from the program since it proved to embrittle upon exposure to elevated temperature creep conditions.

Evaluation reports were received from Pratt & Whitney Aircraft Division of United Aircraft, Wright Aeronautical Division of Curtiss-Wright and the General Electric Company, Aircraft Gas Turbine Division. These reports cover only the 6% Al alloy. In general, the 6% Al alloy was found to possess good creep resistance, good weld bend characteristics and satisfactory fatigue properties. With a load of 40,000 psi at 800°F for 300 hours, a creep extension 0.08% was obtained. An as-welded bend ductility of 3.5 T and endurance limits of 55,000 and 60,000 psi were found. The alloy meets several of the requirements originally listed as desirable for titanium-base alloys early in the program.

Crane Company. WADC TR 55-458, Part I.
RELAXATION BEHAVIOR OF TITANIUM ALLOYS. Ernest A. Sticha. AF 33(616)-2400. February 1956. PB 121068. Order from OTS $1.00.

Relaxation resistance of three titanium alloys in the temperature range 400°to 800°F was determined to assist in evaluating possible use of titanium fasteners in aircraft for the purpose of saving weight. Tests performed in the Crane Company Engineering Laboratories showed A-110AT alloy to have the best resistance to relaxation, C-130AM alloy the next best and the 3Mn complex alloy the least. Differences between the alloys are greatest at 800°F and almost disappear at 400°F. All three alloys suffered some loss of impact resistance during testing at 700°and 800°F but the cause of embrittlement was not apparent in the microstructure. Heat treatment of the alpha-beta alloys and cold working of the all-alpha alloy to higher strength properties failed to improve relaxation resistance at the higher temperature levels.

Battelle Memorial Institute. WADC TR 55-454, Part I.
A STUDY OF FACTORS AFFECTING THE UNIFORM ELONGATION OF TITANIUM AND TITANIUM ALLOYS. Frank C. Holden, Horace R. Odgen, Robert I. Jaffee. AF 33(616)-2783. February 1956. PB 121213. Order from OTS $1.00.

The limits of uniform elongation were measured for typical alpha, alpha-beta, and beta titanium alloys. Each alloy was studied in three microstructural conditions over a temperature range from -75°to 300°C.

Highest uniform elongation was obtained for commercial titanium (alpha), whereas that for the beta-quenched Ti-7.5Cr-7.5Mo alloy was low. The presence of massive alpha in the alpha-beta alloys increases their uniform elongation.

The dependence of uniform elongation on temperature is marked particularly in the two-phase alloys. Maximum values were obtained at about 200C.

Measurements of uniform elongation from residual strain agree well with values of σ_{max}; the values of the flow exponent n (from the flow equation $\sigma = B\sigma^n$) are consistently higher.

Syracuse University. WADC TR 55-325, Part I.
THE EFFECTS OF INTERSTITIAL CONTAMINANTS ON THE NOTCH TENSILE PROPERTIES OF TITANIUM AND TITANIUM ALLOYS Pt. I Iodide and Sponge Titanium. Neil J. Feola, Eugene P. Klier. AF 33(616)-2281. March 1956. PB 121335. Order from OTS $2.75.

The notch-tensile properties of sponge titanium contaminated with oxygen, nitrogen and carbons have been determined. The merit rating of sponge titanium in the notch-tensile test does not conform to that in the impact test.

In the small (0.3 inch diameter) specimen the levels of interstitial contamination in unalloyed titanium which may be expected to give a notch-strength ratio of unity at -65°F are as follows:

(Ti-base O_2 = 0.035%)
Carbon 0.4%
Oxygen 0.13%
Nitrogen 0.25%

Limited data for impact tension and sustained load tests are presented and discussed.

Armour Research Foundation. WADC TR 54-278, Pt 3.
DEVELOPMENT OF TITANIUM-BASE ALLOYS FOR ELEVATED TEMPERATURE APPLICATION. William F. Carew, Frank A. Crossley, Donald J. McPherson. AF 33(616)-2853. May 1956. PB 121467. Order from OTS $2.75.

The principal objective of the work reported herein was a determination of the effects on mechanical properties of complexing the α and β phases of a promising $\alpha+\beta$ type alloy, Ti-6Al-3Mo. Tin and zirconium were employed as α complexers and chromium, manganese, and vanadium

were employed as $\downarrow$ complexers.

$\downarrow$ complexing was found to improve creep resistance and rupture strength, while β complexing reduced these parameters below the level of the base composition.

In addition, age hardening characteristics of Ti-Al-Ag alloys were determined. Limited creep-rupture data indicated inferiority to a Ti-6Al binary composition.

Further studies on the nature of embrittlement in binary Ti-Al alloys were carried out and results of these studies are reported.

WADC TR 56-121.
THE COMBINED EFFECTS OF CARBON, OXYGEN, NITROGEN AND HYDROGEN ON THE PROPERTIES OF TITANIUM SHEET WELDMENTS. John F. Rudy, 1/Lt. June 1956. PB 121491. Order from OTS $1.25.

Four series of interstitial alloys were melted and rolled into sheet. The first series contained three levels of hydrogen ranging up to 0.063 weight percent. The second series contained three levels of the elements oxygen and carbon combined, ranging up to 0.200% oxygen and 0.239% carbon. The third series contained oxygen and nitrogen in three levels ranging up to 0.166% oxygen and 0.120% nitrogen. The fourth series contained nitrogen and hydrogen in three levels ranging up to 0.102% nitrogen and 0.088% hydrogen. In addition to these alloys, a base metal ingot which contained no intentional addition was also melted and rolled into sheet.

Inert gas shielded arc welds were made in 1/8 inch sheets of each of the thirteen alloys. The physical properties of these alloys were determined by bend tests, tensile tests, impact tests, hardness measurements and metallographic observations for both the welded and unwelded material. These data will aid in establishing maximum allowable combined percentages of carbon, oxygen, nitrogen, and hydrogen for weldable titanium sheet.

New York University Research Division. WADC TR 56-146.
PROPERTIES OF ACTIVE EUTECTOID TITANIUM ALLOYS. R. F. Bunshah, H. Margolin. AF 33(616)-2766. June 1956. PB 121481. Order from OTS $1.75.

The microstructure and mechanical properties of Ti-5Cu-3Al, Ti-8Cu-3Al, Ti-5Cu-3Al-2Sn and Ti-8Cu-3Al-2Sn alloys were studied. Room-temperature and elevated-temperature properties are quite comparable to those of annealed commercial alloys. A stable alpha-plus-compound structure shows the best combination of strength and ductility. In order to obtain highest ductility it is necessary to avoid long semi-continuous paths of compound. T-T-T curves and microstructural observations are presented.

Microstructure and mechanical properties of binary Ti-Ni alloys were investigated. They are quite similar to those of binary Ti-Cu alloys.

In line with some recent work on steel, nickel and zinc, some preliminary data on a binary Ti-2Cu alloy show a considerable increase in the strength of this alloy without loss of ductility, by a suitable prestrain and anneal treatment. The strengthening may be attributed to sub-boundaries produced thereby.

Syracuse University. WADC TR 55-325 Pt II.
THE EFFECTS OF INTERSTITIAL CONTAMINANTS ON THE NOTCH-TENSILE PROPERTIES OF TITANIUM AND TITANIUM ALLOYS Part II. Alloy Titanium. E. P. Klier, N. J. Feola. AF 33(616)-2281. August 1956. ASTIA Document No. AD 97199. PB 121575.

The notch-tensile properties of alloy titanium contaminated with oxygen, nitrogen and carbon have been determined. In all six alloys have been studied and the effects of the various contaminants on the notch sensitivity have been presented in discussion of the results for each alloy.

Notch sensitivity has been shown to depend on the tensile strength, and becomes potentially severe as the strength exceeds about 150,000 psi. Metallographic structure and alloy content also profoundly affect notch sensitivity in these alloys.

The effects of testing temperature, contamination level, and strain rate on promoting notch sensitivity in the respective alloys are also discussed.

Cornell Aeronautical Laboratory, Inc. WADC TR 55-510.
INVESTIGATION OF STRESS RELIEF PROCEDURES FOR TITANIUM AND TITANIUM ALLOYS. Franklin J. Gillig. AF 33(616)-2688. August 1956. ASTIA Document No. AD 97200. PB 121570.

The causes and effects of residual stresses in titanium aircraft parts were critically examined and a means of reducing these residual stresses to a practical value by thermal treatments was evolved. The purpose of the work was twofold: (a) To present a summary of residual stress effects and attempted residual stress measurements in titanium aircraft parts; (b) To obtain experimental data on the effectiveness of various time-temperature combinations for stress relief of elastically restrained unalloyed and alloyed titanium sheet.

The fundamental principles underlying the build-up of residual stress are reviewed. The difference between micro and micro stresses is discussed and the relationship between these stresses and the properties of the material are pointed out. Methods of relieving residual stresses are outlined. The X-ray diffraction, brittle lacquer and strain relaxation methods of residual stress analysis were attempted. All but the latter were unsuccessful.

The method of setting up simulated residual stresses in a simple beam specimen and measuring their decay with time and temperature is described. Using this method, relaxation tests were made on four titanium compositions: A-55, C-110M, 6 Al-4 V and A-110AT. Three stress levels corresponding to the room temperature elastic limit and approximately 50 and 75% of the room temperature tensile yield strengths were imposed on the test specimens as simulated residual stresses. As a result of these tests, recommended stress relief treatments are given for the above alloys.

WADC TR 56-343.
STABILITY OF COMMERCIAL ALPHA-BETA TITANIUM ALLOYS. Donald A. Wruck, 1/Lt. August 1956. ASTIA Document No. Ad 97214. PB 121655.

An investigation was undertaken to observe the influence of various factors such as temperature, time-at-temperature, stress, and hydrogen contamination on the stability characteristics of commercial alpha-beta titanium alloys, as well as to gain an insight into the nature of the reactions responsible for the ductility loss in an unstable microstructure. Commercial alloys utilized in one phase or another in this program included Ti-150A, Ti-155A, Ti-140A, C-130AM, C-110M, Ti-6Al-4V, RS-140X, and 3Mn Complex. Tests show that in the absence of hydrogen contamination of the alpha-beta type alloys can be rendered quite stable up to the maximum temperatures at which they retain useful strength for stressed applications. The Ti-150A alloy loses a considerable amount of room temperature ductility after being exposed to 800°F for 200 hours and can subsequently be rendered ductile by annealing at 1200°F for 24 hours. Mechanisms of embrittlement are

discussed, as well as the practical significance of stability.

Sylvania Electric Products, Inc. WADC TR 55-454 Pt 2.
A STUDY OF FACTORS AFFECTING THE UNIFORM ELONGATION OF TITANIUM AND TITANIUM ALLOYS. A. J. Opinsky, L. Sama, L. L. Seigle. AF 33(616)-2352. September 1956. ASTIA Document No. AD 97284.

In a study of factors which affect the limit of uniform elongation of titanium and titanium-base alloys, iodide titanium is shown to have the largest uniform elongation. Other materials of the high uniform elongation class generally had low strengths, although the metastable β-alloys did exhibit moderate strength. The much lower values of the sponge alloys (outside of the metastable β-alloy) are ascribed to interstitial contents.

The effects of grain size and grain shape are secondary to those of alloying and temperature of test. Strain-induced decomposition of metastable β is shown to be a suitable means of increasing the elongation at maximum load. The other idea of increasing the uniform elongation by strain-induced age hardening is still untested; the apparently most likely system failed to age-harden, or even strain-age harden. In the anisotropy studies, maximum uniform elongation and divergence between yield and ultimate strengths is found in the rolling direction of the sheet.

WADC TR 54-616 Pt II.
HYDROGEN CONTAMINATION IN TITANIUM AND TITANIUM ALLOYS Part II. Comparison of Various Methods for Hydrogen Analysis. John W. Seeger, 1/Lt., Jack A. Winstead, 1/Lt. October 1956. ASTIA Document No. AD 110412. PB 12176.

Description of various analytical methods for hydrogen determination in titanium and its alloys are presented, with information on operating procedures and costs.

Laboratories using these methods in general are in disagreement, and vary considerably in analytical reproducibility. Material non-uniformity in hydrogen distribution from position-to-position in a sheet examined in relation to experimental error is found to be a significant factor. A suggested experiment for a single sheet disputed between two laboratories is given as a model by which appropriate experiments can be devised to incorporate the sources for disagreement found in this investigation.

WADC TR 54-616 Pt III.
HYDROGEN CONTAMINATION IN TITANIUM AND TITANIUM ALLOYS Part III. Strain Aging Hydrogen Embrittlement in Alpha-Beta Titanium Alloys. Harris M. Burte. October 1956. ASTIA Document No. AD 110439. PB 121786.

It is now well recognized that hydrogen contamination in alpha-beta titanium alloys can lead to sudden, brittle fracture during the use of these materials. The strain aging embrittlement which causes such fracture has been investigated.

Strain aging hydrogen embrittlement in alpha-beta titanium alloys has its greatest effect on mechanical properties measured at slow strain rates. It can cause low ductility in room temperature tensile tests and premature brittle fracture in room temperature rupture tests. Fracture due to this process tends to be intergranular. Metallographic examination of many hydrogen contaminated alpha-beta alloys shows no evidence for a third phase before or after fracture. In at least one alloy, however, a third phase was visible after fracture. Both alloy composition and micro-structure affect susceptibility to strain aging embrittlement. Increasing test temperature seems to decrease the tendency towards embrittlement. Increasing test temperature seems to decrease the tendency towards embrittlement, but increases the rate at which embrittlement can occur. A mechanism for strain aging embrittlement is proposed. Other types of embrittlement which may be caused by hydrogen are mentioned.

Battelle Memorial Institute. WADC TR 56-269.
AN INVESTIGATION OF THE USEFULNESS OF ELECTRON MICROSCOPY IN THE STUDY OF TITANIUM-ALLOY STRUCTURES. Frank C. Holden, Arthur P. Young, Horace R. Ogden, Charles M. Schwartz, Robert I. Jaffee. AF 33(616)-2900. October 1956. ASTIA Document No. AD 110460.

The electron microscope has been found useful in the study of a number of titanium-alloy structures. Those structures that contain fine particles or that otherwise require high magnification and resolution for complete study are candidates for electron microscopy.

Among the subjects investigated, three appeared of particular interest: (1) the decomposition of martensite, (2) the aging process in alpha-beta and beta alloys, and (3) studies of deformation processes in two-phase alloys.

Suitable techniques for electron microscopy already have been developed, but further work is desirable. It seems probable that the electron microscope will take its place among the other useful tools in the research on titanium alloys.

Battelle Memorial Institute. WADC TR 54-355 Pt 3.
PRECIPITATION HARDENING AND EMBRITTLEMENT OF HIGH-STRENGTH TITANIUM ALLOYS. A. J. Griest, J. R. Doig, P. D. Frost, C. M. Schwartz. AF 33(616)-445. November 1956. ASTIA Document No. AD 110583. PB 121690.

The age-hardening behavior of a series of alpha-beta titanium alloy was investigated. Hardness, tensile, and X-ray diffraction data were used in following the course of the aging behavior of alloys of the systems Ti-Mn, Ti-Mn-Al, Ti-V, Ti-V-Al, and Ti-Fe. The data presented show the effects of solution temperature, aging temperature and alloy composition on the aging reaction. Comparison of the data for the nominal alloys Ti-8 Mn and Ti8Mn-2Al, as well as other data, indicate that the effect of aluminum is to cause the peak strength and hardness to be maintained over longer aging times and to apparently weaken the tendency for omega phase formation.

In a separate phase of the research, a survey was made of possible contributing factors to beta embrittlement (the low ductility characteristic of high-strength alpha-beta alloys after fabrication or heat treatment in the beta field). It was noted that beta-embrittled materials generally have a combination of microstructural features, such as coarse prior beta grain size or acicular alpha precipitates, which might be expected to impair ductility, especially at high strength levels.

Cornell Aeronautical Laboratory, Inc. WADC TR 55-458 Pt II.
RELAXATION BEHAVIOR OF TITANIUM ALLOYS. Franklin J. Gillig. AF 33(616)-2340. December 1956. ASTIA Document No. AD 110673. PB 121978.

A relaxation test unit has been designed which provides practically continuous control of the total strain by automatically decreasing the load as the specimen tends

to elongate due to creep. The unique feature of this equipment is the ability to apply the initial load in a matter of seconds so that very little relaxation occurs during loading.

Relaxation tests have been made using this equipment for three titanium compositions: A-70, C-130AM, and A-110AT. The A-70 and A-110AT were heat treated to produce two different grain sizes and the C-130AM was heat treated to three different grain sizes. Relaxation tests were made on the three compositions in all of the microstructural conditions at room temperature, 600, and 800°F. The effect of grain size on relaxation characteristics was found to be much the same as in creep testing where the larger grain sizes possess greater resistance to deformation only at the higher test temperatures.

Battelle Memorial Institute. WADC TR 56-545.
DEVELOPMENT OF A HEAT-TREATABLE TITANIUM ALLOY HAVING ADEQUATE FORMABILITY. Herbert A. Robinson, Andrew J. Griest, Alvin M. Sabroff, Paul D. Frost. AF 33(616)-2901. January 1957. ASTIA Document No. AD 110737. PB 121942.

A series of 27 experimental alpha-beta titanium alloys were melted, forged, rolled to sheet, and evaluated as potential formable and heat-treatable sheet alloys. Formability was evaluated by true stress-true strain tensile data and by bend tests. In general, the solution temperature had greater effect than alloy composition on the relative formability of the alloys in the as-quenched condition. However, several compositions appeared to offer greater promise than the others from the standpoints of formability and ability to be aged, after forming, to a high strength level. A Ti-3Fe-3Mo-2Al alloy was of particular interest. Additional research on this and other alloys is recommended.

Mallory-Sharon Titanium Corporation. WADC TR 56-304.
THE EFFECT OF MICROSTRUCTURAL VARIABLES AND INTERSTITIAL ELEMENTS OF THE FATIGUE BEHAVIOR OF TITANIUM AND COMMERCIAL TITANIUM ALLOYS. Charles B. Dittmar, G. William Bauer, Dillon Evers. AF 33(616)-2922. January 1957. ASTIA Document No. AD 110726. PB 121972.

The effect of microstructure on the fatigue behavior of the commercial alloys Ti-5Al-2.5Sn, Ti-6Al-4V, and Ti-3Mn Complex has been investigated. Microstructure was found to have little effect on the endurance limit of these alloys unless the structure was extremely coarsened or embrittled.

The effect of interstitial contents representative of the basis for commercial titanium specification, i.e., .07% N_2, .20% O_2, .20% C taken singly or grouped together, has been investigated for unalloyed titanium and the commercial alloys Ti-5Al-2.5Sn, Ti-6Al-4V, and Ti-3Mn Complex. Titanium and the three titanium alloys investigated generally show increased or unchanged fatigue life at all of the interstitial contents specified above.

Battelle Memorial Institute. WADC TR 56-597.
STUDIES OF FACTORS AFFECTING THERMAL STABILITY OF TITANIUM-BASE ALLOYS. Frank C. Holden, Horace R. Ogden, Robert I. Jaffee. AF 33(616)-3208. February 1957. ASTIA Document No. AD 110748. PB 131043. Order from OTS $1.75.

Studies were made to determine the factors that influence the stabilization and the thermal stability of alpha-beta titanium alloys. Thermal stability for three commercial alloys MST-6Al-4V, Ti-155A, and C-130AM, was good for three heat-treatment conditions under creep exposure tests through 800°F. Some loss of ductility was observed for the Ti-155A and C-130AM alloys fabricated in the beta field and annealed in the alpha-beta field.

Substitutions of various beta stabilizers in a Ti-5Cr-5Mo alloy were made to check their effects on thermal stability. The active eutectoid formers, nickel, copper, and chromium, were detrimental; thermal stability was improved by substitutions of manganese or molybdenum, and by aluminum additions.

Small amounts of strain, introduced either before equilibration or before stabilization, had no significant effect on thermal stability of a Ti-5Cr-5Mo alloy.

Mallory-Sharon Titanium Corporation. WADC TR 56-580.
THE EFFECT OF VARIOUS HEAT TREATMENT CYCLES UPON THE MECHANICAL PROPERTIES OF TITANIUM ALLOYS WITH VARIOUS INTERSTITIAL LEVELS. Benjamin F. Hadley, G. William Bauer, Dillon Evers. AF 33(616)-2795. March 1957. ASTIA Document No. AD 118118. PB 131009.

The effect of various heat treatments on the mechanical properties of 5/8" diameter bar from four titanium alloys of five interstitial content levels each was determined. Three major types of heat treatment were employed: (1) a solution treatment and age cycle, (2) a step quench cycle and (3) a solution treatment, isothermal transformation cycle. The alloys investigated were Ti-5Al Complex (Ti-5Al-1.7Cr-1.6Fe-1.6Mo), Ti-3Mn Complex (Ti-3Mn-1Cr-1Fe-1Mo-1V), Ti-6Al-4V and Ti-4Al-4Mn. Interstitial levels were (1) low (100 BHN sponge base), (2) high oxygen (.24%), (3) high nitrogen (.07%), (4) intermediate oxygen and nitrogen (.16% and .04% respectively) and (5) high carbon (.2%).

Three hundred hour creep tests were performed on materials of interstitial levels 1, 2, and 4. The elevated temperature tensile properties, room temperature notch strength, and Charpy impact data of material of interstitial level 4 were determined. Beta transi, T-T-T curves and end quench curves were determined for all material.

The effect of section size on the mechanical properties and the heat treatment response of the Ti-5Al Complex alloy was determined.

Armour Research Foundation. WADC TR 53-230 Pt 3.
STUDY OF EFFECTS OF ALLOYING ELEMENTS ON THE WELDABILITY OF TITANIUM SHEET. John F. Rudy, Joseph B. McAndrew, Harry Schwartzbart. AF 33(616)-206. March 1957. ASTIA Document No. AD 118135. PB 131049.

This investigation follows a previous investigation which covered the weldability effects of the interstitials, carbon, nitrogen and oxygen, when added individually to several titanium base alloys. Included in these alloys were: 5 Al-Ti, 7 Al-3 Mo-Ti, and 25 V-Ti. For the present study a fourth composition, 6 Al-4 V-Ti, was added to complete the list of base alloys which were investigated as reported herein.

To 5 Al-Ti, an alpha alloy, were introduced combination of C, N_2, and O_2, intended to total 0.33% interstitial. Each of the five resulting alloys was divided into 3 parts: the first part was vacuum annealed to reduce the H_2 to less than 50 PRM; the second part was hydrogenated to 100 PRM; and the third part was hydrogenated to 200 PRM. Each of the fifteen alloy heat "parts" was then tested mechanically in the following five conditions: as fabricated to 0.063 inch sheet, as-welded, and after three

separate post weld heat treatments. Mechanical testing included tensile and free bend tests, both oriented such that the major loading stresses were transverse to the welding direction. Weldability was determined by these mechanical test results.

A similar program was accomplished with the 6 Al-4 V-Ti $\alpha+\beta$ alloy. Since background were missing, eleven 6 Al-4V-Ti plus interstitial alloys were produced. Each was divided into a low and a high H_2 part. The 22 alloy parts were similarly tested in five conditions as above.

The base 7 Al-3 Mo-Ti $\alpha+\beta$ alloy with only hydrogen added was mechanically tested at 3 hydrogen levels times the five conditions; as fabricated to 0.063 inch sheet, as welded, and after three heat treatments. This same program, with different post weld heat treatments, was applied to the base 25 V-Ti β alloy. The total number of thermal history-interstitial content-alloy conditions testes was over 200.

The results indicated generally that:

The 5 Al-Ti becomes not weld ductile when the N_2+0_2 additions total 0.25%. This was true with both 0.049% C and 0.123% C. Some ductility was indicated in both alloys. However, the 6 inch long welded bend specimens were not able to fold to less than one inch end distance separation without failing. Ductile welds were obtained ($<$1 inch fold) with N_2+0_2 of 0.16% even with moderately high (0.128%) C. A completely brittle alloy was obtained when N_2+0_2 totaled 0.28%.

The 6 Al-4V-Ti alloy becomes weld embrittled at $C+N_2+0_2$ as low as 0.26% when N_2 is 0.11%. If the N_2 is held lower ($<$ 0.09%), a $C+N_2+0_2$ of approximately 0.29% can be tolerated. Nitrogen appears to be a much more effective embrittler than 0_2. Oxygen up to 0.21% does not cause complete loss of ductility and an 0.17% 0_2 alloy was weldable. An 0.16% N_2 (0.256% $C_2N_2+0_2$), however, was brittle.

Case Institute of Technology. WADC TR 57-251 Pt I.

HIGH TEMPERATURE BRITTLENESS IN TITANIUM ALLOYS. Nick Makrides, W. M. Baldwin, Jr. AF 33(616)-3536. June 1957. ASTIA Document No. AD 130847. PB 131381.

An alpha-titanium alloy (A-110-AT) containing 5% Al and 2.5% Sn, and an alpha-beta titanium alloy (Ti-140-A) containing 2% Cr, 2% Fe, and 2% Mo were each prepared (a) free from impurities, (b) with 0.2% O, (c) 0.2% C, and (d) 0.1% N for a program designed to explore strain aging phenomena, high temperature brittleness, and stress-rupture behavior of titanium alloys.

Tensile tests at different strain rates have been made on the alpha-alloy containing 0.1% N. The tests revealed a brittle range at 680°F (360°C) at a strain rate of 0.05 in/in/min. This brittle range was moved to 1020°F (550°C) employing a strain rate of 100 in/in/min. The minima in the ductility vs. temperature curves for the two strain rates when plotted on log strain rate vs. reciprocal absolute temperature coordinates gives a straight line (assumed) having a slope which is parallel to the line defining the diffusion coefficient of nitrogen in alpha-titanium. On the other hand, tests are still to be made on the impurity-free, alpha-alloy to see whether the brittleness occurs in the absence of nitrogen.

A review was made of the various types of brittleness found at elevated temperatures in metals and alloys so that any brittleness encountered in titanium could be compared and identified.

Battelle Memorial Institute. WADC TR 54-616 Pt IV.

HYDROGEN CONTAMINATION IN TITANIUM AND TITANIUM ALLOYS. Part IV: The Effect of Hydrogen on the Mechanical Properties and Control of Hydrogen in Titanium Alloys. D. N. Williams, F. R. Schwartzberg, P. R. Wilson, W. M. Albrecht, M. W. Mallett, R. I. Jaffee. AF 33(616)-2813. September 1957. ASTIA Document No. AD 131088. PB 131516.

A comprehensive investigation of hydrogen in titanium alloys was carried out. Tests were conducted to gain an insight into the mechanism of the slow-strain embrittlement of titanium alloys by hydrogen, and a tentative theory is presented. Eighty titanium alloys were examined in the stabilized condition to determine the effect of composition on the tendency toward hydrogen embrittlement. The effect of microstructural variations on the tendency toward embrittlement was also studied. In addition to studying the effects of hydrogen on the properties of titanium, factors affecting hydrogen pickup by titanium and methods of removing hydrogen from titanium were investigated. Included were investigations of low pressure solubility, degassing methods, and the pickup of hydrogen from various atmospheres.

Armour Research Foundation. WADC TR 54-278 Pt IV.

DEVELOPMENT OF TITANIUM-BASE ALLOYS FOR ELEVATED TEMPERATURE APPLICATION. Frank A. Crossley, William F. Carew, David W. Levinson. AF 33(616)-2853. November 1957. ASTIA Document No. AD 142147. PB 131 593. Order from OTS $1.75.

The objectives of this research program were as follows:

(1) To determine the effect of forging and heat treatment variables on the mechanical properties of the Ti-7Al-3 Mo alloy. Evaluations were made of tensile specimens, both machined from bar stock and cut from plate fabricated in such a manner as to simulate compressor blade forging practice. The purpose of this dual evaluation was to determine what effect the forged skin had on mechanical properties.

(2) To determine the effect of sponge quality on mechanical properties of 7Al-3Mo alloy.

(3) To evaluate the tensile, creep-rupture and stability properties of the alloys Ti-6Al-3Nb, Ti-6Al-3Mo-2Ge and Ti-6Al-3Mo-4Ge.

The program for the evaluation of the Ti-7Al-3Mo alloy encountered considerable difficulties with the result that the work was halted before evaluation was completed. The material with which the program was started was discarded because of substandard ductility which was attributed to high oxygen content. While considerable data were collected on the replacement material, it too proved to have substandard ductility. The cause of the low ductility in the replacement material could not be attributed to off-chemistry of aluminum, molybdenum, carbon, hydrogen, nitrogen or oxygen. The sulfur content was indicated to be 0.034% by analyses conducted at the Foundation and 0.01% by analyses conducted by Mallory-Sharon. While the low ductility of this material cannot with any degree surety be attributed to sulfur, it is a possibility.

The significant findings from the data collected on the low ductility replacement 7Al-3Mo material were the following:

(1) In heat treating, increasing the solution treating temperature followed by water quenching increases the time required to achieve overaging.

(2) The Widmanstätten structure has creep-rupture properties at 1000°F superior to the equiaxed structure.

(3) The "skin" effect (as represented by comparing test specimens machined from bar stock with specimens cut from forged plate) on creep-rupture properties at 1000°F was considered to be masked and perhaps dominated by the "size" effect, i.e., ratio of surface area to volume. The forged specimens which were higher in area-to-volume ratio were found to have lower creep-rupture strength compared to machined specimens. This result was in agreement with findings of other investigators for the size effect.

It is emphasized that all data for the Ti-7Al-3Mo alloy reported herein unless stated otherwise were determined for low ductility materials.

The 6Al-3Nb alloy contained less β than the 6Al-3Mo alloy and was higher in tensile strength, but lower in ductility at room temperature. The 6Al-3Mo-2Ge and 6Al-3Mo-4Ge alloys were significantly stronger at room temperature than 6Al-3Mo. The 6Al-3Mo-4Ge alloy was very low in ductility.

The alloys in order of increasing creep resistance at 800°F were: 6Al-3Nb, 6Al-3Mo, 6Al-3Mo-2Ge and 6Al-3Mo-4Ge. The alloys in order of increasing 500-hour rupture strength at 1020°F were: 6Al-3Nb, 6Al-3Mo-2Ge and 6Al-3Mo and 6Al-3Mo-4Ge. Only the 6Al-3Mo-2Ge alloy showed loss of ductility in stability tests.

Only the 6Al-3Mo-4Ge alloy was superior in creep-rupture properties to 6Al-3Mo. However, the ductility of this alloy is considered too low for practical application.

Denver Research Institute. WADC TR 57-269. PARTITION OF SOLUBLE CARBON IN Ti-6Al-4V ALLOY. Richard D. Seibel, Richard L. Beck, Leonard E. Olds. AF 33(616)-3349. November 1957. ASTIA Document No. AD 142137. PB 131603.

The mechanism by which alpha-beta titanium alloys are enbrittled by small amounts of interstitial-type solutes such as nitrogen, oxygen and carbon can not be fully understood until more is known of the micro-distribution of these dissolved impurities in the two-phase alloys. This report describes the results of an investigation of possible means for determining the micro-distribution of these impurities, particularly in the more complex alloys.

A useful method was developed for measuring micro-distribution of solutes in alloys. This method was completely independent of the presence of other solute elements, whether interstitial or substitutional. In addition, some information is presented regarding the use of micro-autoradiographic techniques to study the micro-distribution of the interstitial type solutes in two-phase titanium alloys.

Armour Research Foundation. WADC TR 57-360. RESEARCH ON THE EFFECTS OF STRESS, STRAIN, AND TEMPERATURE ON THE EUTECTOID DECOMPOSITION OF TITANIUM ALLOYS. Adolph W. Goldenstein, Arthur G. Metcalfe, William Rostoker. AF 33(616)-3394. November 1957. ASTIA Document No. AD 142142. PB 131610.

Three titanium-chromium alloys have been forged in the α-β range to give six systems with controlled amounts of each phase. The isothermal transformation at 400°, 500°, and 600°C of each of these systems was followed by resistivity, X-ray diffraction, elastic modulus measurements, and metallography. The reactions occurring in the transformation were identified where possible. This transformation study was repeated under a stress which produced 1% creep in 1000 hours. An acceleration of four to seven times in the rate of transformation occurred under the action of this stress. Complete tensile test data were obtained at all stages of the transformation and plotted on the T-T-T diagrams. These reveal that the formations of omega and $TiCr_2$ are the embrittling reactions.

The eutectoid temperature in the Ti-Cr system has been determined to be $670° \pm 5°C$.

New York University. WADC TR 57-420. STUDY OF MICRODISTRIBUTION OF INTERSTITIAL ELEMENTS IN TITANIUM BY INTERNAL FRICTION TECHNIQUES. Devendra Gupta, Sheldon Weinig. AF 33(616)-3393. November 1957. ASTIA Document No. AD 142145. PB 131613.

An investigation of the microdistribution of interstitial elements in Ti and Ti binary alloys and its effect on the mechanical properties was initiated. The experimental techniques which were utilized during this period were internal friction measurements and "hard" tensile testing. The overall study can be separated into three separate sections, i.e. room temperature effects, interstitial stress relaxation, and grain boundary effects. The major results of the investigation are as follows:

(1) A study of the solute atom-dislocation interaction of Ti-O alloys was performed. It was found that the behavior of the phenomenon was in accord with the relationship, $C = Co \exp(V/KT)$. The interaction energy between an oxygen atom and a dislocation was calculated to be 0.015 ev.

(2) The oxygen content required to saturate the dislocations was found to be approximately 1.5 atomic percent. It was also observed that the transition from ductile to brittle behavior occurred at the same composition.

(3) It was suggested that the transition phenomenon was due to the adsorption of oxygen atoms at the dislocations. In view of this suggestion it was possible to predict the proper treatment to shift the onset of the embrittling effect to higher oxygen contents. This was demonstrated experimentally and it was possible to increase the ductility of Ti-O alloys.

(4) It was demonstrated that an interstitial stress relaxation phenomenon could be observed in "superpurity" titanium.

(5) The development of the interstitial peak in alpha titanium is shown to be directly related to the atomic size of the substitutional element in solution with the oxygen. The greater the difference in size factor between the solute and the titanium atom, the greater the enhancement of the "interstitial" peak.

(6) It is suggested that the mechanism whereby the substitutional solute atoms enhance the appearance of the interstital peak is as follows: The out-of-size substitutional atom causes a localized distortion of the lattice which effectively destroys the isotropic nature of the octahedral interstitial sites. Hence, one site becomes energetically favorable to another upon the application of an external stress. This causes a stress-induced interstitial relaxation phenomenon which is manifested by the appearance of an internal friction peak.

(7) The effect of oxygen on the grain boundary stress relaxation phenomenon was investigated. The necessary data is now available for the adsorption analysis and microdistribution part of this overall study of interstitial effects in titanium.

Crane Co. WADC TR 57-470.

DETERMINATION OF TESTS FOR HYDROGEN EMBRITTLEMENT OF TITANIUM ALLOYS. Albert H. Fleitman. AF 33(616)-3244. December 1957. ASTIA Document No. AD 142196. PB 131617. Order from OTS $2.75.

The mechanical properties of two commercial alloys, Ti-8 Mn and Ti-4 Al-4 Mn were investigated in the as-received, helium annealed, vacuum annealed, and 150, 225 and 300 ppm hydrogen conditions. The Ti-8 Mn alloy in the form of 1/16 inch sheet and the Ti-4 Al-4 Mn alloy in the form of 1/2 inch round bar were each ordered from three vendors to represent the range of interstitial content found in present production.

Slow room temperature notched and unnotched tensile and tensile stress rupture tests up to 1000 hours were performed on both alloys. Slow room temperature notched and unnotched torsion, bend and bend stress rupture tests up to 1000 hours were made on Ti-4 Al-4 Mn alloy. None of the unnotched specimens in any of these tests failed in a brittle manner. The notched tensile stress rupture tests showed that the stress required for fracture at a given time slightly decreased with increasing hydrogen content. In contrast, the notched bend stress rupture tests showed an increase in stress required for fracture.

Longitudinal and transverse tear tests at 80, 32 and -40°F were made on the Ti-8 Mn alloy sheet in the vacuum annealed and 300 ppm hydrogen condition. No apparent relationship was found between tear test failures and hydrogen content. Pin shear tests on Ti-4 Al-4 Mn alloy specimens containing similar quantities of hydrogen failed with ductile fractures.

Room temperature unnotched slow tensile tests on prestrained and aged specimens showed good ductility in both alloys. Specimens in the vacuum annealed and 300 ppm hydrogen condition were prestrained 5% in tension at room temperature and aged at 500°F for 24 or 48 hours prior to test.

Comparison of results in this investigation with earlier work on hydrogenized specimens of these titanium alloys indicates that brittle fractures would have been expected in both alloys when tested in stress rupture at the 300 ppm hydrogen level.

The University of Michigan. WADC TR 54-112 Pt II.

INTERMEDIATE TEMPERATURE CREEP AND RUPTURE BEHAVIOR OF TITANIUM AND TITANIUM-BASE ALLOYS. Part II: Influence of Microstructures on Creep-Rupture Properties. Jeremy V. Gluck, James W. Freeman. AF 33(616)-244. December 1957. ASTIA Document No. AD 142198.

An investigation was carried out to establish the relationships between types of microstructure and creep-rupture properties of titanium alloys at 600°F to 1000°F. Chemical composition and the influence of heat treatment was investigated to a limited extent for each type of alloy. The data are fairly complete for some alloys and consist of survey test data for others.

Alpha titanium as represented by commercially pure titanium had the lowest strength. Stable alpha alloys (6% Al and 6% Al - 0.5% Si) and stable beta alloys (30% Mo and 50% V) had similar strengths. These alloys had the highest strength at 800°F and 1000°F where creep was a major factor. The alpha-beta alloys (Ti 150A and Ti 155AX) and the meta-stable beta alloys had high strengths through 600°F and for short time periods at 800°F by virtue of strengthening from transition structures from the beta to alpha transformation.

Compositional differences between alloys had rather small effects in most cases. Properties of the alpha-beta and meta-stable beta alloys were markedly influenced by heat treatment except at 1000°F. The other structures were little influenced by heat treatment. Ductility varied considerably between alloys and with heat treatment and test conditions.

None of the alloys were subject to appreciable creep at 600°F and strength was governed by tensile properties for time periods up to 1000 hours. Increasing tensile strength by cold work or by heat treatment permitted application of high stresses which usually caused immediate failure at 600°F when the stress was above the yield strength. This seemed to be a delayed tensile fracture rather than true creep to rupture. Creep and structural stability were the major factors in strength at 800°F and 1000°F.

The University of Michigan. WADC TR 54-112 Pt III.

INTERMEDIATE TEMPERATURE CREEP AND RUPTURE BEHAVIOR OF TITANIUM AND TITANIUM-BASE ALLOYS. Part III: Effects of Hot Rolling, Embrittlement, and Interstitial Elements. Jeremy V. Gluck, James W. Freeman. AF 33(616)-244. December 1957. ASTIA Document No. AD 142227.

Three major factors involved in the properties of titanium alloys at high temperatures were investigated:

(1) A limited study was made of the influence of hot-working conditions on creep-rupture properties at 600°F to 1000°F.

(2) The changes in room temperature tensile test ductility as a measure of embrittlement during creep testing at 75°F to 1000°F were evaluated for a considerable number of specimens of several alloys.

(3) A limited study was carried out on the influence of hydrogen content and the hardness of the sponge (interstitial alloying elements) used in making alloys. Creep-rupture properties and embrittlement during stressed exposure were evaluated for temperatures of 600°F to 1000°F.

WADC TN 57-396.

THE EFFECT OF HEAT TREATMENT ON THE STABILITY AND CREEP RESISTANCE OF A TI-AL-MO ALLOY. Harold L. Gegel, 1/Lt. January 1958. ASTIA Document No. AD 142283. PB 131673.

An investigation was undertaken to observe the influence of heat treatment on stability of a Ti-Al-Mo ternary alloy. Tests show that the instability of the alloy is innocuous, and that transformation during testing helps to maintain the material strength. The creep resistance of the alloy was improved by heat treatment, and the stress-rupture properties were not deteriorated. A mechanism of embrittlement by heat treatment is discussed.

Titanium Metals Corporation of America. WADC TR 57-630.

THE DETERMINATION OF THE EFFECT OF HEAT TREATMENT ON THE ELEVATED TEMPERATURE STRESS-STABILITY OF TITANIUM ALLOYS. G. A. Lenning, M. L. Greenlee, W. M. Parris, H. D. Kessler. AF 33(616)-3638. February 1958. ASTIA Document No. AD 151000. PB 131724.

The effect of duplex solution and age type heat treatments on the properties of one commercial heat each of the Ti-140A (Ti-2Fe-2Cr-2Mo), Ti-155A (Ti-5Al-1.5Fe-1.5Cr-1.5Mo) and Ti-6Al-4V alloys were investigated. The properties studies included tensile, notch tensile, notch-bend impact and elevated temperature stress-stability.

Manufacturing Laboratories, Inc.
WADC TR 58-20.
DEVELOPMENT OF IMPROVED TITANIUM ALLOYS FOR APPLICATION AT ELEVATED TEMPERATURES. Bernard S. Lement. AF 33(616)-3986. March 1958. ASTIA Document No. AD 151029. PB 131 749.

A metallurgical investigation of the embrittling reaction that occurs in binary Ti-Al alloys was carried out in the range of 6 to 12 wt. pct. aluminum. Changes in bend ductility, hardness, precision length, electrical resistance, lattice parameters, and in microstructure that occur on solutionizing and aging were determined. It was found that the embrittling reaction that occurs on aging in the vicinity of 1020°F (550°C) is characterized by a decrease in both specific volume and electrical resistivity as well as a marked change in the character of striations that exist within the primary grains of the alpha solid solution matrix. The observed changes in properties are best explained on the basis that these striations represent the formation of aluminum-rich segregations. Evidence was found that cracking in a bend test occurs more readily along rather than across these striations. Recommendations are given for future work on this problem.

Armour Research Foundation.
WADC TR 54-546, Pt. II.
PILOT PRODUCTION, FABRICATION AND EVALUATION OF PROMISING TITANIUM ALLOYS. Frank A. Crossley, Benjamin R. Rajala, David W. Levinson. AF 33(616)-2060. March 1958. ASTIA Document No. AD 151010. PB 131 978. Order from OTS $1.25.

Approximately 300 pounds of Ti-7Al-3Mo alloy were fabricated to bar stock and distributed to various jet engine manufacturers. About 70 pounds of hot-rolled, centerless-ground bar was sent to Thompson Products, Inc. They were highly successful in manufacturing 140 J-57 7th stage compressor blades from this material. The endurance limit for blades bench-tested at room temperature under completely reversed stress was 75,000 psi, 5000 psi higher than Ti-6Al-4V alloy blades of the same configuration. Tensile properties for specimens cut from blades and heat treated: 1560°F-1/2 hour-air cooled, 1020°F-24 hours, were as follows: 170,000 psi ultimate tensile strength, 167,000 psi 0.2% offset yield strength, 49% reduction in area, and 16% elongation. As a result of the promising preliminary evaluations, Pratt and Whitney Aircraft, Division of United Aircraft, is preparing to engine test these blades.

Johnston and Funk Titanium Corporation was successful in producing, on an experimental basis, wire in sizes down to 0.020 in. diameter from Ti-7Al-3Mo 9/32 in. diameter rod. No difficulty in supplying production quantities is anticipated.

The high strength condition produced by applying the heat treatment: 1800°F-1 hour-water quench, 1000°F-2 hours to equiaxed Ti-7Al-3Mo alloy material was found to be unstable to aging under stress at 800°F.

Tests conducted by Eaton Manufacturing Company indicated that the alloy is stress-relieved by 2 hours at 1300°F.

The Ti-7Al-3Mo alloy shows considerable promise. This is a fact implicit in the production by Cramet, Inc., of 1500 to 2000 pounds of Ti-7Al-3Mo material for sale, and the announcement by Rem-Cru Titanium, Inc., of the availability of experimental quantities of their version of the alloy containing 6-1/2% aluminum and 3-3/4% molybdenum.

Titanium Metals Corporation of America.
WADC TR 57-639.
A STUDY OF THE FACTORS INFLUENCING THE PROPERTIES OF HEAT TREATABLE TITANIUM SHEET ALLOYS. R. S. Richards, D. L. Day, H. D. Kessler. AF 33(616)-3727. March 1958. ASTIA Document No. AD 151061. PB 131 769.

A number of factors were investigated which influence the properties of two titanium sheet alloys, Ti-4Al-3Mo-1V and Ti-2Al-6Mo-2V, in the annealed, solution treated, and aged conditions. Annealed material exhibits a narrow spread between yield and ultimate tensile strengths along with the best bendability. Solution treated sheet possesses a high degree of uniform elongation with bendability that is only slightly inferior to that of annealed material. The high-strength aged alloy sheet exhibits a lower level of ductility, and is stable under stress at temperatures to at least 800°F.

Elevated temperatures lower the strength, decrease uniform elongation, but increase the bendability and local elongation at fracture. Cold work increases the strength in all three conditions of heat treatment, although deformation at elevated temperatures produces a smaller strengthening effect in annealed and aged material as the temperature increases. Temperatures of 400°-600°F initiate the age hardening reaction in solution treated sheet. Cold deformation of solution treated Ti-4Al-3Mo-1V results in increased aged strengths, while Ti-2Al-6Mo-2V exhibits significant losses in final aged strengths after prior cold work in the solution treated condition.

Successful dimpling operations can be performed at elevated temperatures provided proper heating and forming techniques are used. Solution treated sheet from both alloys is amenable to bending, stretch wrapping, joggling, and hydroforming operations at room temperature, as indicated by results of production forming tests. No appreciable dimensional changes occur in the material as a result of aging.

Battelle Memorial Institute. WADC TR 57-694. METALLURGICAL AND MECHANICAL CHARACTERISTICS OF HIGH-PURITY TITANIUM-BASE ALLOYS. Frank C. Holden, Jerry A. Houck, Horace R. Ogden, Robert I. Jaffee. AF 33(616)-3469. April 1958. ASTIA Document No. AD 151125. PB 131 817.

Studies have been made to establish the relationships between thermal history, microstructure, and mechanical properties for high-purity titanium-base alloys. These have included the following alloy systems: Ti-Al-Mo, Ti-O-Mo, Ti-C-Mo, Ti-O-Al-Mo, Ti-O-Mn, Ti-O-Cu, Ti-C-Cu, and Ti-Cu-Mn. Mechanical test data reported include tensile and flow properties, impact behavior over a range of temperatures, hardness, aging, and cooling-rate test data. The basic physical metallurgical principles involved here are discussed.

Armour Research Foundation. WADC TR 58-182. DEVELOPMENT OF A HEAT TREATABLE TITANIUM SHEET ALLOY. C. Robert Lillie, David W. Levinson. AF 33(616)-3320. August 1958. ASTIA Document No. AD 155796. PB 151 279.

The objective of this research program was to develop heat treatable titanium sheet alloys capable of providing 160,000 psi yield strength, 0.2% offset, at room temperature. The material should be easily rolled to light gages, of the order of 0.020 in., possesses excellent formability in the solution-treated condition, and be stable under stress, as heat treated, for at least 500 hours at 600° and 800°F. Good weldability was secondary in desirability to the foregoing requirements.

The program was divided into two parts, designated as Phase I and Phase II. Phase I had as its purpose the screening of fifteen alloys in order to select the four most promising for more intensive investigation in Phase II. Of the fifteen alloys, four were of the alpha-beta type, utilizing the strengthening effect of a precipitation-hardener, beryllium; seven alloys were alpha-beta type, based on well-known successful combinations of aluminum with the beta-formers, manganese, molybdenum, and vanadium, and dependent for strengthening on the substitution of the alpha-formers, tin or zirconium, for part of the aluminum; the remaining three alloys were of the metastable-beta type, containing 2.5% aluminum for alpha strengthening, and sufficient amounts of manganese, molybdenum, or vanadium to produce a mechanically unstable beta phase after suitable heat treatment. The optimum heat treatments for each of these alloys were determined by bend test screening to establish the best solution-treating temperatures, followed by an aging program to fix the temperatures and times of aging which yielded promising hardness values. These combinations of solution treatment and aging were then checked out by tensile testing.

The four alloys selected for work under Phase II were:

Ti-3Al-7Mo-0.25Be
Ti-3Al-2Sn-4Mn
Ti-2Al-2Sn-7Mo
Ti-2.5Al-7Mn

In Phase II of the program these four alloys were evaluated by bend testing and tensile testing, by measurement of uniform elongation in the solution treated condition, and by stress stability testing for 500 hours at 800° and 600°F.

Two alloys, Ti-3Al-7Mo-0.25Be and Ti-2.5Al-7Mn, reasonably fulfilled the objectives of the program with regard to bend properties and tensile properties, and were stable under a stress of 25,000 psi for 500 hours at 800° F; however, neither alloy is weldable.

New York University. WADC TR 58-105. REINVESTIGATION OF THE SYSTEMS Ti-Al-Cr AND Ti-Al-V. AF 33(616)-3619. August 1958. ASTIA Document No. AD 155850. PB 140 867. Order from LC, Mi $4.80, Ph $13.80.

The Ti-Cr and Ti-V systems have been investigated in the range of 2-36% Cr and 2-36% V in the temperature range 500°-1150°C (Cr) and 500°-900°C (V), with both iodide titanium and Bureau of Mines titanium as base material. Conventional and rapid-quenching techniques were employed.

In the Ti-Cr system, the β / $\alpha+\beta$ boundary was lower than that generally obtained by conventional quenching. The β transus exhibited a change in slope at about 750°C for both iodide titanium and Bureau of Mines titanium. The β / β+TiCr boundary seemed to be shifted towards lower chromium content when the Bureau of Mines titanium was used as base. The eutectoid temperature was found to be between 660° and 670°C for iodide titanium base alloys.

In the Ti-V system, the β / $\alpha+\beta$ boundary was in agreement with most of the earlier determinations. Below 650°C it was found to be greatly obscured by the presence of an impurity phase.

Ti-Al-Cr System:

Five tentative isothermal sections of the titanium-rich corner (up to 40% Al and 32% Cr) of the Ti-Al-Cr system have been constructed at 200-degree intervals between 600 and 1400°C. The following ternary reactions appear to take place:

Liq. + $Ti_2Al \rightarrow \beta$ + TiAl	between	1200-1400°C
$\beta + \varepsilon \rightarrow \alpha$ + Ti_2Al	"	800-1000°C
β + $Ti_2Al \rightarrow \alpha$ + $TiCr_2$	"	600- 800°C

A miscibility gap in the β field is produced by the interaction of the β + Ti_2Al and the β + $TiCr_2$ fields. The ε-phase has been encountered microscopically and this phase is believed to be a binary Ti-Al phase occurring in the binary Ti-Al system in the vicinity of 12% Al below approximately 1100°C.

Ti-Al-V System:

The titanium-rich corner of the Ti-Al-V system has been investigated in the region up to 45 wt% Al and up to 35 wt% V and between 700 and 1400°C. Eight isothermal sections at 100- degree intervals are presented.

The phases encountered in this region were those which have been identified as being present or probably present in the binary Ti-Al system, namely α, β, ε, Ti_2Al and TiAl.

Battelle Memorial Institute. WADC TR 58-161. A STUDY OF BETA EMBRITTLEMENT IN HIGH-STRENGTH TITANIUM ALLOYS. Andrew J. Griest, Arthur P. Young, Herbert A. Robinson, Paul D. Frost. AF 33(616)-5007. August 1958. ASTIA Document No. AD 155835. PB 151 278. Order from OTS $2.25.

Studies were conducted to determine the importance of beta grain size as a contributing factor to the loss of ductility that occurs when titanium alloys are heat treated at temperatures above the beta transus. In a second phase of the research a heat-treatment program was carried out in an effort to develop procedures for restoring ductility in beta-embrittled material. To supplement these studies, a microscopic study of deformation in pertinent microstructures was conducted.

As a result of these investigations, it was concluded that:

(a) The primary cause of beta embrittlement is the large grain size which is obtained so rapidly in many alpha-beta titanium alloys when heated into the beta field. Grain-boundary alpha and acicular intragranular alpha in the microstructure are not detrimental if the beta grain size is fine.

(b) The structures resulting from beta treatments are quite stable and cannot be significantly altered in a beneficial way by heat treatment alone.

(c) Certain alloys (such as Ti-16V-2.5Al) show a relatively low rate of beta grain growth. Development of alloys exhibiting this property appears to offer the most promising means of avoiding or minimizing beta-embrittlement effects.

New York University. WADC TR 58-328. DEVELOPMENT OF ACTIVE-EUTECTOID BASE ALLOYS. R. F. Bunshah, H. Margolin. AF 33(616)-3942. October 1958. ASTIA Document No. AD 202501. PB 151 518.

Titanium-copper alloys form an active eutectoid titanium alloy system in which the decomposition of the beta phase to alpha plus compound occurs rapidly. This alloy development program is a study of the effect of stepwise additions of Al and/or Sn and/or Zr to binary Ti-2Cu, Ti-4Cu, and Ti-6Cu alloys. The alloys were forged as far as possible in the $\alpha+\beta$ field and annealed at 750°C for 24 hours to alpha plus compound. The alloys were evaluated by tensile tests at room and elevated temperatures. Limited stress-rupture and stability tests were also carried out. Several alloys showed excellent tensile properties particularly in the range 1000 to 1200°F. A Ti-6Cu-7Al-6Zr alloy was outstanding, showing a tensile strength of 108,900 psi at 1200°F. These alloys show promise of utility in the 1000 to 1200°F range. Instability apparently associated with the Ti-Al phases was encountered.

Battelle Memorial Institute. WADC TR 58-438. AN INVESTIGATION OF THE EFFECTS OF IMPURITIES AND METALLURGICAL VARIABLES ON THE NOTCH SENSITIVITY OF TITANIUM ALLOYS. Frank C. Holden, Richard W. Douglass, Horace R. Ogden, Robert I. Jaffee. AF 33(616)-5007. December 1958. ASTIA Document No. AD 207078. PB 151624.

The notch tensile properties of three commercial titanium-base alloys, A11OAT (Ti-5, 0A1-2.5Sn), Ti-6Al-4V, and Ti-140A (Ti-2Fe-2Cr-2Mo), were investigated at six interstitial levels, four microstructural conditions, and over a range of temperatures from -196 to 200 C. notch sensitivity generally was increased by increased interstitial content, low temperature, and acicular-type microstructures. In addition, sensitivity to slow-strain hydrogen embrittlement was observed in the temperature range near O C at hydrogen levels well below specification limits.

Armour Research Foundation. WADC TR 58-467.
BRAZING TITANIUM SANDWICH CONSTRUCTION. John F. Rudy, Robert M. Necheles, Harry Schwartzbart. AF 33(616)-5357. January 1959. ASTIA Document No. AD 207904. PB 151663.

The purpose of this project was to study the feasibility of fabricating titanium alloy face sheet honeycomb sandwiches by brazing techniques. The program was conveniently divided into several phases: selection of face sheet alloy and brazing alloys, fabricating of sandwiches by various experimental methods, and mechanical evaluation of the resulting panels. The emphasis was placed on the second phase, fabrication processes.

The survey of possible face sheet alloys is discussed in some detail. The result of this survey was the decision to incorporate Mallory-Sharon's MST 16V-2 1/2Al for the alloy for primary consideration as the face sheet in the brazing process experimentation. In addition to the above alloy, Republic Steel's RS 140 (5Al-2 3/4 Cr-1 1/4Fe) was chosen as an alternative alloy for brazing above the beta transus (-1440°F) of the MST alloy. In making these selections consideration was given to producibility in thin-rolled sections, mechanical properties at room and elevated temperatures, and, possibly most important, the compatibility of the heat treatment cycle with the envisioned practical brazing cycle.

An evaluation of 17 brazing alloys as regards wettability, mechanical strength, oxidation resistance, and corrosion resistance resulted in the recommendation of Ag-28Cu-C.2Li and Ag-0.25Mg-0.2Ni-1Li as the best alloys for brazing titanium-cored panels at a brazing temperature of 1500°F. Ag-28Cu-0.2Li was the stronger of the two. These two alloys plus Ag-7Cu-0.2Li and AG-30Cu-10Sn were found to be satisfactory for brazing stainless steel cored panel at 1600°F. Of these four alloys, Ag-28Cu-0.2Li and Ag-7Cu-0.2Li gave the strongest joints.

The process development phase centered around the development of a quarts lamp radiant heat brazement process. The details of the process, as used, and a discussion of suggested modifications and improvements for application of the same principles to large-production panel fabrication, are presented. Although the process development experiments were conducted with titanium alloy face sheet materials, the results are applicable to sandwich panel production in general. The primary advantages of quarts lamp radiant heat over presently used furnace retort techniques have to do with inherent rapid heating and cooling rates, good temperature uniformity and brazing time control, and general cleanliness of the heat source for atmosphere purity purposes. An itemized list of these advantages is as follows:

(1) Good temperature control over a wide temperature range.
(2) Control over temperature uniformity (or desired non-uniformity) by heating apparatus design.
(3) A clean, portable heat source which can be placed within an atmosphere chamber, allowing rapid heating of the specimen directly. This eliminates the need for single-shot retorts as are presently used in furnace brazing.
(4) Low equipment cost.
(5) Adaptability to jigging for adequate faying surface contact and for required contour (flatness or airfoil curvature).
(6) Possibility for adaptation to continuous production, i.e., relative movement between heat source and panel.

The panels which were produced by the furnace retort and quartz lamp radiant heating processes were evaluated mechanically and by exposure of oxidation and salt spray environments. These evaluations provided data for process variables and material decisions.

Chase Brass and Copper Co., Incorporated. WADC TR 58-409.
HEAT TREATMENT RESPONSE, MECHANICAL PROPERTIES AND STABILITY OF TITANIUM SHEET ALLOYS. Eugene Delgrosso, Peter Kuzmenko, Allan Springmeyer, Brian Weldon, A. Richard Zello, James Chafey. AF 33(616)-3585. January 1959. ASTIA Document No. AD 207791. PB 151623.

The effect of various heat treatments on the mechanical properties of two titanium alloys was determined. The two types of heat treatments utilized were: (1) solution treatment and water quench, (2) solution treatment, water quench and age. The alloys, Ti-4Al-3Mo-1V and Ti-15V-2.5Al, were investigated in the form of approximately 0.040" sheet. Three interstitial content levels of each alloy were investigated.

Beta transus temperatures were determined for the alloys. Room temperature mechanical properties were evaluated for the solution treated and aged conditions of the alloys for both standard and notched tensile configurations. Elevated temperature tensile properties were determined. The effect of deformation, via two degress of rolling or stretching, on aging characteristics were ascertained. Three hundred hour creep tests were conducted at 600°F and 800°F. Creep embrittlement was evaluated. A microstructural examination was conducted. Also, the alloy Ti-4Al-1Pb-1Sn-1V-1Zr was prepared in sheet form and some of its mechanical properties were evaluated.

Battelle Memorial Institute. WADC TR 54-616 Part V.
HYDROGEN CONTAMINATION IN TITANIUM AND TITANIUM ALLOYS PART V. HYDROGEN EMBRITTLEMENT. D. N. Williams, F. R. Schwartzberg, R. I. Jaffee. AF 33(616)-5007. February 1959. ASTIA Document No. AD 209378. PB 151778. Order from OTS $2.25.

Extensive investigations intended to provide information relating to the mechanism of hydrogen embrittlement in titanium-base alloys were carried out. Studies included detailed measurements of the effects of temperature, strain rate, and hydrogen content on hydrogen embrittlement; examination of all-alpha and all-beta alloys for susceptibility to hydrogen embrittlement; calculations of probable hydrogen solubility in alpha and beta titanium; and several other informative investigations. An embrittlement mechanism is outlined. Alloys previously prepared and examined to determine the effects of alloy content and microstructure on hydrogen embrittlement

(WADC TR 54-616, Part IV) were further studied by means of notched stress-rupture tests. This work has resulted in some alteration in previously expressed conclusions regarding alloying and microstructural effects.

Nonferrous (Vanadium) Metals

United States Bureau of Mines. TR 5932.
ZIRCONIUM ALLOYS FOR HIGH TEMPERATURE SERVICE. Stephen M. Shelton. W33-038-47-4493-E. June 1949.

Foote Mineral Company. TR 5943.
RESEARCH ON ZIRCONIUM ALLOYS. Felix B. Litton and S. C. Ogburn, Jr. W33-308-ac-20168. March 1950.

United States Bureau of Mines. TR 52-236.
UNITED STATES BUREAU OF MINES ZIRCONIUM ALLOY INVESTIGATION. Earl T. Hayes, R. L. Carpenter, A. D. Cavett, H. Kato, W. L. O'Brien and O. G. Paasche. AF 33(038)-50-1214-E. November 1952. PB 119891. Order from LC, Mi $3.00, Ph $6.30.

Harvard Univ. Mallinckrodt Chemical Lab., Cambridge, Mass. Published in Jour. Chem. Phys., v. 21: 1122, June 1953.
NOTE ON THE VIBRATIONAL SPECTRA OF $SiHCl_3$, SiH_2Cl_2, and SiH_2Br_2. J. A. Hawkins, S. R. Polo and M. K. Wilson. AF 18(600)-590. March 27, 1953.

Measurement of gasious $SiHCl_3$ were made. The absorption spectrum between 450 cm^{-1} and 700 cm^{-1} is plotted on a graph, and the observed frequencies and their assignments are presented. Investigations were also carried out with SiH_2Cl_2 and SiH_2Br_2. Thermodynamic functions are given for SiH_2Cl_2 and fundamental frequencies are given for SiH_2Cl_2 and SiH_2Br_2.

Armour Research Foundation, Illinois Institute of Technology. TR 52-145.
EXPLORATION OF VANADIUM BASE ALLOYS. W. Rostoker, D. J. McPherson and M. Hansen. AF 33(038)-8517. May 1953.

This is an annual report summarizing the results of work directed toward the development of vanadium-base alloys accomplished during the period May 8, 1951 to May 8, 1952. The alloying characteristics of vanadium with twenty-one solute elements were studied in sufficient detail to provide information on solubility limits, first intermediate phases and reaction between the solid solution and the first intermediate phase. The development of hot forging, annealing, scalping and rolling practices has been pursued. A large number of alloys have been successfully forged. The oxidation characteristics of vanadium alloys have been examined. It has been demonstrated that oxidation behavior is primarily controlled by the presence or absence of molten V_2O_5. Several alloy additions appear to raise the melting point of this oxide.

Duke Univ. Microwave Lab. Durham, N. C. Bound with its Quarterly Progress Report No. 1, February 1, 1953-May 1, 1953; ASTIA Document No. AD 16227. Also published in Jour. Chem. Phys. v. 21: 1710-1713, October 1953.
MICROWAVE SPECTRA AND MOLECULAR STRUCTURES OF $HSiCl_3$, CH_3SiCl_3 and $(CH_3)_3SiCl$. R. C. Mockler, J. H. Bailey and W. Gordy. Sponsored jointly Air Force Office of Scientific Research under AF 18(600)497 and The Research Corp. 1953. ASTIA Document No. AD 16227(b).

The spectra were observed with a Stark modulation spectrometer. A 100-kc square-wave modulation was employed with the receiver tuned to the fundamental frequency. In most instances the frequency sweep method was employed, and the lines were displayed on a cathode-ray scope. Line frequencies were measured with a frequency standard monitored by comparison of the lowest frequency in the multiplier chain with the standard 5-mc frequency broadcast by station WWV. The following molecular constants were obtained from measurements of pure rotational transitions in the microwave region: B_o (in mc) of 2472.45 ± 0.04 for $HSi^{28}Cl_3{}^{35}$, 2346.07 ± 0.04 for $HSi^{28}Cl_3{}^{37}$, 1769.84 ± 0.03 for $C^{12}H_3Si^{28}Cl_3{}^{35}$, 1699.79 ± 0.03 for $C^{12}H_3Si^{28}Cl_3{}^{37}$, 2197.44 ± 0.04 for $(C^{12}H_3)_3Si^{28}Cl^{35}$, and 2147.88 ± 0.03 for $(C^{12}H_3)_3Si^{28}Cl^{37}$. When it was assumed that $d_{SiH} = 1.47$, for $SiHCl_3$, d_{SiCl} was 2.021 and the ClSiCl bond angle was 109°22'. For CH_3SiCl_3, d_{SiC} was 1.896 A and d_{SiCl} was 2.021 A. With all bond angles assumed to be tetrahedral and the CH distance to be that in methane d_{SiCl} was 1.87 A and d_{SiCl} was 2.03 A for $(CH_3)_3SiCl$. (ASTIA abstract).

Armour Research Foundation. WADC TR 52-145 Part 2.
EXPLORATION OF VANADIUM-BASE ALLOYS. William Rostoker, Donald J. McPherson, Max Hansen. AF 33(038)-8517. November 1954.

This report describes the results of work performed during the period May 14, 1952 to May 14, 1953. The forgeability of a large number of binary alloys was investigated. While many proved forgeable, the room temperature ductilities of all but vanadium-titanium and vanadium-zirconium alloys were poor. Ternary alloys based on vanadium-titanium were investigated. Elevated temperature tensile properties have shown much promise.

Studies on improvement of the oxidation characteristics of vanadium alloys have proved fruitful; chromium and aluminum additions were found to be particularly effective. A number of alloys have demonstrated sufficient resistance for 100 hours at 700°C.

Limited studies were conducted on the systems vanadium-oxygen and vanadium-nitrogen.

Armour Research Foundation. WADC TR 52-145 Part 3.
EXPLORATION OF VANADIUM-BASE ALLOYS. Albert S. Yamamoto, William Rostoker. AF 33(038)-8517. January 1955.

This is the third annual report summarizing the results of work performed during the period May 15, 1953 to June 14, 1954. The oxidation study on vanadium alloys has been further pursued and the conclusion has been reached that satisfactory oxidation resistance at elevated temperature of vanadium-base alloys cannot be rendered by alloying alone but possibly by electroplating. Major efforts have been directed toward evaluating the elevated temperature tensile and stress-rupture strengths of the alloys under development. Carbon additions have proven beneficial as far as the forgeability is concerned. In certain cases, however, superior tensile ductilities are observed while no effect in stress-rupture behavior results. The current availability of less expensive vanadium-aluminum alloy produced by the alumino-thermic reduction process has promoted a feasibility study of its use. It is found that the alloys made of this are as

competitive in mechanical properties as the equivalent alloys based on the very expensive calcium-reduced vanadium metal.

A range of alloy compositions containing additions of titanium and aluminum have shown stress-rupture behavior which is as good or marginally better than the best titanium-base alloys.

Harvard Univ. Malinckrodt Chemical Lab., Cambridge, Mass. Report No. 9. AFOSR-TN-56-5.

INFRARED SPECTRA AND MOLECULAR STRUCTURES OF SiH_3F, SiH_3Cl, AND SH_3Br. C. Newman, J. K. O'Leane. AF 18(600)590. January 1956. ASTIA Document No. AD 85509. Also published in Jour. Chem. Phys., v. 25: 885-859, Nov. 1956.

The structure of the e-type fundamentals of SiH_3F, SiH_3Cl, and SiH_3Br has been resolved and analyzed. The values of the small amount of inertia thus obtained have been combined with microwave measurements to yield the dimensions of the SiH_3 group in these molecules. It is shown that the differences in the structure of the SiH_3 group are probably within experimental error. Several revisions have been made in the vibrational assignments for SiH_3F and SiH_3Cl. (Contractor's abstract)

Illinois Univ. Dept of Ceramic Engineering, Urbana. Presented at meeting of the Amer. Ceramic Soc., Basic Science Div., Clemson Coll., S. C., Nov. 1-2, 1956.

AN INVESTIGATION OF THE ALLOTROPIC FORMS OF SILICA IN CERAMIC COATINGS (Abstract). J. H. Lauchner. AF 18(603)28. November 1956.

The allotropic forms of silica, present in ceramic coatings due to devitrification or as a result of a direct addition, were identified by thermal strain, x-ray diffraction, and differential thermal analyses of ceramic coated composites. The effective differential coefficients of thermal expansion were determined from thermal strain data and a quantitative estimate of the crystalline content in the coatings was established. (Contractor's abstract)

WADC TN 58-54.

HIGH TEMPERATURE ELECTRICAL INSULATION MATERIALS FOR MISSILES AND AIRCRAFT. Dr. R. N. Evans. April 1958. ASTIA Document No. AD 151183.

A review on the development of special-purpose electrical materials is presented together with new areas of materials development which may profitably be exploited. The environmental factors to which high speed aircraft and missiles are exposed are discussed. A materials program designed to meet these measurements is presented and its relation to programs on end-item development is pointed out.

Bausch & Lomb Optical Co. WADC TR 58-275. Part II.

FABRICATION OF INFRARED TRANSMITTING MATERIALS BY HOT PRESSING TECHNIQUES. Norbert J. Kreidl, Eugene C. Letter, Harold C. Hafner, Dean A. Buckner, J. Raymond Hensler. AF 33(616)-5123. October 1959. ASTIA Document No. AD 233358.

Studies of BaF_2 hot pressing were continued during the past contract period. BaF_2 compacts with good transparency in the visible and infrared were produced under pressures of 20,000 to 60,000 psi at temperatures below 900°C. Emphasis was placed on the use of higher pressures at lower temperatures in order to avoid contaminations due to high temperature reactions and to take advantage of the use of less pure starting material.

The hot pressing of polycrystalline magnesium fluoride was studied over a pressure range of 20,000 to 60,000 psi and at temperatures up to 900°C. Compacts with excellent transmissions have been fabricated.

Preliminary work was done on the hot pressing of a number of other promising materials. Strontium and calcium fluoride were successfully fabricated into compacts with excellent infrared transmissions. Lithium fluoride, LaF_3 and K_2ZrF_6 were also hot pressed. Ferric oxide, MgO, ZnO, Co_3O_4, Cu_2O and CoO. Al_2O_3 were the oxides studied, and in addition, PbS and CdS were hot pressed.

Some further work was done in attempting to clarify the role of voids in the scattering of radiation by a polycrystalline compact. Also, the anomalous absorption bands observed in the transmission of BaF_2 and MgF_2 hot pressed compacts were studied and partially elucidated.

Plastics

TR 4648.

MECHANICAL PROPERTIES OF PLASTIC MATERIALS AT NORMAL AND SUBNORMAL TEMPERATURES. Robert T. Schwartz, Donald A. Shinn, and Ture T. Oberg. June 1941.

TR 4989.

CREEP AND TIME FRACTURE STRENGTH OF PLASTIC MATERIALS UNDER TENSILE STRESSES. Bernard Chasman. July 1943.

TR 4969.

PROPERTIES OF EXTRUDED THERMOPLASTIC PLASTIC TUBINGS. Howard S. Bergen, Jr., 1/Lt. July 1943.

TR 4998.

BEARING STRENGTH OF PLASTIC MATERIALS. R. T. Schwartz and Edward Dugger, Jr. August 1943.

TR 5012.

IMPACT DATA FOR PLASTIC MATERIALS AT VARIOUS TERMPERATURES. Donald A. Shinn. August 1943.

TR 5004.

SHEAR STRENGTH OF PLASTIC MATERIALS. R. T. Schwartz and Edward Dugger, Jr. September 1943.

TR 5062.

VARIATION OF TENSILE STRENGTH AND ELONGATION OF PLASTIC MATERIALS WITH TEMPERATURE. Robert T. Schwartz. December 1943.

TR 5077.

PLASTICS IN AIRCRAFT. Jean B. Cahan. January 1944.

TR 5698.

EFFECT OF OUTDOOR EXPOSURE ON FLEXURAL AND OTHER PROPERTIES OF SOME PLASTIC MATERIALS. John K. Long. May 1948.

Cornell Aeronautical Laboratories, Inc. TR 5686. RAIN EROSION PROPERTIES OF PLASTIC MATERIALS. PARTS 1 and 2. R. T. Schwartz. W33-038-ac-14248, Exhibit "L". May 1948.

Owens-Corning Fiberglas Corp. TR 5748. PROPERTIES OF GLASS FABRIC REINFORCED LOW PRESSURE LAMINATES. C. D. Jones. W535-ac-37069. January 1949.

National Bureau of Standards. TR 5940. FLEXURAL PROPERTIES OF SOME GLASS FABRIC BASE PLASTIC LAMINATES AT ELEVATED TEMPERATURES. B. M. Axilrod and M. A. Sherman. P. O. (33-038)-49-516-E. November 1949.

The Formica Company. TR 5941. DEVELOPMENT AND TEST OF LOW-PRESSURE MOLDED GLASS FABRIC BASE SILICONE PLASTIC LAMINATES. George E. Power. W33-038-ac-19948. February 1950.

Cornell Aeronautical Laboratories, Inc. TR 6190. A STUDY OF THE RAIN EROSION OF PLASTICS AND OTHER MATERIALS. John L. Beal and Norman E. Wahl. W33-038-514. April 1951.

Bjorksten Research Laboratories. TR 6220. Suppl. 1. DEVELOPMENT OF SIZINGS FOR GLASS FABRIC IN POLYESTER-RESIN LAMINATES. Johan Bjorksten, James E. Henning, Luther L. Yaeger, and Robert J. Roth. AF 33(038)-8902. December 1951.

AF Technical Report No. 6220 describes the development of a glass fabric sizing consisting of vinyl trichlorosilane in conjunction with beta chloroallyl alcohol. Glass-fabric polyester-resin laminates utilizing this sizing on the fabric retained 90% or more of their dry flexural strengths after immersion in boiling water as compared to a 60% retention for laminates with commercial chrome complex (Finish 14) sized glass fabric.

Additional compounds were investigated as sizings for Number 181 glass fabric. None of these proved as effective as vinyl trichlorosilane in conjunction with betachloroallyl alcohol.

Pilot plant equipment for continuous processing of glass fabric by both liquid phase and vapor phase application of the silane sizings was constructed and operated and the effects of varying the processing conditions were studied. The properties of laminates from glass fabric treated by either process have been comparable.

The liquid phase processing involves passing the fabric through a 3.5% xylol solution of an equimolar mixture of vinyl trichlorosilane and betachloroallyl alcohol, drying to remove xylol, washing to hydrolyze chlorine remaining on the fabric, and drying. In vapor phase processing, the fabric is sized in a chamber containing vinyl trichlorosilane vapor, washed, and dried.

Shell Development Company. TR 52-5. HIGH STRENGTH EPON LAMINATES. F. C. Hopper and D. W. Elam. AF 33(038)-19587. January 1952. PB 119613. Order from LC, Mi $3.60, Ph $9.30.

Epoxide (EPON) resin glass fabric base laminates have been developed with high mechanical strength properties at room temperature. Initial work was conducted to determine which type of laminating resin, (some of which were liquid and some solid), what type of application procedure, and what type and amount of curing agent, would give the optimum mechanical strength properties. It was found that applying the solid EPON resins 1001, 1004, and 1007, from solution gave more reproducible and higher mechanical strength properties than the liquid EPON resins RN-34 and RN-48.

Laminates fabricated using EPON 1001 with four parts of dicyandiamide per hundred parts of resin and 181-114 glass fabric gave consistent flexural strength values of at least 75,000 psi dry and 62,000 psi wet at room temperature. These epoxide resin, EPON 1001, laminates were cured at a pressure of 25 psi and at a temperature of 165°C (329°F) using four parts of dicyandiamide per hundred parts resin as the curing agent. The fabric was impregnated with a solution of equal weights of solid resin and acetone and dried for 20 minutes at 95°C. It was found that the greatest flexural strengths were obtained with cured laminates of 28 to 32 per cent resin content. Ultimate dry compressive strengths at room temperature, ranging from 57,000 to 74,500 psi, have also been obtained using EPON 1001 resin. Special finishes for the glass cloth resulted in EPON 1001 laminates having flexural strengths above 80,000 psi.

In general, it has been found that EPON resins are capable of producing laminates with higher strength at room temperature than other types of low pressure laminating resins, but the elevated temperature properties appear to be poorer than those of other low pressure laminates. The important point, as far as aircraft structural applications are concerned, is that it appears that edgewise compressive strengths up to twice that of other low pressure laminates may be obtainable.

United States Rubber Company. TR 6602. HEAT RESISTANT LAMINATING RESINS. Robert G. Nelb, Charles H. Alexander, and Paul M. Elliott. AF 33(038)-11821. June 1952.

A heat resistant, low pressure molded, polyester resin has been developed which will produce a glass fabric base laminate with good strength properties at elevated temperatures. Flexural ultimate strengths of the laminate using 181-144 fabric and the new resin designated as Vibrin X-1047 were from 43,000 to 47,000 psi, and from 23,000 to 31,000 psi after 1/2 hour exposures to 300°F and 500°F respectively when tested at these temperatures. A second resin producing laminate having somewhat lower high temperature flexural strengths, but which was self-extinguishing, was developed and designated Vibrin X-1422. Each of these resins utilized the trifunctional monomer, triallyl cyanurate, as the cross linking agent, together with conventionally constructed alkyds.

It was determined that, in order to obtain these properties for short-time exposures, postcures of 3 hours at 500°F, 8 hours at 450°F or larger periods at lower temperatures were required.

The fabrication techniques for these resins are similar to those of other polyester resins except, in order to prevent blistering of the laminate on postcure, it was found that moisture on the fabric, due to the humidity of storage, must be removed before impregnating or that the laminate must be dried by heating

in an oven for 24 hours at 220°F prior to postcuring.

The electrical properties, dielectric constant and loss tangent, at 10,000 megacycles of the Vibrin X-1047 casting and glass fabric base laminates are very good. The room temperature properties, dry, for the casting and laminate meet the requirements for radar use of the Air Force specification covering polyester resins. The loss tangent and dielectric constant increase only slightly with temperatures up to 500°F.

An improved method of preparing triallyl cyanurate in large laboratory batches in yields of 95% has been developed. This method uses sodium hydroxide in place of metallic sodium which has been previously used. The purity and color of the triallyl cyanurate have also been improved provided the starting material, cyanuric chloride, is of good grade.

Cincinnati Testing and Research Laboratories. TR 52-161 (R).
DEVELOPMENT OF HIGH-STRENGTH, HEAT-RESISTANT PHENOLIC LAMINATING RESIN. M. N. Korelitz. W33-038-ac-21090. July 1952.

The development of a high temperature resistant glass base phenolic laminate is described. Flexural ultimate strength values of the laminate with 181-114 glass fabric at 500°F after one-half hour at 500°F pressed at 15-35 psi pressure average 40,000 psi. This is for 1/8 in. thick laminates up to 36 in. x 36 in., the larger size panels being bag molded. On these panels bag molded by North American Aviation, values of 35,000-45,000 psi were obtained at elevated temperatures. The higher values for bag molded panels have been obtained on 36 in. x 36 in. direct compression panels but they have not been obtained consistently to date. On direct compression panels 36 in. x 36 in. values from 25,000-30,000 psi have been obtained consistently at 500°F after 1/2 hr at 500°F. Values up to 55,000 psi can be obtained consistently for small laminates (such as those used in experimental compressor blades for jet engines) made at pressures in the range of 200-300 psi.

Initially these large panels 18 in. x 18 in. x 1/8 in., and 36 in. x 36 in. x 1/8 in., when handled in the same manner as 6 in. x 6 in. x 1/8 in. panels, resulted in sheets with deteriorated surfaces and lowered flexural strength at elevated temperatures, (20-25,000 psi). Since the material is applicable where larger sections with good high temperature resistance is necessary, work was continued in attempting to make satisfactory large panels.

It was found that different methods of impregnation of the resin on the glass and different methods of handling were necessary. Work along these lines has reached the point where the objective of 40,000 psi flexural strength at 500°F after 1/2 hr at 500°F has been obtained on specimens from panels 36 in. x 36 in. x 1/8 in. (Bag molded).

Fabrication techniques for parts of various sizes and configurations are described.

Some data on long-time temperature tests are reported. Some preliminary evaluations of laminates with various finishes on the glass cloth are presented.

The material used in making all panels except those for checking the finishes were made on production size equipment.

WADC TR 52-24.
DEVELOPMENTS CONCERNING AIRCRAFT GLASS FIBER PLASTIC LAMINATES. Gail A. Clark. July 1952. PB 112387. Order from LC, Mi $6.50, Ph $22.50.

Cornell Aeronautical Laboratory, Inc. TR 52-20(R).
A STUDY OF THE RAIN EROSION OF PLASTICS AND METALS. John L. Beal, Roy R. Lapp, and Norman E. Wahl. AF 33(038)-514. September 1952.

Studies of the erosion behavior, as well as the factors influencing the rate of erosion, at 500 mph, of soft, resilient, polymetric materials and hard, relatively inelastic materials such as metals, were carried out. Neoprene base coatings designated as Gates N-79 (N-700-9 with N-300-9 catalyst) and Goodyear 23-56 (both with bostik 1007 primer) are considered to have the most satisfactory rain erosion resistance applied to structural plastics and metals at speeds up to six hundred and fifty miles per hour.

The rain erosion resistance of metals commonly used in exterior aircraft construction was tested. Of those metals, titanium had the best erosion resistance, which may be attributed to its toughness.

Studies of the general nature of the phenomenon of rain erosion indicate that it is one of impact fatigue.

National Bureau of Standards. TR 52-75.
EFFECTS OF LAMINATING PRESSURE ON THE MECHANICAL STRENGTH OF GLASS FABRIC POLYESTER LAMINATES. J. E. Wier and D. C. Pons. P. O. (33-038)-50-1463-E. November 1952. PB 111506. Order from OTS $1.25.

The effects of laminating pressure and mold type on the mechanical strength and related properties of woven glass-fiber reinforced plastics were determined on laminates bonded with three commercial resins and laminated at four pressures and in two types of molds.

The resins used in making the laminates were diallyl phthalate DAP 65/55, made by the Shell Chemical Company, Plaskon 911-11, a polyester made by the Libbey-Owens-Ford Glass Company, and Selectron 5003, a polyester made by the Pittsburgh Plate Glass Company. The glass fabric used was Fiberglas 181-114 made by the Owens-Corning Fiberglas Corporation. Laminating pressures of 1, 10, 30, and 50 lb/in.^{2} were used. Laminating was done in a closed mold, where the resin was confined, and between open platens (free-edge) where the excess resin was free to flow out under pressure.

The finished laminates were tested for flexural strength and modulus, tensile strength and modulus, and compressive strength. The strengths were determined on specimens both in a dry condition and in a wet condition after 30 days' immersion in water. Other tests included specific gravity, resin content, void content, and water absorption.

The flexural, tensile, and compressive strengths of laminates bonded with the three resins generally increase with an increase in laminating pressure. Laminates attained highest strengths at laminating pressures of 30-50 lb/inch^2.

It was observed that although the tensile strength of the laminates increased with an increase in molding pressure, the tensile breaking load remained relatively constant. No consistent trend was found in the effect of laminating pressure on the percent loss of strength due to water immersion.

An increase in molding pressure also generally increased the specific gravity and decreased the voids content of the laminates.

Some effects of resin aging were observed in laminates bonded with Plaskon 911-11 and Selectron 5003 resins.

United States Department of Agriculture. TR 52-183.
ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 BULLETIN, "PLASTICS FOR AIRCRAFT." Donald G. Coleman. AF 18(600)-70. December 1952.

Developments in the program of research in plastics for aircraft conducted by the U. S. Forest Products Laboratory are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Thirteen technical reports issued during the several years this project has been under way are abstracted.

Shell Development Company. TR 52-5, Suppl 1.
HIGH STRENGTH EPON LAMINATES. F. C. Hopper and D. W. Elam. AF 33(038)-19587. December 1952. PB 1196135. Order from LC, Mi $2.40, Ph $4.80.

A laminate made from 181-114 glass fabric and EPON 1001 resin cured with dicyandiamide had a flexural strength of 76,900 psi at room temperature and 76,200 psi at 160°F (after 1/2 hour at 160°F). The compressive strength was 70,200 psi at room temperature.

With EPON laminates of this type, either boiling in water or aging at 95-100% relative humidity and 100°F for one hour caused a greater loss in strength than soaking for 30 days.

Improved Volan finish was the best of four new fabric finishes used in laminating with EPON 1001 and was the only finish better than 114.

Remarkably high strengths at elevated temperatures were obtained with an experimental resin known as EPON X-12100. Laminates made with this resin had a flexural strength above 60,000 psi at 392°F after 30 minutes at that temperature.

Liquid EPON resins 828 and 834 cured with dicyandiamide also produced laminates resistant to temperatures up to 300°F. After 1/2 hour at that temperature flexural strengths up to 44,000 psi were obtained. Aging for 200 hours at 300°F resulted in 52,900 psi flexural strength in one laminate. At higher temperatures, strengths fell off markedly.

Dimethyl cyanamide, a liquid curing agent, used with EPONS 828 and 834 produced laminates having up to 53,100 psi flexural strengths at 392°F. This curing agent is toxic.

Flexural strengths at 300°F after 1/2 hour at 300°F up to 54,400 psi were obtained also in laminates made with mixtures of Plyophen 5015 (a phenolic resin) and EPON 1001, cured with dicyandiamide.

Of four large laminates 1/8" x 38" x 96" made with EPON 1001 at 25 psi, one was of excellent quality and another was considered good. There should be no difficulty in making EPON laminates of any large area.

University of Louisville. TR 52-105.
RAIN EROSION OF MATERIALS. Gordon C. Williams. AF 33(038)-12606. December 1952. ASTIA Document No. AD 1763.

High speed flight through rain causes erosion damage to exterior plastic and metal parts of aircraft, particularly leading edges. This report summarizes the work conducted at the University of Louisville to study rain erosion and suitable materials or coatings to resist rain erosion.

The original objective of this project was to determine the rain erosion properties of some plastic and metal materials at supersonic speeds, but tests were able to be conducted only from average speeds of 250 to 700 mph, employing a water spray to simulate rain.

Neoprene coatings were found to provide good rain erosion resistance for plastic laminates.

Dow Corning Corporation. TR 6223, Suppl. 1.
DEVELOPMENT OF LOW-PRESSURE LAMINATES OF GLASS FABRIC AND SILICONE RESINS. Kenneth R. Hoffman. AF 33(038)-9201 S-1. May 1953.

Methods for the production of low pressure laminates of Dow Corning Silicone Resin 2104 on a commercial scale have been worked out. Data are given on the preparation of laminating stocks, catalysis, preforming, laminating, bag molding, and molding in matched metal dies. The preparation of duct work and tubing are also discussed.

An evaluation of the use of the new glass fabric sizings with silicone resins shows that most can be used with Dow Corning Silicone Resin 2104, but heat cleaning gives the best results because of the consistent properties of the glass.

An evaluation of new resins, new sizes, catalysts, and additives for Dow Corning Silicone Resin 2104 are given.

TR 52-142.
POST-MOLDING SHRINKAGE CHARACTERISTICS OF SOME THERMO-SETTING PLASTIC MOLDING MATERIALS. Steven T. Marshall, 2/Lt., and Charles P. Ellis, Jr. May 1953. PB 119042. Order from LC, Mi $2.70, Ph $4.80.

The dimensional stability as to post-molding shrinkage of commercially available melamine, phenolic and polyester plastic molding materials was was investigated by means of accelerated conditioning procedures which produced shrinkage corresponding to that obtained on parts in service in long-time aging. It is concluded, that of the materials tested, the polyester mineral filled molding material demonstrated the greatest degree of dimensional stability.

Bjorksten Research Laboratories. TR 6220, Suppl. 2.
DEVELOPMENT OF GLASS SIZINGS TO OBTAIN IMPROVED PROPERTIES OF GLASS FIBER LAMINATES MADE WITH MELAMINE, PHENOLIC, SILICONE, AND EPOXIDE RESINS. Johan Bjorksten, Karl Guenther, Howard L. Gottlieb, and Robert J. Roth. AF 33(038)-8902. June 1953.

Various functional organo-silicon derivatives were investigated as glass fabric sizing for improving glass fiber laminates made with melamine, phenolic, silicone and epoxide resins. These sizings were compared with commercially available glass finishes and the vinyl trichlorosilane sizing previously developed for polyester-resin glass fiber laminates. The criteria used for evaluation were the dry and wet flexural strengths of the laminates prepared with these components.

For laminates made with the specified resins, the following results were obtained:

1. Epoxide Resin - Chlorophenyl trichlorosilane and aminophenyl trichlorosilane improved laminate properties significantly.

2. Silicone Resin - chlorophenyl trichlorosilane improved laminate properties slightly.
3. Melamine Resin - aminophenyl trichlorosilane improved laminate properties significantly.
4. Phenolic Resin - none of the organosilicon derivatives were effective in improving the properties of laminates with a 114 finish.
5. Application of vinyl trichlorosilane sizing was ineffective in improving laminates made with these resins.

National Bureau of Standards. TR 53-192, Part 1 (R).
MECHANISM OF RAIN EROSION. PART 1 - IMPACT PRESSURE IN SOLID-LIQUID SPHERE COLLISIONS. Olive G. Engel. D. O. 33(616)-53-9. July 1953.

An equation for the pressure which results on impact of a water sphere is developed. The pressure equation and other results, which can be deduced from the assumptions made, are able to offer an explanation of certain experimental rain erosion data. Specifically, (1) the larger raindrops of a three-inch-per-hour rainfall should be more destructive than the smaller drops of a one-inch-per-hour rain, (2) the diameter of eroded holes at a velocity of 400 miles per hour should lie in about the range which is observed, and (3) on the assumption of a spall type tensile failure glass should be expected to be rain erosion resistant whereas hard plastics and aluminum should fail. The results are also in agreement with the experimentally observed fact that the horizontal wash (which exists first as spray) has a greater velocity than the impacting surface.

National Bureau of Standards. TR 53-192, Part 2.
MECHANISM OF RAIN EROSION. PART 2 - A CRITICAL REVIEW OF EROSION BY WATER DROP IMPACT. Olive G. Engel. Delivery Order No. 33(616)-53-9. August 1953.

A critical review of the literature on the subject of erosion by water drop impact which includes some recent work done in this laboratory has been made. The types of experimental apparatus generally used by the investigators, and the factors which they found determine the extent of the erosion damage, are briefly discussed. Results of microscope and X-ray studies are presented. Theoretical estimates of the impact pressure, the results of piezoelectric pressure measurements, and theories which have been advanced as to the mechanism of the erosion process are reviewed. Questions which are still unanswered, or in regard to which further research should be done, are pointed out.

The views and conclusions of the investigators are in many cases presented in their own words. A large number of the quotations are translations.

U. S. Department of Agriculture. TR 52-183, Suppl. 1.
ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 BULLETIN, "PLASTICS FOR AIRCRAFT." Donald G. Coleman. AF 33(038)-51-4326E. October 1953.

Developments in the program of research in plastics for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1953 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Four technical reports issued during the fiscal year are abstracted.

Furane Plastics Incorporated. TR 53-56.
DEVELOPMENT OF FURANE RESIN GLASS FABRIC BASE PLASTIC LAMINATES. John Delmonte, John Knudsen and Leland Smith. AF 33(600)-17708. November 1953. PB 111418. Order from OTS $1.00.

Furane resins, prepared from abundant non-critical materials were used to fabricate glass fabric base plastic laminates for testing against the requirements of MIL-R-7575 Specification. In this report the influence of various catalysts upon the resin are shown. In the preparation of 1/8th inch thick laminates, furane resins activated with acid catalysts required post curing to achieve good physical properties. However, one proprietary catalyst, Reagent CM, yielded laminates with good physical properties without the necessity of post cure. No. 181 glass cloth with Garan finish was used exclusively. Differences are brought out between a stiff and soft finish.

To attain optimum results, laminating techniques were carefully evaluated upon small (6 in x 8 in) laminates including: precuring (optimum was 90 minutes at room temperature); storage or closed assembly (permissible up to 24 hours); contact time at laminating temperature (30 to 60 seconds was desirable); and breathing (generally within 30 to 60 seconds). The techniques required coordination to achieve best properties.

Low molecular weight and high molecular weight furane resins were examined, and Resin XL was developed in an attempt to meet MIL-R-7575 Specification requirements. Modifications with phenolic resins also showed promise. Flexural strengths of 60 to 65,000 psi and edgewise compressive strengths of 35 to 40,000 psi appeared readily obtainable on small laminates.

Laminates of large area (2 ft x 2 ft) gave lower and less consistent values for strength properties than the smaller laminates. The area in the center portion of the large laminates showed lower test values than those specimens nearest the edges.

Battelle Memorial Institute. TR 53-491.
HIGH-TEMPERATURE CREEP-RUPTURE PROPERTIES OF GLASS-FABRIC-PLASTIC LAMINATES. John VanEcho, Gale R. Remely and Ward F. Simmons. AF 33(038)-10818. December 1953. PB 121082. Order from OTS $1.50.

Creep and creep-rupture tests, in both tension and compression, were conducted at room temperature, 300°, and 500°F on three glass-fabric-base laminates. Resins used in these three materials were a silicone, DC-2104; a phenolic, CTL-91LD; and a polyester resin, PDL-7-669. Deterioration tests and some preliminary short-time tensile tests were also made on these three materials.

The creep and creep-rupture tests indicate that the PDL-7-669 material has the highest strength in tension at all three test temperatures of 80°, 300°, and 500°F. The highest compressive strength, on the other hand, was displayed by the CTL-91LD material under nearly all test conditions. However, both CTL-91LD and PDL-7-669 materials show a rapid decline in creep strength with time at 500°F. Beyond 50 hours' test time, the DC-2104 laminate

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displays the highest compressive creep-rupture strength at this temperature.

The short-time tensile tests show about the same strength relationship among the three test materials as do the creep-rupture tests. That is, the PDL-7-669 laminate is the strongest in tension and the CTL-91LD material appears to have superior strength in compression. These two materials, however, maintain their tensile strength superiority at 500°F possibly because they were held at the test temperature for only short periods of time.

The deterioration-test results show that the DC-2104 silicone resin material is definitely the most stable of the three laminates after being exposed at 300° and 500°F for various periods of time. The long-time creep-rupture strength of these laminates at 500°F and perhaps 300°F, appears to be closely related to their high-temperature stability.

National Bureau of Standards. TR 53-192, Part 3.

MECHANISM OF RAIN EROSION. PART 3 - MECHANISM STUDIES ON PLASTICS AND METALS. Olive G. Engel. AF 33(616)53-9. December 1953.

Rain erosion damage on brittle materials of low tensile strength has been reproduced in a parallel study of the damage caused by the impact of steel spheres and of deforming lead pellets on methyl methacrylate plastic. Analysis of the damage in the parallel study involving steel spheres and lead pellets has essentially clarified the mechanism of rain erosion on brittle materials of low tensile strength.

Investigation made with metals include preliminary studies of the effect of polish, of general hardening, and of surface hardening. Although clues have been found which seem to explain how the erosion progresses on metals after it has been initiated, the precise mode of formation of the first pit nucleus has not been ascertained. Three possible modes of formation are discussed.

National Bureau of Standards. TR 53-307.

FLEXURAL TESTS OF STRUCTURAL PLASTICS AT ELEVATED TEMPERATURES. John E. Wier and Dorothy C. Pons. AF 33(038)-51-4358. January 1954. PB 119102. Order from LC, Mi $3.90, Ph $10.80.

Flexural strength and modulus of elasticity at elevated temperatures were determined on 1/8 inch thick glass-fabric laminates bonded with three types of commercially-available heat resistant resins. The resins were: 1) a silicone, DC 2104; 2) a polyester, PDL-7-669; and 3) a phenolic, CTL-91LD. The laminates were made of glass-fabric, Fiberglas 181 with finishes 112 (heat cleaned), Garan, and 114. These finishes were used with the silicone, polyester, and phenolic resins, respectively.

The laminates were exposed to temperatures of 300, 400, 500, 600, and 700°F for periods ranging from one-sixth of an hour to 1000 hours. The tests were made at the elevated temperatures. Loss of weight due to exposure to elevated temperatures was also determined.

It was observed that of the three types of laminate only the silicone bonded type retained enough strength to be tested after 10 hours' exposure to 700°F. However, the initial strength of the silicone bonded laminate at 73°F and at some of the lower elevated temperatures was much lower than that of the other two types of laminate. The phenolic bonded laminate gained weight after exposure to the lower range of elevated temperatures.

National Bureau of Standards. TR 53-192, Part 4.

MECHANISM OF RAIN EROSION. PART 4 - CAVITATION AS A RESULT OF WATERDROP COLLISIONS WITH SOLID SURFACES. Olive G. Engel. AF 33(616)53-9. January 1954.

A search was made to determine whether or not cavitation takes place in a waterdrop when impact occurs between the drop and a solid surface. Five arrangements of the lights and camera were tried in an effort to obtain maximum resolution in high speed moving pictures of any bubbles which may form. Some positive evidence was obtained showing that cavitation does occur. Further tests with carbon dioxide-saturated water and with argon-saturated water, which are now planned, should either reinforce or nullify this evidence. The wash configurations which result from impact of a waterdrop against surfaces having different degrees of smoothness, and against materials having different degrees of resilience, were observed with high speed moving pictures.

Cornell Aeronautical Laboratory Inc. WADC TR 53-185.

A STUDY OF THE RAIN EROSION OF PLASTICS AND METALS. Roy R. Lapp, Raymond H. Stutzman, Norman E. Wahl. AF 33(600)-6469. February 1954.

Shell Development Company. TR 52-5, Suppl. 2

HIGH STRENGTH EPON LAMINATES. F. C. Hopper. AF 33(038)-19587. March 1954. ASTIA Document No. AD 33331. PB 119613s2. Order from LC, Mi $4.50, Ph $12.30.

Mixtures of one part EPON 1001 with 2 parts Plyophen 5023, cured with dicyandiamide and using 181-Volan A glass fabric, yielded laminates with good strength at room temperature and at elevated temperature after short-time exposure. Resistance to aging at 500°F was poor, but was better than that of Plyophen 5023 alone. Strengths typical of good samples follow:

Test Temp., deg. F.	Exposure, hrs.	Flexural, ult., psi-
Room	Indef.	70,000
300	1/2	55,000
500	1/2	45,000
500	200	7,000

A laminate so made passed the mechanical strength requirements of Military Specification MIL-R-7575A, but failed to meet the requirements of weight and thickness charges on immersion in water and various chemicals.

Fabric impregnated with mixtures of the two resins exhibited poor storage stability. Laminates made with fabric stored 35 days at room temperature were high in resin and had less than one-half of their initial flexural strength at 500°F after 1/2 hour at 500°F, although resistance to aging at 500°F was not impaired.

EPON 1004 could be used successfully in place of EPON 1001 and Lebec 102,210 was equal in performance to Plyophen 5023.

EPON X-12100 (P-2592) yielded laminates which were less strong than the foregoing on short-time exposure, but were more resistant to high temperature aging. After 200 hours at 500°F, the strength of one laminate at 500°F was 12,000 psi.

This was increased to about 15,000 psi in other laminates by the addition of a small amount of copper 8-quino-linolate as stabilizer. The addition of 10 to 20 PHR[a]) of Plyohen 5023 appeared to effect even better stability, yielding laminates having flexural strengths of 16,700 and 19,600 psi at 500°F after aging.

Laminates containing EPON X-003.12.0, cured with dicyandiamide, had the highest strengths so far obtained with an EPON resin at 300°F (64,600 psi), but failed badly at 500°F.

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a) PHR = parts per hundred of resin and is equivalent to per cent based on the resin.

A new curing agent, known as "CL", greatly improved the elevated temperature strength of EPON 828. It could not be used with the more heat resistant solid EPON resins, such as EPON X-12100, which must be applied from solution, because it cured the resin during drying of the fabric to remove the solvent.

With the possible exception of EPON 828 cured with CL, EPON 1001 containing dicyandiamide continued to yield laminates of higher room temperature strength than any other EPON resin. Wright Air Development Center TR No. 52-5; Supplement No. 1 describes this type of laminate with which flexural strengths of 76,900 psi and compressive strengths up to 70,200 psi at room temperature have been obtained.

Forest Products Laboratory. WADC TR 52-183 (Supl 2).
ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 BULLETIN, "PLASTICS FOR AIRCRAFT". Donald C. Coleman. AF 33(038)-51-4326E. October 1954.

Developments in the program of research in plastics for aircraft conducted by the U.S. Forest Products Laboratory during fiscal year 1954 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Two technical reports issued during the fiscal year are abstracted.

United States Rubber Company. WADC TR 53-371.
HEAT RESISTANT POLYESTER LAMINATING RESINS. Marvin Botwick, William Cummings, Paul M. Elliott. AF 33(600)-16825. October 1954.

The heat resistance of Vibrin X-1047 laminates is notably influenced by the finish on the glass cloth employed. The heat resistance of such laminates is a more searching test of cloth quality than the measurement of green flexural strengths. A 301-finish is generally satisfactory for use with heat resistant polyesters.

Two monomers, triallyl aconitate and diallyl Δ^4-3, 6-methanotetrahydrophthalate, in combination with Alkyd X-1038 provide two new resins which show good retention of flexural strength at 300-400°F. With the triallyl aconitate resin, a flexural strength of 27,000 psi is maintained through 408 hours at 300°F. At 400°F the flexural strength is still 26,500 psi after 408 hours exposure. The resin derived from diallyl Δ^4-3, 6-methanotetrahydrophthalate is still increasing in strength between 192 and 408 hours at 300°F (38,900 to 42,100 psi) and does not lose strength in the same interval at 400°F (38,000 to psi level.).

It has been demonstrated that a cured foam of 10 lbs./cu. ft. density can be prepared from Vibrin X-1047 using the technique developed under Bu. Ships Contract Nobs-54183 (1773).

A modified poly (bismethyloixylene maleate) alkyd is superior to the modified poly (ethylene maleate) used in Vibrin X-1047 where resistance to long term exposure to 500°F is involved. Using a mixed triallyl cyanurate - diallyl Δ^4-3, 6-methanotetrahydrophthalate monomer system, rather than triallyl cyanurate alone, affords a superior heat resistant polyester. At the same time the materials cost of the resin is reduced.

Forest Products Laboratory. WADC TR 55-31.
CURING OF VOID-FREE GLASS-CLOTH-REINFORCED LAMINATES AT ROOM TEMPERATURE. Bruce G. Heebink. AF 33(038)51-4326E. March 1955. PB 111823. Order from OTS $1.00.

There is a demand for polyester-resin systems that cure at room temperature for use with glass-fiber reinforcing for such applications as prototype aircraft parts or for repair of radomes. Many oxygen-liberating catalysts have been found that will promote cure at room temperature, but care must be exercised in their choice and proportions to avoid too rapid or too slow curing conditions.

Void-free test laminates were made of 181 glass cloth that yielded acceptable strength properties when using one of these room-temperature curing combinations with a typical polyester resin.

An unusual small-void formation developed with certain specific rolls of cloth, which was thought to be due to a chemical reaction between the catalysts and the finish on the cloth.

National Bureau of Standards. WADC TR 54-326.
DETERMINATION OF RESIN CONTENT OF GLASS FIBER POLYESTER LAMINATES CONTAINING A CALCIUM CARBONATE FILLER. S. D. Toner. AF 33(038)51-4060. March 1955. ASTIA Document No. AD 74456.

The presence of calcium carbonate filler in unoriented glass-fiber reinforced polyester laminates introduces appreciable errors when determining the resin content by the accepted method.

The temperature range, 538° to 593°C, required to burn off the resin is higher than that at which calcium carbonate begins to dissociate. The reduction of calcium carbonate to calcium oxide and the subsequent evolution of carbon dioxide results in a greater loss in weight than would be expected if no filler were present. A quantitative method of restoring the carbon dioxide to the reduced filler consists of treating the residue with an excess of a concentrated ammonium carbonate solution to convert the calcium oxide formed back to calcium carbonate.

Specimens were prepared from predetermined amounts of resin, glass-fiber mat and calcium carbonate to give the following composition, by weight: resin, 50 percent; glass fiber, 37.5 percent; and calcium carbonate, 12.5 percent.

The specimens were ignited to constant weight in a muffle furnace, cooled, and weighed. The residue was treated with ammonium carbonate and the increase in weight due to absorbed carbon dioxide was determined. Hot 3N hydrochloric acid was added to the treated residue in an attempt to remove, quantitatively, the calcium carbonate.

Results obtained in these experiments indicate that treatment of the residue with ammonium carbonate will reduce the error in the resin content from an average 3.75 percent to less than 0.1 percent. The acid treatment cannot be used to give quantitative determination of the calcium carbonate, the resin of the glass since about 10 percent of the glass dissolves in this treatment.

Forest Products Laboratory. WADC TR 53-483. EFFECTS OF FABRIC FINISH AND WET EXPOSURE ON STRENGTH PROPERTIES OF GLASS-CLOTH POLYESTER LAMINATES. Fred Werren. AF 33-(038)51-4066. AF 33(038)51-4326. March 1955.

Strength tests were made of polyester laminates reinforced with 181 glass fabric, having 114 finish, a methacrylic chrome complex, and three of the newer fabric finishes. The tests were made after normal conditioning and after varied exposure conditions, including immersion in boiling water, immersion in water at room temperature, and conditioning in an atmosphere at 100°F and near 100% relative humidity.

This report presents the results of tests made to determine the effect of various exposure conditions, including long-time exposure in a humid atmosphere, on the strength properties of the various laminates. The tests show that the laminates made of fabric with the newer finishes were consistently higher in flexural properties after wet exposure than the 114 finish.

The United States Rubber Company. WADC TR 54-474, Part 1. EVALUATION AND DEVELOPMENT OF PLASTIC LAMINATED BACKING BOARD MATERIALS. Wendell H. Smith, Charles C. Surland. AF 33(616)-484. March 1955.

New plastic laminate backing board constructions produced with variations of resins, fabrics, fillers, and fabricating procedures were evaluated and compared with a representative group of commercially available backing board materials.

Test devices were developed to determine biaxial strength properties and resistance to fluid ram action and augment the information obtained by conventional mechanical property tests and cube gunfire tests.

New backing board constructions were produced. Test work and development of methods and equipment were carried out, as well as gunfire and cube gunfire tests.

An improved backing board construction was developed using widely spaced rovings of glass fiber bundles impregnated with polyester resin.

Further work on extensions to this contract will permit more complete investigation of variations in the improved backing board construction, and the use of the developed test methods should provide better screening of improvements.

National Bureau of Standards. WADC TR 53-192, Part 5. MECHANISM OF RAIN EROSION PART 5. FURTHER STUDIES ON CAVITATION IN LIQUID DROPS ON IMPACT. Olive G. Engel. AF 33(616)53-9. March 1955.

The search for evidence of cavitation in the flow of a waterdrop after it impinges against a glass plate at approximately its terminal velocity in air was continued. The schlieren arrangement discussed in the preceding report was used. The interpretation of pictures obtained with this method is discussed, and the evidence of bubble formation which was found in waterdrops saturated with argon gas, waterdrops saturated with carbon dioxide gas, and in drops of hydrant water is presented. An equation for the time dependence of the radius of the radial flow of the drop after collision with the glass plate is developed. The time dependence of the velocity of the radial flow is considered. Results of a measurement of the impact force are given.

Shell Development Company. WADC TR 52-5, Supl. 3. HIGH STRENGTH EPON LAMINATES. Frank C. Hopper. AF 33(038)-19587. April 1955. ASTIA Document No. AD 66441. PB 11961303. Order from LC, Mi $6.30, Ph $19.80.

This report is designed to present an accurate account of the present status of research on EPON resin laminating with reference to this contract. It describes in detail all work performed during the past year and summarizes previous work dating back to the inception of the contract.

Wet and dry laminating systems are described. Wet systems based on EPON 828 yield laminates with exceptional flexural and compressive strengths at room temperature. Resistance to water and solvents is excellent. Strength retention at elevated temperatures up to 300°F is good.

Dry laminating systems offer good strength from room temperature to 500°F. Laminates made from EPON X-12100 cured with Curing Agents E or F retain up to 36,000 psi flexural strength at 500°F after aging 200 hours at 500°F. Even stronger after short exposure to 500°F, but less resistant to aging at that temperature, is a mixture of EPON 1001 with Plyophen 5023 (a phenolic resin) cured with dicyandiamide.

Forest Products Laboratory. WADC TR 55-37. DIMENSIONAL STABILITY OF PLASTIC MOLDING COMPOUNDS. B. G. Heebink, W. G. Youngquist. D. O. (33-616)-53-20. May 1955.

Cellulose filled melamine molding compounds have been replaced for use as insert insulating material for aircraft multipin connectors by mineral filled polyester compounds with satisfactory results, because of the greater dimensional stability of the latter. The unsatisfactory materials changed in dimensions after being in service for a long period of time to such an extent that the connectors could no longer be connected. This report covers quantitative tests for dimensional stability on a number of different molding compounds of the following types:

1. Cellulose filled melamine
2. Mineral filled alkyd
3. Mineral filled diallyl phthalate

Both high temperature dry condition tests and high temperature high humidity tests were run. The results of the high temperature dry tests show that the cellulose filled melamine compounds all had to from 10 to 40 times the dimensional changes of the other two types. There was not the same clear cut difference in the high temperature high humidity tests. Therefore, the high temperature dry condition test is a more effective screening test.

National Bureau of Standards. WADC TR 54-540.
EVALUATION OF NON-WOVEN GLASS FIBER REINFORCEMENTS FOR LOW PRESSURE MOLDED POLYESTER RESINS FOR AIRCRAFT APPLICATIONS. F. W. Reinhart, M. F. Toompas, M. C. Sloane, I. Wolock. AF 33(038)-51-4060. May 1955.

The strength properties of several glass-fiber reinforced polyester moldings and sheets made from similar materials were determined. The constructions tested were: tote boxes made from glass mat, and from glass fiber roving preforms with no filler, and with filler; flat sheets made from the same combinations of resins, glass fiber and filler as used in the tote boxes; window frames made from glass fiber roving preforms; and small panels made from glass fiber reinforced molding compound. The results indicate that the bottoms of the tote boxes made from preforms with no filler had slightly higher strength properties than the corresponding sheets. The bottoms of the tote boxes made from glass mat had strength properties approximately equal to the corresponding sheets, and the bottoms made from preforms with filler had lower strength properties than the corresponding sheets. The strength properties of the bottoms of the window frames were approximately equivalent to those of the corresponding tote boxes with no filler and were, in general, higher than those of the corresponding flat sheets. The sides of the tote boxes and the flanges of the window frames had lower strength properties than corresponding construction in the flat sheets. The panels made from the glass fiber reinforced molding compound had much lower strength properties than the other moldings tested. In general, the strength properties of the moldings decreased after immersion in water.

United States Rubber Company. WADC TR 53-371. Part 2.
HEAT RESISTANT POLYESTER LAMINATING RESINS. William Cummings, Fred J. Foster, Robert G. Nelb. AF 33(600)-16825.

Diallyl carbate in combination with triallyl cyanurate and VIBRIN 135 alkyd provides a new resin with improved retention of flexural strength after aging 192 hours at 500°F. This new resin has been coded VIBRIN X-1068. VIBRIN X-1068 glass fabric laminates exhibit flexural strengths of 26,000 - 30,000 psi at 500°F after being aged for 192 hours at 500°F. The finish on the glass fabric plays a very significant role in the hot strength of the laminates. The 301 and Garan finishes have given the best results. VIBRIN X-1068 laminates exhibit improved craze resistance over laminates prepared from the conventional triallyl cyanurate based heat resistant polyester resins.

Cornell Aeronautical Laboratory, Inc. WADC TR 53-185. Part 2.
A STUDY OF THE RAIN EROSION OF PLASTICS AND METALS. Roy R. Lapp, Raymond H. Stutsman, Norman E. Wahl. AF 33(600)-6469. May 1955.

Many engineering problems have been encountered with the advent of subsonic and supersonic aircraft. One of these problems is the phenomena of erosion, during flight through rain, of coatings, structural plastic and metal parts on the exterior of high speed aircraft.

This report is a compilation of test data obtained, mostly at 500 mph and 1 inch per hour rainfall, on various metallic and non-metallic aircraft materials, using the rotating arm erosion apparatus.

Flight tests in rain on a jet aircraft were carried out to obtain actual service test data. Air foil shaped specimens of various coatings and plastics were attached to the wing leading edge, and preliminary flights through rain at 500 mph made. These flight tests corroborated laboratory tests and the fact that neoprene coatings such as Gaco N-79 and Goodyear 23-56 possess the greatest erosion resistance of non-metallic materials tested to date.

Johns-Manville Corporation. WADC TR 55-186.
ASBESTOS FIBER REINFORCED PLASTIC LAMINATES. Conrad C. Spatz. AF 33(616)-2257. November 1955.

An investigation was started to determine the effectiveness of asbestos in reinforcing low-pressure laminating resins. This report covers the evaluation of laminates prepared from various asbestos papers and a phenolic resin.

Laminates were made that meet the MIL-P-8013 (USAF) specifications on tensile, edgewise compressive, and flexural strengths at room temperature for glass mat reinforced polyester laminates. Dry flexural strength was retained 100% after two hours boiling in water and after immersion in water for sixty days at room temperature. Room temperature flexural strength was retained close to 100% after seven days exposure to 300°F and tested at 300°F. A 65% strength loss occurred after 1/2 hour at 500°F with no further loss up to seven days exposure. The paper was found to be fairly non-directional in strength.

Owens-Corning Fiberglas Corporation. WADC TR 55-290.
THE DEVELOPMENT OF FIBROUS GLASSES HAVING HIGH ELASTIC MODULI. George R. Machlan. AF 33 (616)-2422. November 1955. PB 111785. Order from OTS $3.00.

A fifty percent increase in modulus of elasticity of fibrous glass was achieved by the continuous formation of fibers of a calcium aluminate glass in a small textile glass bushing.

The resistance of these glasses to chemical attack by water and water vapor is much less than that of commercially produced textile fibrous glass. The glasses are resistant to hydrofluoric acid but are completely soluble in hydrochloric acid.

The dielectric constants of these glasses were found to be higher than the dielectric constant of present textile glass and the loss tangents were found to be approximately the same as that of present textile glass.

Several compositions were investigated which contained tin oxide as the major constituent but no glasses were found.

An exploratory study of glass-plastic combinations from the calcium aluminate glass was initiated. Volan A was found to be the best coupling agent tested for this glass.

The United States Rubber Company. WADC TR 54-474. Part 2.
EVALUATION AND DEVELOPMENT OF PLASTIC LAMINATED BACKING BOARD MATERIALS. Charles C. Surland. Sup Agreement No. S3(54-1266). January 1956.

Different types of backing board materials were tested by internal explosion techniques to examine the feasibility of duplicating gunfire damage. Pressure deflection tests on a simulated airframe installation were made to relate mechanical properties of the backing boards to their support characteristics for both self-sealing and bladder type fuel cells.

Emperical equations were developed for the deflection of backing boards under fluid pressures. The tests included encountered the maximum and minimum spans which would normally be in aircraft structures. Equations

were correlated with the physical properties of the backing boards as the basis for establishing a method of specifying backing boards.

Production trials of USV CR 88 backing board were made to examine the feasibility of production by continuous methods and furnish materials to the WADC.

Cornell Aeronautical Laboratory. WADC TR 55-308.

THE RAIN EROSION OF AIRCRAFT MATERIALS. R. R. Lapp, Raymond H. Stutzman, Norman E. Wahl. AF 33 (600)-6469. January 1956.

Many engineering problems have been encountered with the advent of subsonic and supersonic aircraft. One of these problems is the phenomenon of erosion during flight through rain of coatings, structural plastic, and metal parts on the exterior of high speed aircraft.

This report is a compilation of data on all the metallic and non-metallic aircraft materials tested from May 1947, to August 1955, using the rotating arm erosion apparatus. Most of the tests were conducted at 500 mph in 1 in/hr rainfall; however, the early tests were conducted at speeds as low as 150 mph.

The materials which were evaluated include plastics, both thermoplastic and thermosetting types, elastomeric materials, lacquers and enamels, inorganic materials such as glass and ceramics, and metals.

This report summarizes factors affecting the rain erosion resistance of materials such as velocity, airfoil radius, coating support, the influence of angle of impact, type of core material and surface defects.

The data compiled here are referenced to the specific report in which the tests are described in detail at the time they were conducted.

The purpose of this report is to evaluate the performance of a number of products for a specific application. Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

Cornell Aeronautical Laboratory, Inc. WADC TR 55-342.

INVESTIGATION OF HEAT RESISTANT POLYESTER LAMINATES. Norman E. Wahl, Norman M. Preston. AF 33(616)-2515. March 1956.

Various methods of eliminating the cracking of triallyl-cyanurate copolymer resins reinforced with 181-301 glass cloth were investigated.

The use of .010" asbestos surface overlay on glass cloth laminates used with commercially available triallylcyanurate resins was found to be the most practical approach to prevent cracking in production applications.

Several new triallylcyanurate copolymers that exhibit reduced water absorption and heat loss were developed. Data on two resins which eliminated the cracking are described.

National Bureau of Standards. WADC TR 53-192, Pt 8.

MECHANISM OF RAIN EROSION Part 8 On the Breakup of a Waterdrop in the Zone Behind a Detached Shock Wave. Olive G. Engel. AF 33(616)-53-9. May 1956.

The change of pressure, density, and velocity through the shock wave caused by an object moving at supersonic velocity in still air, and the change of pressure, density, and velocity through the zone of separation between the shock wave and the object are calculated. The mechanism of waterdrop breakup is discussed and the critical diameter of a waterdrop that should be able to survive in the conditions in the zone behind a detached shock is found.

Forest Products Laboratory. WADC TR 55-319.

WEATHERING OF GLASS-FABRIC-BASE PLASTIC LAMINATES. Fred Werren, B. G. Heebink. DO (33-616)-53-20. May 1956. ASTIA Document No. AD 101284. PB 121390. Order from OTS $1.50.

Eleven different glass-fabric-base plastic laminates, made with 9 different laminating resins, were subjected to outdoor weathering at 5 sites having entirely different weather conditions. After completion of the exposure cycles, the laminated panels were tested in tension, compression, and flexure at the U. S. Forest Products Laboratory. Data on the effect of exposure on the mechanical properties and the appearance of the laminates after exposure periods of 3 months and 12 months are presented in this report.

Another series of panels is still undergoing exposure at all sites, and this series will be tested after 3 years' exposure. Upon completion of tests of these panels, all data will be compiled in a final report.

Data obtained from the tests made to date show that the effect of outdoor exposure varies with the type of laminate and condition of exposure. The laminate materials showed no severe reductions of strength after outdoor exposure for one year. Most of the materials showed average reductions from 5 to 10 percent. However, one type of material, that being the heat-resistant polyester resin laminates, showed reduction in strength averaging about 20 percent.

Forest Products Laboratory. WADC TR 52-183, Sup 4.

ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 BULLETIN "PLASTICS FOR AIRCRAFT". Donald G. Coleman. AF 33(616)-56-9. September 1956. ASTIA Document No. AD 97329. PB 121695. Order from OTS $0.50.

Developments in the program of research in plastics for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1956 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Six technical reports issued during the fiscal year are abstracted.

Forest Products Laboratory. WADC TR 56-206.

MECHANICAL PROPERTIES OF POLYESTER LAMINATES REINFORCED WITH HIGH MODULUS GLASS FABRIC. Fred Werren. DO 33(616)-56-9. September 1956. ASTIA Document No. AD 97314. PB 121683. Order from OTS $0.75.

Comparative strength tests were made of three polyester laminates fabricated at the U. S. Forest Products Laboratory. Two of the laminates were reinforced with H. M. 18 glass fabric, a fabric designed to produce laminates with a high modulus. One laminate had a resin content of 41.9 percent and the other of 34.8 percent. The third laminate was a typical laminate reinforced with 181 glass fabric, and it had a resin content of 36.7 percent. Tension, compression, and flexure tests were made of each laminate, and the strength and elastic properties are presented in this report. The average mechanical properties of the 181 laminate were generally as good as or higher than those of the 2 H. M. 18 laminates.

Some comparative strength data on an H. M. 18 polyester laminate fabricated and tested by the Cincinnati

Testing and Research Laboratories are also included in this report. The resin content of this laminate was only 29.1 percent, and the mechanical properties were higher than those of the laminates with greater resin content fabricated at the Forest Products Laboratory.

The results of this study show that the mechanical properties of an H. M. 18 laminate at a resin content of about 36 percent would probably be about the same as or lower than those of a 181 laminate at a comparable resin content. The data indicate, however, that a reduction in resin content may appreciably increase the strength and elastic properties of an H. M. 18 laminate. This relationship is also true for a 181 laminate; however, there is some indication that the increase in mechanical strength with decrease in resin content in a 181 laminate is not as great as for a H. M. 18 laminate.

WADC TR 56-296.
NUCLEAR RADIATION OF REINFORCED PLASTIC RADOME MATERIALS. Robert C. Tomashot, Douglas G. Harvey, 1/Lt. September 1956. ASTIA Document No. AD 97254.

Nine different reinforced plastic laminate materials, each made with a different laminating resin, and one alkyd-isocyanate foam core-glass fabric faced sandwich material were subjected to integrated gamma radiation dosages up to 10^9 roentgens. After completion of the radiation exposures, the materials were tested to determine the flexural, tensile, and compression strengths under both standard and wet conditions. The mechanical properties of the heat resistant plastic laminates were also determined at elevated temperature.

Dielectric constant and loss tangent measurements were conducted on irradiated samples to determine the effect of radiation on the electrical properties of these materials.

Data obtained from these tests show that the mechanical properties were not significantly affected except for one epoxy type resin laminate. None of the materials showed any significant change in electrical properties due to radiation.

Cornell Aeronautical Laboratory. WADC TR 53-185, Pt 3.
SUMMARY REPORT ON THE RAIN EROSION OF AIRCRAFT MATERIALS. Roy R. Lapp, Raymond H. Stutzman, Norman E. Wahl. AF 33(616)-2758. September 1956. ASTIA Document No. AD 97317.

This report summarizes the data obtained on the relative rain erosion resistance, on the whirling arm tester, of a number of materials at subsonic speeds.

Several solvent type air dry coatings based upon neoprene were evaluated for conformance to Military Specification MIL-C-7439B. A modified Gaco N-79 neoprene coating was the only coating of the series tested that had satisfactory rain erosion resistance.

Polyurethane materials tested in the form of sheet or coatings applied to standard glass reinforced test specimens, while not as erosion resistant as neoprene, had very good erosion resistance and will be investigated more intensively.

Tests on fluorinated ethylene materials show that they possess a relatively low degree of rain erosion resistance.

Erosion tests were conducted on a number of white coating materials based upon alkyd enamels, vinyl, silicone, butyl, polysulfide and chlorosulfonated ethylene elastomers. Of all these white materials tested, coatings based upon Hypalon alone and combinations of white pigmented neoprene and polyacrylic rubber appeared to merit further study.

Of the outstanding glass and ceramic materials tested, Corning Glass 1710 took 45 minutes to erode while a ceramic body, Alsimag 576 of Alumina, lasted for approximately 420 minutes.

Comparison of current epoxy and polyester glass reinforced laminates indicates that the erosion resistance of standard test specimens of epoxy-glass laminates have four to five times the erosion resistance of similar laminates made with polyester resins.

Neoprene coated thin walled laminates, such as those used on hot air deiced radomes, were found to have very poor rain erosion resistance.

In collaboration with the National Bureau of Standards, studies on the mechanism of erosion and tests were conducted on a variety of different materials under specific conditions.

Forest Products Laboratory. WADC TR 56-264.
EVALUATION OF LOW-DIELECTRIC GLASS FABRIC. Fred Werren, B. G. Heebink. DO 33(616)-56-9. October 1956. ASTIA Document No. AD 110459. PB 121859. Order from OTS $0.75.

Glass-fabric-base plastic laminates, reinforced with fabric made of low-dielectric glass fibers and standard "E" glass fibers, were evaluated on the basis of mechanical and electrical properties. Six different laminating resins were used. Comparative data obtained from these tests showed that laminates made with low-dielectric fabric were superior in electrical properties but inferior in mechanical properties to comparable laminates made with the standard "E" glass fabric.

National Bureau of Standards. WADC TR 55-256.
EFFECTS OF MOLDING PRESSURE ON THE STRENGTH PROPERTIES OF SEVERAL TYPES OF GLASS-FIBER REINFORCED PLASTICS. Samuel D. Toner, Frank W. Reinhart, Irvin Wolock. PO AF 33(616)-53-14. December 1956. PB 121196. Order from OTS $2.00.

An investigation was made to determine the effects of molding pressure on the mechanical properties of glass-fiber reinforced laminates fabricated from a polyester resin reinforced with woven glass fabric or with 2 oz. or 8 oz. unoriented glass fiber mat.

The laminates were fabricated in an open mold at molding pressures of 1, 10, and 100 lb/in^2, and in a closed mold at 10, 100, and 500 lb/in^2. The resin content of the free-edge panels varied with the molding pressure, while that of the closed-edge panels was maintained at a constant value for each type of reinforcement.

Flexural, tensile and compressive strengths and flexural and tensile moduli of elasticity were determined for dry specimens as well as for specimens that had been immersed in water at 73.5°F (23°C) for thirty days. Specific gravity, resin content, and voids content were also determined.

The test results indicate that the properties of laminates fabricated in the closed mold do not vary appreciably with changes in molding pressure. However, the flexural and tensile properties of panels molded in the open mold increase as the pressure increases from 1 to 10 lb/in^2 but do not change appreciably at higher molding pressure. Compressive strength shows a tendency to decrease with increasing molding pressure.

The results obtained for the closed-mold panels were in close agreement with those of the open-mold panels having the same reinforcement and similar resin content.

Forest Products Laboratory. WADC TR 56-522. EFFECT OF THICKNESS ON STRENGTH OF EPOXY AND PHENOLIC LAMINATES REINFORCED WITH GLASS FABRIC. Kenneth H. Boller. DO 33(616)-56-9. March 1957. ASTIA Document No. AD 118098. PB 131044.

Results of tensile and compressive tests on laminates made with 3 types of resin and 2 types of reinforcement in thicknesses ranging from 1/100 inch to 1/8 inch showed that the stress at failure decreased with decreasing thickness. The magnitude of the decrease varies with the material. The decrease for 0.030 inch material, about the thinnest used in aircraft, may be as great as 12 percent of the strength of 1/4 inch thick material.

Battelle Memorial Institute. WADC TR 57-37. ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF EPON-PLYOPHEN PLASTIC-GLASS FABRIC LAMINATE. Gilbert M. Gynn, John A. VanEcho, Ward F. Simmons. AF 33(616)-3215. April 1957. ASTIA Document No. AD 118211.

Basic design data on heat-resistant laminated and transparent plastics have been obtained for the benefit of airframe designers. The material discussed in this report is a glass fabric reinforced plastic made with a particular epoxy-phenolic resin.

The test results contained in this report were obtained from creep and creep-rupture, flexural, deterioration, and short-time tensile and compression tests. These results show that after exposures ranging from 0 to 1000 hours at test temperatures of 80° to 1000°F the material displays a variety of properties. In general, the ultimate strengths decrease with increases in temperature and exposure time. This trend of decreasing strength with increasing temperature was not of a constant rate, however, for the various types of tests.

At the lower temperatures, the compression strength of the material was found to be greater than the tension strength but lesser than the flexural strength. At higher temperatures, however, the tensile strength is greater than either the flexural or compression strengths.

The results presented in this report are not to be considered precise design information, but rather a general indication of strength properties.

National Bureau of Standards. WADC TR 53-192, Part 7. MECHANISM OF RAIN EROSION. PART 7 - MECHANISM STUDIES ON 1100 AND 3003 ALUMINUM. Olive G. Engel. AF 33(616)-53-9. April 1957. ASTIA Document No. AD 118223.

A study of deformation marks made by impingement of steel spheres and of deforming lead pellets against 2S aluminum plates has proved helpful in analyzing the behavior of and the damage caused by waterdrops in collisions with 2S aluminum at both subsonic and supersonic relative impingement velocities. Results of the study with steel spheres and deforming lead pellets are described. A mechanism by which high-speed rain erosion may take place on 2S aluminum and on other soft metals with similar properties is advanced. The understanding of this specific mechanism is helpful in that it supplies evidence for the development of the basic mechanism of rain erosion of materials in general including plastic materials which are of particular interest to the aircraft industry.

Battelle Memorial Institute. WADC TR 56-581. ROOM- AND ELEVATED-TEMPERATURE PROPERTIES OF NA-91LD PHENOLIC RESIN LAMINATE. John A. VanEcho, James W. Garrabrant, Ward F. Simmons. AF 33(616)-3215. April 1957. ASTIA Document No. AD 118214.

This report contains engineering data for a glass fiber laminate fabricated under low (vacuum-bag) pressures from 91LD phenolic resin and 181-Volan A glass cloth. The material is identified as NA-91LD in this report. Some comparative data are included for another phenolic resin laminate (CTL-91LD) made under high mold pressures.

The property data reported include short-time tensile and compression, creep and creep-rupture in tension and compression, flexure, and deterioration. Temperatures employed range from 80°F to 1000°F with prior exposures at these temperatures ranging from zero holding time to 1000 hours.

This phenolic resin laminate shows relatively good elevated- and room-temperature strength properties. Results have been fairly reproducible and consistent among the four panels tested. The flexural strength and deterioration properties of the vacuum-bag molded laminate are very similar to those of the press-molded phenolic. The creep and creep-rupture and short-time tensile strengths of the vacuum-bag laminate appear, generally, to be superior to the press-molded material. The reverse is true with regard to the short-time compression strength and elastic modulus properties.

Improvements in the finish on the glass fabric, curing techniques, and resin processing methods can considerably increase the strength properties that can be obtained with high pressure CTL-91LD laminates. However, the test data reported represent the values obtained from laminates, fabricated under specific conditions and are useful in showing the properties available by this method of fabrication.

Battelle Memorial Institute. WADC TR 56-231. SHORT-TIME ELEVATED-TEMPERATURE TENSILE AND COMPRESSION PROPERTIES OF GLASS-FABRIC-PLASTIC LAMINATES. John A. VanEcho, Gale R. Remely, Ward F. Simmons. AF 33(616)-3215. April 1957. ASTIA Document No. AD 118220.

Basic property data on various heat-resistant reinforced and transparent plastic materials have been obtained for the benefit of airframe designers. Materials discussed in this report include three reinforced plastics made with typical heat-resistant types of silicone, phenolic, and polyester triallyl cyanurate resins.

Based on test results contained herein, which consists of short time static tensile and compression data after exposure to various temperatures and conditioning periods, it is shown that, generally the phenolic resin laminate possesses the highest strength after short time exposures at temperatures as high as 500°F. The silicone resin material exhibits a superior strength at the higher temperatures and long exposures. For some intermediate temperatures and exposure times, the triallyl cyanurate-polyester resin laminate has the superior comparative strength. Thus, each of the three test materials possesses superior strength properties at some of the test conditions with no one laminate being superior at all conditions.

The results presented are considered merely as an indication or trend of strength properties rather than as precise design information. As a minimum, check data should be obtained on the particular lot or batch of material actually being used.

Taylor Fibre Co. WADC TR 56-270. INVESTIGATION OF THE EFFECTS OF GLASS FABRIC GEOMETRY ON THE STRENGTH PROPERTIES OF LOW PRESSURE GLASS FABRIC BASE STRUCTURAL LAMINATES. W. R. Bowditch, E. L. Johnson. AF 33(616)-2543. May 1957. ASTIA Document No. AD 118324. PB 131372.

This report describes work done to develop glass fabrics which make stronger plastic laminates than are produced by the glass fabrics which are commercially available today.

In the initial evaluation program, seventeen (17) experimental fabrics and two (2) high modulus fabrics were compared to four (4) standard glass fabrics in laminate form. Five (5) experimental fabrics were selected for further evaluation after improvements were made in crimp, twist, fiber diameter and weave. Based on data obtained, further consideration and study should be made to determine the practicality of

a. Using Final Fabric S-14619-1
b. Using the original HM-9 fabric or the HM-9 fabric made from ECK Filaments
c. Using the HM-18 (original) or the HM-18 fabric made from ECK filaments
d. Using a 143 type fabric with ECG or ECK filaments in the warp direction.

Stanford Research Institute. WADC TR 56-534, Part II. NUCLEAR RADIATION EFFECTS ON STRUCTURAL PLASTICS AND ADHESIVES. Part II Extension of Literature Survey. R. Y. Mixer, Shirley B. Radding. AF 33(616)-3632. June 1957. ASTIA Document No. AD 130838.

A survey was made of literature on radiation chemistry available during the period 30 September 1956 to 1 February 1957. Information pertinent to the effect of radiation on the syntheses and the properties of polymers, elastomers, and adhesives was tabulated. A brief summary of the more general observations on the effects of radiation on these materials was prepared.

The literature available prior to 30 September 1956 was covered in Part I.

Battelle Memorial Institute. WADC TR 57-246. ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF VIBRIN 135 GLASS FABRIC-PLASTIC LAMINATE. Gilbert M. Gynn, John A. VanEcho, Ward F. Simmons. AF 33(616)-3215. July 1957. ASTIA Document No. AD 130881.

Basic engineering data have been obtained for the benefit of airframe designers on a glass-fabric laminate containing Vibrin 135, a polyestertriallyl cyanurate (TAC) resin. These data include results of creep and creep-rupture in both tension and compression, flexural, deterioration, and short-time tensile and compression tests.

The strength properties of Vibrin 135 were found to be dependent upon both the temperature and the length of time the material was exposed to a given temperature. The test results indicate that the laminate is relatively strong and stable for exposures to 1000 hours at low temperatures (80°, 300° and 400° F). At higher temperatures, however, the strength of Vibrin 135 decreased rapidly with increases in either temperature or exposure time.

The results reported are for particular and specified panels of the test material, and should not be considered as precise design data for all panels of Vibrin 135 laminate.

National Bureau of Standards. WADC TR 53-192, Part IX. MECHANISM OF RAIN EROSION. PART IX - Observation of the Fragmentation of Waterdrops in a Zone behind an Air Shock. Olive G. Engel. AF 33(616)53-9. July 1957. ASTIA Document No. AD 130909.

Observations made on the fragmentation of two waterdrop sizes after collision with air shocks that were moving at three different supersonic velocities are reported. The possible mechanism of various aspects of the fragmentation process are discussed. The experimental observations indicate that high-speed-rain-erosion damage should not be observed on spheres having a diameter as large as 4 ft and moving with a Mach Number in the range of 1.3 to 1.7 in rain having a drop diameter of 1.4 mm. Drops of this size should be reduced to mist in the zone of separation between the detached shock and the surface of the sphere according to the results that are reported. A means to extend this protection to spheres of smaller diameter or to rain of larger size is pointed out. The need for further experimental observation of the time required for the fragmentation of waterdrops using shocks moving at higher Mach Numbers is indicated to verify and extend the information.

Stanford Research Institute. WADC TR 56-534, Part III. NUCLEAR RADIATION EFFECTS ON STRUCTURAL PLASTICS AND ADHESIVES. Part III Experimental Research. R. Y. Mixer, D. B. Parkinson. AF 33(616)-3632. August 1957. ASTIA Document No. AD 131029.

Nuclear radiation damage mechanisms are presented for model compounds representing several typical aircraft structural adhesives and laminates. Laminates were prepared from eight resin-curing agent systems and irradiated to 10^9 10^{10} rep for determination of the threshold dose for damage. The threshold dose for one system was found to be less than 10^8 rep. In the case of typical amine-cured, epoxy-type adhesives, radiation appears to cleave the amine groups with volatilization of some fragments and to crosslink the polymer chain. Small amounts of chemical changes result in large changes in physical properties of the cured adhesive. Beta and gamma irradiation have produced equal effects on one vinyl-phenolic and one epoxy-type adhesive. One organic scintillator, 2, 5-diphenyloxazole, appears to be partially effective in preventing irradiation damage in an expoxy type adhesive.

Cornell Aeronautical Laboratory. WADC TR 57-200. ASBESTOS REINFORCED LAMINATES. Norman E. Wahl, Harold M. Preston. AF (616)-2926. September 1957. ASTIA Document No. AD 131041.

The use of long fibered asbestos as a reinforcement for typical polyester, epoxy, silicone and phenolic resins was investigated. Low pressure laminates were prepared and the physical properties were determined.

It was found that the molding pressure and the resin content of asbestos laminates had a considerable influence on their ultimate physical properties. Asbestos reinforced laminates molded at 15, 30 and 60 psi had considerably lower mechanical properties than glass reinforced laminates molded at the same pressure with the same resin. Molding pressures from 200-800 psi and resin contents of 28-38 percent by weight of the asbestos laminate are required to achieve physical properties that are comparable to

glass cloth reinforced laminates. Of the various types of resins investigated, the phenolic resins with asbestos felts resulted in laminates with the most reproducible and the highest physical properties at room temperature. However, at elevated temperatures the silicone resins with the asbestos gave the highest strengths. After exposure to temperatures of 700° and 800°F for 192 hours and tested at 700° or 800°F, these silicone asbestos laminates generally retained 50% or more of their room temperature strength. For applications where plastic laminates must be used at temperatures of 700° or 800°F, the silicone asbestos laminates merit consideration.

It was observed that the resin content of an asbestos laminate could not be lowered to any great degree by increasing the pressure during molding, as can usually be done with laminates using glass as a reinforcement. If lower resin contents are required, it is necessary to treat the asbestos by various methods such as the use of a solvent diluted resin with subsequent removal of the solvent in an oven or drying tower with the proper amount of heat.

The final properties of asbestos laminates depend on many factors such as fiber treatment, asbestos binders, resin content, flow and gel time of the resin, layup of the plies, molding temperature pressure and posture. It is apparent from this study that a great deal has to be done to develop a technique of impregnating and molding asbestos felt in order to obtain higher physical properties with various types of resins.

Forest Products Laboratory. WADC TR 52-183, Suppl. 5.

ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 BULLETIN "PLASTICS FOR AIRCRAFT." Donald G. Coleman. AF 33(616)-56-9. October 1957. ASTIA Document No. AD 142081.

Developments in the program of research in plastics for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1957 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Eight technical reports issued during the fiscal year are abstracted.

Johns-Manville Corporation. WADC TR 55-186, Part II.

ASBESTOS FIBER REINFORCED PLASTIC LAMINATES. Conrad C. Spatz. AF 33(616)-2257. October 1957. ASTIA Document No. AD 142044.

The investigation to evaluate the effectiveness of medium length and short asbestos fibers in reinforcing low-pressure laminating resins was continued. This report covers the evaluation of laminates prepared from various experimental asbestos papers and phenolic resins.

A number of these laminates meet the mechanical test requirements of Military Specification MIL-P-8013 for polyester-glass mat laminates. Several of these laminates meet the flexural and edgewise compressive strength requirements of some of the glass cloth laminates in this specification. The tensile strength of these laminates is below the requirements of glass cloth laminates. The best asbestos-reinforced laminates retain 75 per cent ultimate flexural strength and up to 100 per cent flexural modulus at 500°F after one-half hour at 500°F.

WADC TR 57-352.

EVALUATION OF A VARIATION IN GLASS COMPOSITION OF GLASS FIBERS FOR PLASTIC LAMINATES. G. P. Peterson. October 1957. ASTIA Document No. AD 142065.

A variation of "E" type glass, designated 1145 "E" glass was developed by Owens-Corning Fiberglas Corporation to replace the standard "E" type glass designated 621 "E" glass for use in glass fibers for glass fiber reinforced plastic laminates. The evaluation program included the fabrication and evaluation of laminates with: (1) 181 style glass cloth woven from both types of glass fiber, (2) Owens-Corning 136 and 139 finishes on each type of glass fiber, (3) typical polyester (Paraplex P-43) and heat resistant polyester (Vibrin X-1068) resins. Room temperature, elevated temperature, chemical immersion, water immersion, fatigue and electrical tests were conducted to provide a comparison of the properties of laminates made from the two glasses. Electrical tests were also made on bulk glass samples of the two glasses. The 1145 "E" glass fiber was satisfactory and equivalent to the standard 621 "E" type glass fiber in all respects except for dielectric constant properties. At room temperature the dielectric constant at 8.5 KMC of the 1145 "E" glass was somewhat higher than that of 621 "E" glass, which is undesirable, but within acceptable limits. However, the dielectric constant at 8.5 KMC of the 1145 "E" glass increased far more rapidly with increasing temperatures than that of 621 "E" glass, and to an unacceptable extent. Consequently the 1145 "E" glass would not be satisfactory for radomes for radar guidance control purposes. Since the proposed alternate composition is not acceptably equivalent to the 621 "E" glass, the 1145 "E" glass is not considered satisfactory for Air Force reinforced plastic applications.

Johns-Manville Corporation. WADC TR 55-186, Pt III.

ASBESTOS FIBER-REINFORCED PLASTIC LAMINATES. Wayne O. Jackson. AF 33(616)-2257. November 1957. ASTIA Document No. AD 142139.

The investigation of medium length and short asbestos fibers as reinforcement for low-pressure laminating resins for use in structural laminates was continued. This report covers the evaluation of laminates prepared from medium length asbestos fiber paper and phenolic, epoxy, and polyester resins.

A number of these laminates meet the mechanical test requirements of Military Specification MIL-P-8013A for glass mats. Several laminates meet the flexural and edgewise compressive strength requirements of glass-cloth laminates. Phenolic, epoxy and polyester laminates retained 100% of their flexural strength and flexural modulus after three months' exposure to outdoor weathering conditions. No change was noticed after one year for the phenolic laminates while the epoxy and polyester laminates' tests are continuing for one year.

National Bureau of Standards. WADC TR 53-192, Part XI.

MECHANISM OF RAIN EROSION. PART XI - Effect of Residual Stresses and of Molding Variables on the Erosion Resistance of Nylon. Olive G. Engel. AF 33(616)-53-9. November 1957. ASTIA Document No. AD 142115.

It is shown that residual stresses in the original plastic sheet material and the use of improper molding conditions for the fabrication of test specimens may provide an incorrect rain-erosion-resistance rating for the material in question and misleading evidence in regard to the failure mechanism of it. Test results indicate that properly molded nylon FM-10001, which was heat treated by the manufacturer

to remove residual stresses in the plastic sheet, is one of the most rain-erosion resistant of the rigid plastic materials that have been evaluated at impingement velocities up to 600 mi/hr. This rigid plastic closely approaches the rain-erosion-resistance of neoprene elastromers at 600 mi/hr.

Battelle Memorial Institute. WADC TR 57-531. ROOM- AND ELEVATED-TEMPERATURE STRENGTH PROPERTIES OF BAKELITE BV-17085 GLASS FABRIC-PLASTIC LAMINATE. Gilbert M. Gynn, John A. VanEcho, Ward F. Simmons. AF 33(616)-3215. November 1957. ASTIA Document No. AD 142136.

Basic design data including creep and creep-rupture, flexural, deterioration, and short-time tensile and compression properties were obtained for Bakelite BV-17085, a phenolic resin-glass-fabric laminate. The creep and creep-rupture data were obtained at 80, 300, and 500°F with rupture times ranging from about to 1000 hours. The other properties were determined at 80 to 1000°F with prior exposures at the test temperatures for times ranging from 0 to 1000 hours. The creep and creep-rupture specimens were not exposed to elevated temperatures prior to testing.

The over-all strength properties of BV-17085 are relatively good with useful load-carrying ability as high as 1000°F for time periods of less than about 1 hour. The short-time tensile strength of the material seems to be largely independent of test temperature or prior exposure within limits of the test conditions. The short-time compression, creep-rupture, and flexural strength properties, on the other hand, vary to a large degree with both test temperatures and prior exposure or exposure during test.

Stability of the test material, as determined by exposure at elevated temperatures for various periods of time, is quite good at 300 and 400°F. At higher temperatures, deterioration of the laminate is directly related to the temperature and time of exposure.

The test data discussed in this report should be considered only as property trends because of the wide degree of scatter generally found in the property values of glass-fabric laminates including BV-17085.

WADC TR 57-638, Part I.
EFFECTS OF HIGH INTENSITY THERMAL RADIATION ON STRUCTURAL PLASTIC LAMINATES. Herbert S. Schwartz Ben J. Lisle. Dec. 1957. ASTIA Document No. AD 142181.

Polyester, silicone and phenolic glass-fabric-base plastic laminates were exposed to thermal radiant energy up to an intensity of 25 cal/cm^2-second, with and without mechanical stress, to determine their resistance to mechanical and physical degradation.

Absorptivities of thermal radiant energy from a 6000°K source were computed from spectral reflectance and transmittance data measured over the region from 0.4 to 2.0 microns.

The resistance of the laminates to thermal radiation measured by degradation of mechanical properties was found to be a function of absorptivity, heat resistance of the laminating resin, and stress applied. Based on mechanical property considerations, the optimum material for resisting the effects of high intensity radiant energy and moderate stresses was found to be the silicone laminate, and for low intensities over a wide range of stresses was found to be the phenolic laminate.

Battelle Memorial Institute. WADC TR 57-574. ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF CONOLON 506 PLASTIC-GLASS FABRIC LAMINATE. Gilbert M. Gynn, John A. VanEcho, Ward F. Simmons. AF 33(616)-3215. December 1957. ASTIA Document No. AD 142190.

Basic engineering properties for Conolon 506, a phenolic resin-glass fabric laminate, are presented in this report. The data included were obtained from creep and creep-rupture, flexural, deterioration, and short-time tension and compression tests. The tests were conducted at temperatures ranging from 80 to 1000°F with prior exposures at these temperatures ranging from about 0 to 1000 hours.

In general, the creep-rupture strength was found to be greater in tension than in compression. The minimum creep rates were found to be relatively low since very little plastic strain was measured after loading. The flexural and short-time tensile and compressive strengths, and flexural and tensile elastic moduli were deleteriously affected by increases in either temperature or exposure time. Also, under the conditions of this evaluation, the short-time tensile strength exceeded the short-time compressive strength. The results of this evaluation merely indicate property trends and should not be considered as precise design data.

National Bureau of Standards. WADC TR 53-192. Pt X. MECHANISM OF RAIN EROSION. PART X - A Review and Evaluation of the Present State of the Problem. Olive G. Engel. AF 33(616)53-9. December 1957. ASTIA Document No. AD 142240.

A review of the rapidly accumulating literature on the subject of erosion by waterdrop impingement has been made. The types of experimental apparatus that have been used by the investigators, and the factors that have been found to determine the extent of the erosion damage, are briefly discussed. Results of microscope and X-ray studies of eroded surfaces, and of parallel studies of damage marks produced by the impingement of steel spheres, deforming lead pellets, oil-filled gelatin capsules, and waterdrops are presented. Several theoretical estimates of the impact pressure that results from the collision of a waterdrop with a solid surface are reviewed. The result of a piezoelectric measurement of this pressure is given. Some of the theories that have been advanced in regard to the mechanism of the erosion process and of the micromechanism of failure are discussed. The important role that design can play both in mitigating the erosion and in completely bypassing the problem under some conditions is pointed out.

WADC TR 57-674.
THE EFFECTS OF HYDRAULIC FLUIDS ON THE PROPERTIES OF GLASS FIBER REINFORCED PLASTIC LAMINATES. D. F. Starks. February 1958. ASTIA Document No. AD 150972.

The object of this investigation was to provide a wider range of mechanical strength and physical property information on the effects of immersion on some reinforced plastic materials in chemical types of fluids being considered for future use at the projected use temperatures. Weight and thickness change determinations were conducted on the flexural specimens of the five (5) plastic materials utilized, with four (4) experimental hydraulic fluids. Flexural, tensile and compressive strength tests were conducted both before and after immersion.

The various immersion conditions utilized for the materials were as follows: (1) 24 hours at room temperature, (2) 7 days at room temperature, (3) 24 hours at 160°F for Paraplex P-43 polyester only, (4) 24 hours at 250°F for the heat resistant materials, (5) 24 hours at 400°F in OS-45 silicate, and (6) 24 hours at 500°F in F-50 silicone. The immersions at 250°F, 400°F and 500°F were conducted utilizing the heat resistant materials only.

Immersion in the high temperature fluids under the various conditions used had negligible effect on the mechanical properties of the plastic materials. The room temperature immersion in the fluids had negligible effect on the weight and thickness physical properties, however, variations in weight and thickness were determined from the elevated temperature immersions at 400°F and 500°F, particularly.

Forest Products Laboratory. WADC TR 58-27.
MECHANICAL PROPERTIES OF PLASTIC LAMINATES REINFORCED WITH NOL 24 - FINISHED GLASS FABRIC. Kenneth E. Kimball. DO 33(616)56-9. March 1958. ASTIA Document No. AD 151091.

This report presents the results of tests of three parallel-laminated, glass-fabric-base, plastic laminates in which the glass fibers had been treated with a universal type of chemical finish. The finish was developed by the Naval Ordnance Laboratory and is designated as NOL 24. This finish was tested with a 181 glass fabric and polyester, epoxy, or phenolic resin.

The laminates were tested in tension, compression, and flexure after normal and wet conditioning, with some tests at elevated temperatures. Data are presented both in tables and as typical curves.

Also included is a table of comparative data showing how the laminates reinforced with NOL 24-finished fabric compared with similar laminates reinforced with fabric having other finishes.

Owens-Corning Fiberglas Corporation. WADC TR 55-290, Part II.
THE DEVELOPMENT OF FIBROUS GLASSES HAVING HIGH ELASTIC MODULI. J. A. Waugh, V. E. J. Chiochetti, H. I. Glaser, R. Z. Schreffler. AF 33(616)-2422. May 1958. ASTIA Document No. AD 155502. PB 131927.

This report describes the work done to draw fiberized glass having a high modulus of elasticity, from two kinds of glass cullet; that containing calcium aluminate and that containing beryllium oxide.

Fiberized glass was obtained from each although not in any quantity. Devitrification problems made the calcium aluminate fibers "brashy", brittle, and rough. It is not expected that any known "tip environment" control will eliminate this devitrification problem.

A small amount of fiberized glass was obtained from the cullet containing beryllium oxide. This was smooth in texture and had a good appearance. Pure powdered beryllium oxide is extremely toxic and studies were made after this substance was incorporated into the glass melt to determine if such toxicity remained a hazard when the cullet was remelted. It proved to be no hazard although in the interests of good precautionary measures, a hood for optimum fume control is recommended.

In the initial evaluation program, six (6) different bushings were used incorporating eight (8) different tip designs or modifications in an effort to fiberize calcium aluminate glass cullet. Results were generally unsuccessful.

In the subsequent (Supplemental Agreement) evaluation regarding beryllium oxide glass cullet, one (1) bushing incorporating two (2) different tip designs was used in the attempted fiberization. Results were generally successful.

Cornell Aeronautical Laboratory. WADC TR 53-185, Part 4.
THE STUDY OF EROSION OF AIRCRAFT MATERIALS AT HIGH SPEEDS IN RAIN. Roy R. Lapp, Donald H. Thorpe, Raymond H. Stutsman, Norman E. Wahl. AF 33(616)-2758. May 1958. ASTIA Document No. AD 155501.

The results obtained on the relative rain erosion resistance, at subsonic speeds, of a large number of different types of materials, are reviewed in detail in this report.

Spray or brush-on types of coatings were evaluated for conformance to Military Specification MIL-C-7439B. Only those coatings based upon neoprene had erosion resistance meeting the requirements.

Erosion tests were conducted on a large number of white coatings based upon various pigmented polyacrylics, silicones, chlorosulfonated ethylene and neoprene elastromers. Of all these white materials tested, coatings based upon Hypalon alone and combinations of white pigmented neoprene and polyacrylic rubber appeared to merit further study.

Of the glass and ceramic materials tested, alumina bodies with over 90% alumina had the best erosion resistance.

Comparison of current epoxy and polyester glass reinforced laminates indicates that the erosion resistance of standard test specimens of epoxy-glass laminates have four to five times the erosion resistance of similar laminates made with polyester resins.

In collaboration with the National Bureau of Standards, studies on the mechanism of erosion and tests were conducted on a variety of different materials under specific conditions.

Johns-Manville Corporation. WADC TR 55-186, Part 4.
ASBESTOS FIBER-REINFORCED PLASTIC LAMINATES. Charles L. Rohn. AF 33(616)-2257. June 1958. ASTIA Document No. AD 155647.

The use of short fiber asbestos paper as a reinforcement in low pressure laminating resins has been investigated. The results of this investigation, based primarily upon mechanical properties of laminates at room temperature, are recorded in WADC Technical Reports 55-186, Parts I, II, and III.

The context of this report, a continuation of the former investigation, emphasizes high temperature strength properties and strengths of the laminates after exposure to wet conditions. A specific heat-resistant phenolic resin combined with the same asbestos reinforcement was used in this study.

The optimum strength properties of these asbestos laminates are dependent upon the fabricating techniques used. Variables such as laminating pressure, time and temperature, pre- and post-cure, and resin content have considerable effects upon the final properties of the laminate. Most of these techniques were developed during earlier work on asbestos laminates and were adopted for this investigation.

National Bureau of Standards. WADC TR 53-192, Part 12.
MECHANISM OF RAIN EROSION, PART 12. PITS IN METALS CAUSED BY COLLISION WITH LIQUID DROPS. Olive G. Engel. AF 33(616)-57-12. July 1958. ASTIA Document No. AD 155756.

An equation is presented that may provide a means of predicting corresponding-velocities-for-equal-pit-depth for collisions of metal specimens with drops of mercury and with drops of water. The purpose for determining such corresponding velocities is to provide a relatively low velocity test using high density liquid drops to determine the ability of materials of this kind to withstand damage as a result of collision with waterdrops at very much higher velocities.

Forest Products Laboratory. WADC TR 57-182.
PROPERTIES OF ALKYD-ISOCYANATE FOAMED-IN-PLACE CORE. P. M. Jenkinson, E. W. Kuenzi. DO 33(616)56-9. September 1958. ASTIA Document No. AD 155884.

This report presents the results of flatwise compression, flatwise tension, shear, and a few flexure tests of sandwich consisting of facings of 0.03-inch-thick, glass-fabric laminate with a core of 0.50-inch-thick, alkyd-isocyanate foam. Test specimens and methods of test are described. Mechanical properties were evaluated at 75° and 160° F. for cores with densities of 10 and 20 pounds per cubic foot. These properties are presented in tables 1 and 2. Core strength decreased markedly as temperatures increased. Flatwise compressive strength at 160°F. was only 60 percent of that at 75° F. Exposure of the core to high humidity reduced the modulus of elasticity to 60 percent of the normal value. Flatwise compression stress-strain curves show that stresses at 2 percent core strain were about three-fourths those obtained at 10 percent head movement. The results of tests to determine moisture sorption and dimensional change for core material immersed in water and for core material exposed to 100 percent relative humidity at 100° F. are also included. Curves illustrate the variation of percentage changes of these properties with time. Moisture sorption as much as 2.5 percent and changes in dimensions up to 1.2 percent were observed for the core exposed to high humidity.

Forest Products Laboratory. WADC TR 52-183, Suppl. 6.
ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17 HANDBOOK "PLASTICS FOR FLIGHT VEHICLES." Donald G. Coleman. AF 33(616)-56-9. October 1958. ASTIA Document No. AD 203784.

Developments in the program of research in plastics for flight vehicles conducted by the U. S. Forest Products Laboratory during fiscal year 1958 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Three technical reports issued during the fiscal year are abstracted.

House Glass Corporation. WADC TR 58-285.
DEVELOPMENT OF HIGH MODULUS FIBERS FROM HEAT RESISTANT MATERIALS. Albert H. Lasday. AF 33(616)-5263. October 1958. ASTIA Document No. AD 202500.

This report deals with the development of new glass fiber forming compositions in order to produce high temperature continuous fiber monofilaments having high modulus of elasticity and strength. These properties need to be maintained at least to 1000°F. The most important objective is to produce a glass fiber whose elastic modulus divided by specific gravity is at least 9 million psi.

In order to develop such a glass fiber, the realm of metal oxide compositions melting at temperatures beyond 3,000°F must be investigated since the composition variables below 3,000°F (the region of conventional refractories and glass melting) already have been well explored. Furthermore, several high melting temperature oxides, some known to be glass producers, are also high modulus materials in bulk polycrystalline form.

Fiberization of these high temperature melting materials involves the development of new techniques. Two promising approaches have been developed. In the first, a glass rod of the high melting composition was made by dropping molten glass from a batch rod melting in a ring burner into a graphite mold. This glass rod was then heated inside a tubular graphite susceptor in an inductor coil, and when the glass reached the right temperature, a fiber could be drawn. The second approach involves drawing a fiber from a horizontal refractory cone, made of the same material as the batch rod. The cone is also heated in a graphite inductor. Molten glass droplets are melted from a batch rod using a ring burner and allowed to drop onto a concave cup in the upper surface of the cone.

Using an alumino-silicate composition based on kaolin, test fibers were made which gave indication of high elastic modulus. Some fibers tested as high as 19 million psi, although the data exhibits great scatter due to the crude way in which fibers were prepared.

WADC TN 58-222.
EVALUATION OF THE SPLIT-LINE OPTICAL DISTORTION TEST METHOD. Robert N. Smith, Captain, Technical Photographic Division, John R. Meyer, 1/Lt. October 1958. ASTIA Document No. AD 203526.

The results of an evaluation of the split-line technique for photographically determining optical distortion in aircraft transparencies are presented. These results show that the split-line method is not operationally simple, is unreliable, and biased. The time consuming task of evaluating a photograph for split lines would probably more than offset any gain obtained by devising a set-up for fast, routine operation.

Since the final acceptance of a transparency is by visual inspection, it is concluded that the most necessary item to be determined is a criterion of acceptance on the basis of visual examination.

Goodyear Aircraft Corporation. WADC TR 58-298.
EXPLORATORY INVESTIGATION OF INORGANIC FIBER REINFORCED INORGANIC LAMINATE FINAL REPORT. George Margo. AF 33(616)-5251. October 1958. ASTIA Document No. AD 203643.

Since the upper temperature limit of fiber-reinforced, organic- or silicone-bonded laminates is approximately 650°F for long time exposure, this program was initiated to explore the potential of inorganic materials for extending the thermal limit of structural laminates. The evaluation was based on fabrication and testing of laminates. Glass and asbestos were used as the primary reinforcement, but some preliminary work was done with a high-silica glass. The cements evaluated included sodium silicate, potassium silicate, calcium aluminate,

polymeric silicic acid sol, magnesium oxysulphate, ethyl silicate, and aluminum phosphate.

This report describes the fabrication and testing of the laminates and includes a tabulation of laminate properties and test results. The main objective was the development of a laminate of structural integrity that would meet a stated target flexural strength at 1000° F after 10 hours at 1000°F. Although good structures were produced, it was determined that improvements in both reinforcement and cement are necessary to achieve the program goals.

Forest Products Laboratory. WADC TR 58-356. FIBER-RESIN BOND IN REINFORCED PLASTICS. Charles B. Norris, James T. Heller. DO 33(616)56-9. October 1958. ASTIA Document No. AD 202503.

A technique is developed for determining the rigidity of the bond between reinforcing strands of glass fibers and the resin in which they are embedded. This technique involves a method of accurate determination of the tensile stiffness of the strands; a method of casting small beams of resin reinforced with glass strands that are concentrated near one edge; an accurate method of testing such beams centrally loaded so as to induce shear between the strands and the resin; and a method of calculating the shear force on the strands, the bending stiffness from the test, and the expected bending stiffness if the bond is infinitely rigid. Comparison between the calculated stiffness of the beam and its test value in bending yields a measure of the shear rigidity of the bond between the strands and the resin.

This technique is described in detail, and some results are given for Epon 828 resin reinforced with glass strands that consist of 204 individual filaments, each about 0.00038 inch in diameter. The filaments were finished with starch-oil or A-1100 finish or heat-cleaned.

Forest Products Laboratory. WADC TR 58-486. EFFECTS OF VARIOUS METHODS OF WET CONDITIONING ON THE STRENGTH PROPERTIES OF SEVERAL GLASS-FABRIC-REINFORCED PLASTIC LAMINATES. Bert P. Munthe, Robert L. Youngs. DO 33(616)-58-1. March 1959. ASTIA Document No. AD 210225.

This report presents the results of tests to evaluate the effects of various types and durations of wet exposure on the tensile, compressive, and flexural properties of epoxy, polyester, phenolic, and silicone laminates reinforced with 181 glass fabric that had various types of fabric finish. The tests were made after normal conditioning and after various periods of wet exposure. Wet conditions used were (1) immersion in water at 73°F., (2) exposure to an atmosphere at 100°F. and approximately 100 percent humidity, or (3) boiling in water.

Tensile strengths were reduced about equally by 30 days in water at 73°F. or by 2 hours in boiling water. Compressive strengths were reduced about the same amount by either 2 hours in boiling water, 30 days in water at room temperature, or 30 days at high humidity. Modulus of rupture was reduced about equally by equal periods of immersion in water at room temperature or exposure to high humidity at 100°F. At either of these conditions, the reduction after 30 days was generally similar to that observed after exposure in boiling water for 1/2 or 1 hour, but was generally less than that observed after exposure for 2 hours in boiling water.

The various laminates differed considerably in their response to the various wet exposure conditions. However, the results indicate that a 2-hour wet exposure in boiling water is a reasonable substitute for the standard 30-day immersion in water at room temperature. Any discrepancy is likely to be the conservative side, with greater strength reduction after the 2-hour period in boiling water than after 30 days in water at room temperature.

Stanford Research Institute. WADC TR 58-618. QUALITY IMPROVEMENT IN GLASS-FIBER-REINFORCED PLASTIC STRUCTURAL DIELECTRICS FOR HIGH-FREQUENCY ANTENNAS. Henry J. Sang. AF 33(616)-3997. April 1959. ASTIA Document No. AD 212905.

An accelerated version of the Rangoon test condition, simulating a humid tropical climate, was developed for plastic laminates. Four different types of material were evaluated in the Rangoon condition and in eighteen different accelerated conditions. The best of the accelerated conditions was AR-9, which produced in twelve days effects similar to those produced by the Rangoon test in 120 days. The AR-9 test condition is recommended as a replacement for the present water-immersion and boil tests.

An exploratory study was made of quality control of Vibrin 136 A resin for high-temperature use. Twenty-three different varieties of laminate were made and tested for strength and strength retention at 500°F. The strength properties were found to vary greatly with the manner of processing and curing. Representative varieties of laminate were resistant to deterioration in the accelerated Rangoon condition. The most critical aspect of the resin is its tendency to blister during post-curing; until this problem is solved, effective quality control cannot be secured. The best laminate made had a strength of 42,700 psi in flexure at 500°F after 192 hours aging at 500°F.

WADC TR 58-555. REVIEW OF STRUCTURAL PLASTICS. Robert C. Tomashot. April 1959. ASTIA Document No. AD 212560.

This report is a compilation of papers presented at the Wright Air Development Center-University of Dayton joint review on Structural Plastics, 10 - 11 September 1958, Dayton, Ohio. The papers, for the most part, reviewed work completed or in progress under Materials Laboratory research and development contracts.

Cornell Aeronautical Laboratory, Inc. WADC TR 53-185, Part 5. A STUDY OF EROSION OF AIRCRAFT MATERIALS AT HIGH SPEEDS IN RAIN. Roy R. Lapp, Raymond H. Stutsman, Norman E. Wahl. AF 33(616)-5455. April 1959. ASTIA Document No. AD 212901.

This final report summarizes the results of comparative rain erosion tests on various types of materials for aircraft. The tests were conducted on the whirling arm test apparatus which simulates high speed flights through rain under controlled conditions. Flat and airfoil specimens fabricated from plastics or metals, with and without coatings, as well as ceramic materials were mounted on the leading edge of the whirling arm and evaluated for erosion resistance at 500 mph in 1 in/hr rainfall.

In the tests on elastomeric coatings, an antistatic radome coating system developed by the Gates Engineering Company was recommended for approval under Class II of Specification MIL-C-7439B. The

coating system consisted of N-15 primer, N-79 neoprene and a thin Gates anti-static surface coating.

In another group of tests it was observed that the erosion resistance of neoprene coatings approved under Class I of the specification, was significantly lower after exposure to gamma radiation at dosage levels of 5 to 35 megareps.

Preliminary tests indicate that the erosion resistance of standard neoprene coatings was increased about three-fold when the neoprene was treated with an antiozonant solution, however, it is questionable whether this treatment of neoprene coatings would be practical for use under service conditions because the neoprene becomes soft and sticky.

Weathering tests in Florida, Panama and Wisconsin indicated that the erosion resistance of standard Goodyear 23-56, Gaco N-79 neoprene coatings and an experimental white radome coating developed by Gates Engineering Company were reduced substantially at each location after one year outdoor exposure.

Initial studies indicate that the rain erosion resistance of recently developed thermoplastic polycarbonate laminates is better than polyester or epoxy laminates.

In collaboration with the National Bureau of Standards, various tests were conducted to study the mechanism of rain erosion and to improve the erosion resistance of currently used materials.

Methods of trying to combine the water of crystallization of chrysotile asbestos fibers so as to improve the strength retention of laminates exposed to temperatures of 900° - 1000°F were investigated. In general, most asbestos fiber treatments tended to degrade the fiber making them brittle and lower in strength. In this work it was found that the use of silane finishes A-1100 and A-172 on 9526 asbestos felt gave laminates with higher flexural strength at room temperature when used with DC-2104 and DC-2106 silicone resins.

Forest Products Laboratory. WADC TR 59-27. EFFECT OF TYPE OF REINFORCEMENT ON FATIGUE PROPERTIES OF PLASTIC LAMINATES. G. H. Stevens, K. H. Boller. DO 33(616)-58-1. May 1959.

Fatigue strength values are presented for four epoxy and two phenolic laminates subjected to axial loading. The epoxy resin laminates were reinforced with 181-Volan A. glass fabric and with plies of continuous, unwoven glass fibers. The phenolic resin laminates were reinforced with parallel or cross-laminated lay-ups of asbestos mat. S-N curves are presented, representing the fatigue data after the laminate had been cycled in the fatigue machine from about 1,000 to 10 million cycles with 0 mean stress at 900 cycles per minute. Fatigue tests were made at 73°F and 50 percent relative humidity and at 100°F and 100 percent relative humidity.

When laminates were loaded parallel to a principal fiber direction, an endurance limit was generally not reached even after 10 million cycles of loading. After 10 million cycles, the fatigue strength of the epoxy laminates varied from about 15 to 35 percent of the corresponding lowest value of tensile or compressive control strength. The fatigue strength of all laminates at any number of cycles was usually about 10 to 30 percent less for wet specimens than for dry specimens, but a reduction up to 45 percent was indicated.

The presence of a small circular notch generally had only a small effect on the fatigue strength of the laminates, irrespective of the angle of loading or number of cycles. The difference in fatigue strength between notched and unnotched specimens was usually less than 10 percent.

Properties of epoxy laminates reinforced with continuous, unwoven glass fibers vary greatly with the orientation of the plies and direction of unloading, both under static and repeated loading. In these and the other laminates evaluated, however, the fatigue strength values at 10 million cycles had a much smaller spread than did the static strength data.

Forest Products Laboratory. WADC TR 58-668. EVALUATION OF LAMINATES REINFORCED WITH CHEMICALLY CLEANED AND FINISHED LOW-DIELECTRIC GLASS FABRIC. Gordon H. Stevens. DO 33(616)-58-1. May 1959. ASTIA Document No. AF 214423.

This report presents evaluations of mechanical and electrical properties of laminates reinforced with 181 glass fabric that had been chemically cleaned before finish was applied. Each laminate was made of Paraplex P-43 resin reinforced with (1) 181-Volan A standard "E" glass fabric, (2) 181-Volan A low-dielectric glass fabric, (3) 181-Garan low-dielectric glass fabric, or (4) 181-NOL 24 low-dielectric glass fabric. The mechanical properties of epoxy laminates of Epon 828 resin and 181-Volan A glass fabric -- one of standard "E" glass and the other of low-dielectric glass -- were also evaluated.

Laminates reinforced with chemically cleaned low-dielectric glass fabric were lower in mechanical properties than similar laminates reinforced with heat-cleaned glass fabric. The polyester Volan A laminates of chemically cleaned low-dielectric glass fabric were between 50 and 60 percent as strong as the corresponding laminates of heat-cleaned low-dielectric glass fabric. The strength ratio of the epoxy laminates was between 70 and 85 percent. A polyester laminate made of chemically cleaned low-dielectric glass fabric had about 30 to 45 percent of the strength of the comparable laminate made of heat-cleaned "E" glass fabric. The strength of the heat-cleaned low-dielectric laminate, however, was 50 to 85 percent of that for the laminate of heat-cleaned "E" glass fabric. The polyester laminate reinforced with chemically cleaned "E" glass fabric was 2 and 11 percent stronger in tension and compression, respectively, and 5 to 13 percent weaker in dry and wet flexural strength, than the similar laminate of heat-cleaned "E" glass fabric.

Comparative values from 181-Volan A polyester laminates indicate that laminates made with chemically cleaned fabric have poorer electrical properties than comparable laminates made with heat-cleaned fabric. The dielectric constants were about 10 percent greater and the loss tangents 4 to 6 times as large for the laminate of chemically cleaned low-dielectric glass fabric. The dielectric constants were about 15 to 25 percent larger for laminates of standard "E" glass fabric than for laminates of comparable low-dielectric glass fabric. The loss tangents for laminates of chemically cleaned low-dielectric glass fabric were 3 to 4 times those for laminates of both heat-cleaned and chemically cleaned "E" glass fabric. The loss tangents for laminates of heat-cleaned low-dielectric glass fabric, however, were about 30 percent lower for laminates of heat-cleaned "E" glass fabric than for laminates of chemically cleaned "E" glass fabric.

WADC TR 59-574.
BEHAVIOR OF PLASTIC MATERIALS IN HYPERTHERMAL ENVIRONMENTS. Donald L. Schmidt. June 1959.

A systematic investigation of the behavior of plastic materials in very high temperature air is reported. Several test models of various compositions and constructions were exposed in the air plasma of a one megawatt stabilized electric arc. Both descriptive and quantitative data were obtained on the characteristics and physical properties of the ablative materials.

A thermal analysis of quasi steady-state ablation at the stagnation point of the test models was performed. Individual heat transfer parameters were numerically estimated for the plastic materials and test conditions under consideration.

University of Dayton. WADC TR 59-171.
AN EVALUATION OF AN IMPROVED PROCEDURE FOR DETERMINING THE COMPRESSIVE PROPERTIES OF REINFORCED PLASTICS. Richard J. McBride. AF 33(616)-5500. June 1959. ASTIA Document No. AD 216531.

A necked-down specimen has been compared with the standard straight-sided specimen in an attempt to obtain better edgewise compressive strength data on reinforced plastic materials. This evaluation program included ultimate compressive strength tests and modulus of elasticity measurements on both the necked-down compression specimen and the standard straight-sided compression specimen. The apparatus utilized for strain measurements were Tuckerman Optical Strain Gages; Huggenberger Extensometers; SR-4 Strain Gages; and the PC-7M Compressometer. Short and long-time elevated temperature tests and water immersion tests were also conducted for additional comparison. The test data obtained comparing the two specimens indicated that the use of the necked-down specimen results in (1) 26 percent higher strength values, (2) comparable stress-strain properties with all four types of strain measuring apparatus, (3) consistent failure in the gage length, and (4) comparable spread in the test data obtained. Based on these data, the necked-down compression specimen should be utilized in place of the straight-sided specimen to obtain design data on reinforced plastics.

C. T. L., Inc. WADC TR 58-549.
HEAT RESISTANT LOW PRESSURE PHENOLIC RESIN GLASS. Donald C. Biedenkapp. AF 33(616)-3625. June 1959. ASTIA Document No. AD 226517.

The studies undertaken to obtain a phenolic laminating resin suitable for use at 600°F are presented in this report. The best material obtained, a silane modified phenolic, designated CT 37-9X, is shown to have strength properties at elevated temperatures which are better than those obtainable from previously available heat resistant phenolic resins.

Subsidiary studies are presented on the effectiveness of oxidation barrier coatings for glass reinforced plastic laminates in preventing loss of strength at elevated temperatures, attempts to produce butyl titanate resin systems, the effects of pre-stress on the strength of laminate panels, and the comparative effects of temperature on laminate panels reinforced with glass fibers and with asbestos fibers.

Forest Products Laboratory. WADC TR 52-183, Sup. 7.
ANNUAL REPORT ON RESEARCH IN ANC-17 HANDBOOK "PLASTICS FOR FLIGHT VEHICLES." Donald G. Coleman. AF 33(616)-58-1. September 1959. ASTIA Document No. AD 231734.

Developments in the program of research in plastics for flight vehicles conducted by the U. S. Forest Products Laboratory during fiscal year 1949 are summarized. In general, the approach has been to derive criteria mathematically, and then to check by test. Two technical reports issued during the fiscal year are abstracted.

Forest Products Laboratory. WADC TR 59-216.
EFFECT OF ELEVATED TEMPERATURES ON WEIGHT LOSS AND FLEXURAL PROPERTIES OF THREE REINFORCED PLASTIC LAMINATES. Kenneth H. Boller, Kenneth E. Kimball. DO33(616)-58-1. September 1959. ASTIA Document No. AD 232962.

This report presents data showing the effects of elevated temperatures for various exposure periods on the deterioration (weight loss) and flexural strength properties of three reinforced plastic laminates; a silicone-glass laminate (DC 2106 resin and 181 heat-cleaned glass fabric), a phenolic-glass laminate (CTL-91LD resin and 181-A1100 glass fabric), and a phenolic-asbestos laminate (R/M Pyrotex felt Style 41-RPD).

The test methods are described and the data are presented in tables and curves.

Forest Products Laboratory. WADC TR 59-229.
STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES AT ELEVATED TEMPERATURES DC 2106 Resin and 181 Heat-Cleaned Glass Fabric. Kenneth H. Boller, Kenneth E. Kimball. DO33(616)-58-1. September 1959.

Several reinforced plastic laminates that show promise of having good strength properties at elevated temperatures are being tested to determine their strength and elastic properties. This report describes the methods used in testing such laminates after exposure periods of up to 1,000 hours. Flexural, tensile, compressive, interlaminar shear, and bearing tests parallel to the warp direction are made to determine the effects of high temperature and time exposed at a given temperature on the strength properties. Tensile tests at 45° to the warp direction are made to obtain data from which edgewise shear strength and modulus of rigidity can be calculated. Creep and stress-rupture data are obtained under both tension and compression loads. In addition to strength properties, data are obtained on the deterioration of the laminated material at elevated temperatures.

This is the first of several reports that are planned as a part of this work. Data for laminated material made of DC 2106 resin and 181 heat-cleaned glass fabric are presented in both tables and charts. In general, it is shown that strength properties decreased with an increase in temperature. At temperatures up to and including 600°F, strength properties were usually not appreciably affected regardless of the time they were held at the high temperature.

Monsanto Chemical Co. WADC TR 59-328.
HEAT RESISTANT LAMINATING RESINS. Morton H. Gollis, John J. O'Connell. AF 33(616)-5682. October 1959. ASTIA Document No. Ad 231007.

This report describes the progress made toward the development of a heat resistant polymer for use as a laminating resin with glass fabric. This resin should be thermally stable and have good mechanical properties at 700°F.

Three main areas of investigation were undertaken: chelate polymers from dyes and dye intermediates, chelate

polymers of bisbiguanides, and polyisocyanurates. Favorable results were obtained with polyisocyanurates and dye or dye intermediate chelates.

Polyisocyanurate-glass fabric laminates prepared from 4,4'-diphenylmethane diisocyanate have shown good thermal stability and mechanical properties at 500°F, but decompose slowly at 700°F. The prospect of increasing thermal stability by slight modification of the polymer molecule or polymerization technique appear promising.

Chelate polymers derived from dihydroxyanthraquinone and orthohydroxyazo compounds have been prepared with molecular weights of about 12,000. These polymers have good thermal stability, but are highly crystalline and will require modification to improve their mechanical properties before testing as laminating resins.

WADC TR 56-296, Sup 1.
NUCLEAR RADIATION OF REINFORCED PLASTIC MATERIALS. Richard L. Keller. December 1959. ASTIA Document No. AD 232888.

The threshhold of degradation caused by gamma radiation for eight reinforced plastic laminate materials. The test materials were subjected to three levels of integrated gamma radiation with a maximum dosage of 8.3 x 10" ergs per gram of carbon. After completion of the radiation exposures, the conventional mechanical strength properties were determined under both standard and wet conditions. The mechanical properties of the heat resistant plastic laminates were also determined at elevated temperatures.

The effects of simultaneous exposure of both gamma radiation and elevated temperature on structural plastic materials were also investigated. Three of the reinforced plastic laminate materials were subjected to integrated gamma radiation dosages for different periods of time at 500°F. Mechanical tests were conducted on the materials to determine if simultaneous radiation and temperature exposure caused more degradation than would be expected from separate environmental exposures. It was found that one resin system had the same mechanical properties after simultaneous exposure as after separate environmental exposures, and two other resin systems exhibited an increase in the mechanical strength properties determined after simultaneous exposure as compared to separate environmental exposures.

WADC TR 58-357.
SOME PROPERTIES OF NOL-24 FINISHED FABRIC REINFORCED PLASTIC LAMINATES. Richard L. Keller. December 1959. ASTIA Document No. AD 234683.

Glass-fabric-base plastic laminates, reinforced with Style 181 fabric having NOL-24 finish, were evaluated on the basis of mechanical strength properties under both standard and wet conditions, as well as at elevated temperatures. Nine different laminating resin systems were used. The results obtained are compared with results of tests on similarly prepared laminates using a glass fabric finish considered to be optimum for the particular resin employed. For phenolic and epoxy resin laminates, the strength properties averaged about 15 percent lower for NOL-24 finished fabric reinforced laminates. However, the heat resistant epoxy and heat resistant epoxy and heat resistant polyester laminates made with NOL-24 finished fabric showed considerably lower strengths, ranging up to a 46 percent decrease for long time elevated temperature conditions. It is concluded that the NOL-24 finish is not universal in terms of mechanical strength properties under the conditions of tests used.

Cornell Aeronautical Laboratory, Inc. WADC TR 59-368.
THE STUDY OF ABLATION OF STRUCTURAL PLASTIC MATERIALS. Franklin A. Vassalo, N. E. Wahl, G. A. Sterbutzel, J. Beal. AF 33(616)-5683. December 1959. ASTIA Document No. AD 234779.

Results of ablation tests conducted on reinforced plastics at moderately severe heating conditions are reported. The materials tested include laminates of melamine, phenolic, and silicone reinforced with glass fabric as well as phenolic and silicone asbestos laminates. Experimental data are given for rate of material loss, rate of temperature rise at points within the body, depth of heat penetration, and loss of material strength both during and after ablation. The test apparatus provides a stream of high temperature nitrogen which is directed onto test specimens in which thermocouples are located. Body shapes considered are 20° wedges and square edged plates.

Analysis of the obtained data is presented for dependence of heat penetration on rate of ablation. The heat penetration is associated with the thickness of the thermal layer which is shown to be strongly dependent on the rate of ablation varying from as much as 0.300 in. to as little as 0.010 in. over a range of ablation rates from 0.007 to 0.10 in./sec.

Dynamic and post-exposure mechanical properties were determined. Relationships for determining the thickness of the thermal layer and dynamic strength were used to calculate predicted values which in most cases were reasonably close to experimental results. Rates of ablation and effective heats of ablation over a range of heat flux from 0 to 200 Btu/ft^2-sec. are given for each material tested.

Forest Products Laboratory, Forest Service. WADC TR 59-569.
STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES AT ELEVATED TEMPERATURES (CTL-91LD Phenolic Resin and 181-A1100 Glass Fabric). Kenneth H. Boller. DO33(616)-58-1. September 1960.

Several reinforced plastic laminates that show promise of having good strength properties at elevated temperatures are being tested to determine their strength and elastic properties. This is the second of several reports that are planned as a part of this work, and presents the results of mechanical tests on a phenolic resin laminate reinforced with glass fabric (CTL-91LD resin with 181-A1100 fabric). Data were obtained after exposure periods of up to 1,000 hours from such tests as weight loss, flexure, tension, compression, interlaminar shear, and bearing. Strength tests were made parallel to the warp direction to determine the effects of temperature and time on the strength properties. Tension tests were also made at 45° to the warp direction to obtain data from which edgewise shear strength and modulus of rigidity can be calculated. Creep and stress-rupture data were obtained under both tension and compression loads.

The data are presented in both tables and charts. In general, it is shown that strength properties decrease with increases in temperature. Temperatures of 300° and 400°F and duration of exposure at these temperatures have little effect on strength; however, temperatures of 500°F and above, as well as durations of exposures exceeding one-half hour, reduce the strength substantially. Some properties are affected more than others so that each should be judged separately.

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Forest Products Laboratory. WADC TR 52-184, Sup. 7.

SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON COMPOSITE CONSTRUCTION FOR FLIGHT VEHICLES. Donald G. Coleman. AF 33(616)-58-1. January 1960. ASTIA Document No. AD 234453.

Developments in the program of research in composite construction for flight vehicles conducted by the U. S. Forest Products Laboratory during fiscal year 1959 are summarized. In general, the approach has been to derive design criteria mathematically, and then to check by test. Six technical reports issued during the fiscal year are abstracted.

University of Dayton. WADC TR 59-604.

AN EVALUATION OF MACRO-TEST PROCEDURES FOR DETERMINING THE MECHANICAL PROPERTIES OF REINFORCED PLASTICS. Richard J. McBride. AF 33(616)-5500. February 1960. ASTIA Document No. AD 235127.

This report presents the results of tensile, compressive, and flexural tests conducted on a variety of test specimens to establish macro-test procedures for the evaluation of reinforced plastic materials. This macro-test program is an initial step in the solution of a more difficult and complicated program area involving the development of micro-test methods. Room temperature tests were conducted on polyester, phenolic, heat resistant polyester and silicone laminates. Modulus of elasticity measurements were taken on macro-size tensile, compressive, and flexural specimens of polyester laminates. Based on the data obtained, the use of these macro-specimens as a means of obtaining initial strength and modulus of elasticity properties of newly developed polymers and resin systems is recommended whenever only limited quantities of the materials are available.

Stanford Research Institute. WADC TR 59-668. Part I.

A STUDY OF THE MECHANISM OF ABLATION OF REINFORCED PLASTICS. R. Y. Mixer, C. W. Marynowski. AF 33(616)-5964. February 1960.

Laminated specimens made from three resins (phenolic, silicone, and melamine) and five reinforcements ("E" glass, Refrasil, asbestos, nylon, polyethylene), with varying resin contents and fiber orientations, were exposed to various selected enviroments (oxygen and nitrogen r-f discharges, an arc-image in air and argon, and an argon plasma jet). The chemical phenomena that occurred during ablation were studied by means of analyses of gaseous decomposition products, and examination of emission spectra from the boundary layer. These phenomena were correlated with physical properties and weight loss data. The severity of these environments was compared. Novel experimental methods for determining plasma jet flux densities and specimen thermal diffusivities are described.

Forest Products Laboratory. WADC TR 52-184, Sup 6.

SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON COMPOSITE CONSTRUCTION FOR FLIGHT VEHICLES. Donald G. Coleman. AF 33(616)-58-1. February 1960. ASTIA Document No. AD 236668. PB 149183. Order from LC, Mi $1.80, Ph $1.80.

Developments in the program of research in composite construction for flight vehicles conducted by the U. S. Forest Products Laboratory during 1958 are summarized. In general, the approach has been to derive design criteria mathematically, and then to check by test. Four technical reports issued during the fiscal year are abstracted.

Owens-Corning Fiberglas Corporation. WADD TR 60-24.

HIGH MODULUS, HIGH TEMPERATURE GLASS FIBERS FOR REINFORCED PLASTICS. Philip J. Frickert, Ralph L. Tiede, Helmut I. Glaser, Allan B. Isham. AF 33(616)-5802. March 1960. ASTIA Document No. AD 251783.

A glass was developed which had a forty-five percent higher modulus of elasticity than the commercial glass used in the fibrous glass industry. Glass filament strands of this glass were produced at a rate of several pounds per hour from a single fiber-forming position and these strands were twisted and plied into yarns for weaving. 181-style cloth was woven, heat cleaned, and finished for the preparation of glass-plastic laminates. Strength and modulus of elasticity properties of laminates tested under standard conditions, and after fluid and thermal exposures, are reported.

Armour Research Foundation. WADC TR 59-600. Part I.

MECHANISM OF REINFORCEMENT OF FIBER-REINFORCED STRUCTURAL PLASTICS AND COMPOSITES. J. S. Islinger, K. Gutfreund, R. G. Maguire, O. H. Olson. AF 33(616)-5983. March 1960. PB 161498. Order from OTS $1.25.

This research effort was undertaken to study the mechanism of reinforcement in fiber-reinforced structural plastics and composites. Analytical and experimental approaches included physical-chemical investigations of fiber-matrix interfaces, optical investigations of composites, and studies of the mechanical aspects of reinforcement.

Data are presented for chemisorption studies and for microscopic investigations of fiber-resin composites using multiple beam interference microscopy.

Several analytical techniques are summarized for describing the mechanical behavior of fiber-reinforced composite materials. Included are newly developed theories for the behavior of prismatic bars reinforced by uniformly distributed, parallel, short fibers, and for the transverse interface stress produced in axially loaded parallel fiber-resin composites.

WADC TR 60-90.

THERMAL PARAMETERS OF RE-ENTRY ABLATIVE PLASTICS. Donald L. Schmidt. March 1960.

The unique behavior of plastic materials in very high temperature environments provides a means for hyperthermal shielding of reentry vehicles. High aerodynamic heating rates are accommodated in a self-regulating fashion by the ablative degradation of the material.

The ablation of plastic materials is a complex energy dissipative process in which chemical degradation, phase changes, and gas injection into the boundary layer take place with an attendant removal of surface material. The magnitude of incident energy absorbed and dissipated in the ablative process depends critically upon certain materials and environmental parameters.

This report presents the important chemical and physical aspects of plastics ablation, with major emphasis on the energy transfer processes involved. The absorption and dissipation of heat by internal conduction, phase changes, mass transfer, chemical reactions and radiant emission are reviewed. From the analytical treatment presented, certain desirable characteristics of ablative plastics are given.

WADD TR 60-203.

OPTICALLY TRANSPARENT MATERIALS FOR SPACE FLIGHT VEHICLES. Robert E. Wittman. April 1960.

Requirements for optically transparent materials in manned re-entry, orbital or general space flight vehicles for human vision are outlined.

A general survey to define the conditions imposed by natural and induced environments on these materials is reported.

The present capabilities of transparent materials as related to the predicted environments to be imposed on space flight vehicles are included. Resultant conclusions for research and development to improve certain properties of presently available optically transparent materials are given.

Cornell Aeronautical Laboratory, Inc. WADC TR 59-368, Part II.

THE STUDY OF ABLATION OF STRUCTURAL PLASTIC MATERIALS. Franklin A. Vassallo, Norman E. Wahl, Gerald A. Sterbutzel, John L. Beal. AF 33(616)-5683. April 1960.

Results of ablation research conducted on reinforced plastic materials at moderately severe heating conditions are reported. The materials investigated include laminates of melamine, phenolic, and silicone resins reinforced with glass fabric as well as phenolic and silicone asbestos laminates. Experimental data are given for rate of material loss, effective heats of ablation, and depth of material degradation. The exposure apparatus provides a stream of high temperature nitrogen which is directed onto test specimens causing ablation to take place. The principal body shape considered is the 1/2" flat faced dowel.

High temperature exposures of composite bodies in which a substrate of high thermal conductivity is bonded to a layer of ablative material are reported. Experimental data include measurements of substrate temperature rise and rate of ablation. The temperature data are shown to be satisfied by the equation

$$\frac{Ts - To}{Tc - To} = 1 - \left(1 - \frac{vt}{Lo}\right)^{\frac{1}{\frac{\pi}{2} n \left(1 - \frac{vt}{Lo}\right)}}$$

which is developed in the report. Duscussion of heat sink effectiveness of the substrate is given.

Pittsburgh Plate Glass Company. WADD TR 60-72.

IMPROVED HIGH TEMPERATURE RESISTANT TRANSPARENT PLASTIC MATERIALS. Earl E. Parker, Thomas L. St. Pierre, Walter E. Stickley, Edward A. George, Michael R. Dzugan. AF 33(616)-5602. May 1960.

This research effort was initiated in an effort to improve the high temperature capabilities of one chemical type of optically transparent plastic, for use in Air Force flight vehicle glazings. Specific improved formulations achieved many of the mechanical and optical target properties.

Attempts to orient the improved formulations toward a resistance to crack propagation met with very limited success. Detailed descriptions of the orientation procedures developed and subsequent results are included.

National Bureau of Standards. WADC TR 53-192, Part XIV.

MECHANISM OF RAIN EROSION - PART XIV. Pits in Metals Caused by Collision with Liquid Drops and Rigid Steel Spheres. Olive G. Engel. AF 33(616)-59-3. May 1960. ASTIA Document No. AD 234625.

A pit-depth-versus-velocity equation developed earlier for high-speed collision of liquid drops and soft, ductile metal spheres against targets of the soft and medium-hard metals was tested further with experimental data obtained using target plates of electrolytic tough pitch copper, 1100-0 aluminum, and 2024-0 aluminum, the static strength properties of which were measured by testing tensile specimens. The projectiles used to produce the pits were mercury drops, water drops, and steel spheres. It was found that the numerical constants in the equation are different for projectiles that flow during and as a result of the collision than for projectiles that do not flow (hardened steel spheres). Curves calculated by the equation were found to be in acceptable agreement with experimental pit-depth-versus-velocity data for collisions of the indicated projectiles with target plates of the three metals used, with the exception of the case of steel-sphere impingement against 2024-0 aluminum alloy. In this case work-hardening of the target metal seems to foster a mode of pit formation that was not considered in the development of the pit-depth-versus-velocity equation.

Thermodynamics Laboratories. WADC TR 59-459.

EFFECTS OF HIGH TEMPERATURE, HIGH VELOCITY GASES ON PLASTIC MATERIALS. Frank P. Baltakis, Donald E. Hurd, Roy F. Holmes. AF 33(616)-3421. June 1960.

Twenty blunted, conical specimens of plastic materials were exposed to a Mach 1.82, 6300°F total temperature rocket exhaust jet to determine their erosion characteristics.

These specimens were composed of phenolic, epoxy, or Teflon resin in various proportions to Refrasil, extracted natural silicate, asbestos, glass, cotton, ceramic fiber, or nylon reinforcement in varied orientations.

The lowest stagnation point erosion rate was experienced by a Refrasil-phenolic specimen with "perpendicular-to-base" reinforcement orientation. The next lowest erosion rate was displayed by an extracted silicate-phenolic specimen with "cloth-wrap" reinforcement orientation.

Composition and properties of the erosion environment are presented.

Armour Research Foundation. WADC TR 59-600, Part II.

MECHANISM OF REINFORCEMENT OF FIBER-REINFORCED STRUCTURAL PLASTICS AND COMPOSITES. J. S. Islinger, K. Gutfreund, J. Brauskiewicz, O. H. Olson. AF 33(616)-5983. June 1960.

This is a continuation of the research effort undertaken to study the mechanism of reinforcement in fiber-reinforced structural plastics and composites. Analytical and experimental approaches include physical-chemical investigations of fiber-matrix interfaces, optical investigations of composites, studies of the mechanical aspects of reinforcement and investigations of the feasibility of graphite whiskers as reinforcements.

Data are presented for chemisorption studies aimed at determining the nature of interfacial bonds and the function of each component in the glass-surface treatment-resin composite. Microscopic studies of fibers terminating within a resin are described, together with interference microscopy investigations.

Data are also presented for flexure tests of polyester specimens reinforced by a single layer of glass fibers located near one surface with fiber placement and treatment variations. A limited study of residual stresses in such a composite by analysis and by photoelastic methods is also presented.

The development of micro-tensile specimens for assessing the feasibility of graphite whiskers as reinforcements for plastics is described.

WADD TR 60-380.
REINFORCED PLASTICS AT VERY HIGH TEMPERATURES. Donald L. Schmidt, George P. Peterson. June 1960.

The recent development of thermally stable reinforced plastics is an important contribution to the advancement of high temperature materials technology. Heat resistant reinforced plastics are presently available for extended use at temperatures up to about 500°F, and short-time use at temperatures up to about 1,000°F. These materials also have structural insulative capabilities in extremely high temperature environments (over 5,500°F), like those encountered in hypersonic re-entry and advanced propulsion systems.

A general review of high temperature reinforced plastic materials is presented in this report. Structural properties and characteristics of various plastics at temperatures up to 1000°F are discussed, using illustrative data. The behavior of reinforced plastics in very high temperature environments is briefly reviewed. Performance characteristics are given in terms of both materials and environmental variables.

Forest Products Laboratory. WADD TR 60-177, Part I.
STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES AT ELEVATED TEMPERATURES - PART I. Phenolic-Asbestos, R/M Pyrotex Felt Style 41-RPD. Kenneth H. Boller. DO 33(616)-58-1. June 1960.

Several reinforced plastic laminates that show promise of having good strength properties at elevated temperatures are being tested to determine their strength and elastic properties. This report, the third of the series, presents results of mechanical tests on a phenolic resin laminate reinforced with an asbestos felt (R/M Pyrotex Felt Style 41-RPD). Data were obtained after exposure periods of up to 1,000 hours from such tests as weight loss, flexure, tension, compression, interlaminar shear, and bearing. Most of the tests were made parallel to the machine direction of the asbestos sheets to determine the effects of high temperature and time on the strength properties. Tension tests were made at both 0° and 45° to machine direction to obtain data from which edgewise shear strength and modulus of rigidity can be calculated. Creep and stress-rupture data were obtained under both tension and compression loads.

In general, the strength properties of the phenolic-asbestos laminate decrease with increases in temperature. Specifically, the strength properties decrease with the first application of heat but then often increase with continued exposure or increased temperatures up to 500°F. Finally, after some critical time at temperature, the properties decrease with additional time at temperature. All tests except interlaminar shear show that over 50% of the room-temperature strength was retained when laminates were exposed for 1/2 hour at temperatures up to 800°F. The specific magnitude of these strengths are presented in tables and curves.

WADC TN 59-251.
DEGRADATION EFFECTS OF HIGH INTENSITY THERMAL RADIATION ON PLASTIC RADOME MATERIALS. Rex W. Farmer. July 1960.

The thermal degradation characteristics of three representative reinforced plastic constructions were investigated at incident radiation intensities of 10 and 20 cal/cm^2-sec using a carbon arc heat source.

The glass fabric reinforced materials studied included a polyester laminate and plastic sandwich construction with honeycomb and alkydisocyanate foam cores. Evaluation criteria, based upon visual examination of the test specimen, were defined in terms of the total incident energy required to cause various levels of degradation.

Goodyear Aircraft Corportation. WADC TR 59-656.
DETERMINATION OF PROPERTIES OF SANDWICH MATERIALS SUBJECTED TO RAPID RISING TEMPERATURES SUMMARY TECHNICAL REPORT. AF 33(616)-5762. July 1960. ASTIA Document No. AD 246543.

This report presents the results of an investigation of the mechanical properties of metal and plastic sandwich construction determined under rapid surface heating conditions for maximum surface temperatures up to 2000°F and maximum rate of surface temperature rise of 100°F to 200°F per second.

The evaluation of nine types of sandwich constructions included tests in the 650°F to 1000°F range for the plastic faced specimens, between 700°F and 900°F for aluminum and bonded stainless steel specimens, and 1300°F to 2000°F for the brazed and resistance welded types. The exposure periods at temperature ranged between 10 and 90 seconds. The testing loads selected ranged from 20 percent to 80 percent of room temperature ultimate strengths.

The comparative performance of the different groups of sandwich specimens can best be summarized by the results of the edgewise compression static tests. Deflection readings of the specimens were reported during the thermal shock period and are found in the tables.

Cornell Aeronautical Laboratory, Inc. WADD TR 60-125.
THE EFFECTS OF HIGH VACUUM AND ULTRAVIOLET RADIATION ON PLASTIC MATERIALS. Norman E. Wahl, Roy R. Lapp. AF 33(616)-6267. July 1960. ASTIA Document No. AD 245211.

This study was concerned with the behavior of plastic materials exposed to simulated conditions of pressure, temperature and near ultraviolet as encountered outside the earth's atmosphere.

Glass fiber reinforced plastic laminates, of three types, were exposed to vacua in the order of 10^{-6} to 10^{-7} mm Hg and ultraviolet flux ranging from 1/3 to 2-1/2 times the solar constant of 2 calories cm^{-2} min^{-1}. The periods of exposure ranged from 3 to 500 hours. The equilibrium temperatures of the laminates varied from 250° to 465°F depending on the intensity of the ultraviolet employed.

After exposure to ultraviolet radiation equivalent to the solar constant and vacuum, for periods up to 500 hours, no great change in dimensions or loss in weight of the laminates was observed. The strength of the polyester specimens, however, tends to increase while the phenolic and epoxy laminates decrease slightly in strength.

Laminates exposed to ultraviolet flux of 4.9 calories cm^{-2} min^{-1} and vacuum for three hours show considerable loss in weight and flexural strength.

Bendix Products Division. WADD TR 60-110. THERMAL PROTECTIVE SURFACES FOR STRUCTURAL PLASTICS. James W. Vogan. AF 33(616)-6393. July 1960. ASTIA Document No. AD 244760.

An exploratory materials research program has been conducted on high temperature insulating and erosion resistant surface materials, which are suitable for short time thermal protection of structural plastics.

New heat insulating material composite systems were investigated. These multicomponent structural elements were composed of an outer refractory surface layer, a thin metallic film, a porous inorganic heat insulating layer and various substrate plastics, with all elements integrally, mechanically bonded together.

The individual components of the protective surfaces were evaluated for desirable properties using subsonic and supersonic high temperature gas streams. With the aid of these results composite heat-protective surfaces were formulated and evaluated in the same test environments. Experimental results showed a significant lag in heat penetration into the substrate plastic during hyperthermal exposure. Tungsten faced ceramics exhibited good thermal erosion resistance and heat insulation in high temperature reducing atmospheres, and nickel-zirconia protective surfaces were optimum in high temperature oxidizing environments.

The principal problems encountered with the heat insulating composite systems were suitable methods of fabrication and maintenance of structural integrity in the interface regions during test exposure. Considerable progress was achieved on these two problem areas, and technical effort is continuing. In addition, new and improved heat protective surfaces and exploratory methods of fabrication were investigated.

Rohm & Haas Company. WADD TR 60-402. EVALUATION OF BIAXIALLY STRETCHED TRANSPARENT PLASTICS. D. A. Hurst. AF 33(616)-3994. August 1960. ASTIA Document No. AD 245276.

This program was conducted in an effort to improve the crack propagation resistance by hot stretching methods, of a developmental thermosetting type of transparent plastic material. This material, a modified acrylic known as Selectron 400, has an elevated temperature structural capability well above that of any plastic glazing material available in quantity today.

Conditions under which Selectron 400 can be stretched without breaking were very critical and were not fully successful. Three basic processing steps were required; hot stretching without causing an appreciable amount of cross-linking; relatively low temperature irradiation to cause sufficient cross-linking of the stretched material to prevent relaxation of the degree of stretch at elevated temperatures; oven treatment at an elevated temperature to produce the cross-linking action. A great deal of tearing and breakage of sheets resulted in very low yields of stretched material. The effect of varying the stretching conditions were studied with only mild success in improvement.

Crack propagation resistance of the cross-linked stretched Selectron 400 was very poor. In order to determine whether the limited amount of stretching possible to obtain had any significant effect on the crack propagation resistance, tests were conducted on stretched material without cross-linking. The results indicated that hot stretching essentially produced no gain in crack propagation resistance.

WADD TR 60-101. CONFERENCE ON BEHAVIOR OF PLASTICS IN ADVANCED FLIGHT VEHICLE ENVIRONMENTS. H. S. Schwartz. September 1960. ASTIA Document No. AD 247100.

This report is the collection of papers presented at the Materials Central, WADD conference on "Behavior of Plastics in Advanced Flight Vehicle Environments" held in Dayton, Ohio, on 16-17 February 1960.

The purpose of this conference was to review the recent progress in studies on plastics exposed to severe thermal environments and simulated extremely high altitude environments. The papers presented are primarily on internal and contractual programs sponsored by the Plastics Branch of the Nonmetallic Materials Laboratory. Papers on work performed by other organizations in the Department of Defense and by their contractors were also presented and are included.

Forest Products Laboratory. WADD TR 60-202. EFFECT OF ENVIRONMENT ON MECHANICAL PROPERTIES OF GLASS FABRIC PLASTIC SANDWICH. P. M. Jenkinson, E. W. Keunzi. DO 33(616)-58-1. September 1960. ASTIA Document No. AD 247160.

This report presents the results of flatwise flexure and edgewise compression tests on three constructions of glass-fabric plastic sandwich that were exposed at the seashore in south Florida for 3 months and for 1 year. Included are results of tests of sandwich panels that were subjected in the laboratory to 100°F and 100 percent relative humidity for 16 months. The three constructions had facings approximately 0.03 inch thick of glass-fabric laminate treated with polyester, epoxy, and heat-resistant phenolic resins. Two panels of polyester sandwich were evaluated, one painted and one unpainted. Cores were of glass-fabric honey-comb treated with the same type of resin as the facings except that the core for the sandwich with epoxy resin facings was treated with nylon-phenolic resin.

Tests of all constructions were made at room temperatures, and also at 500°F for the heat resistant sandwich.

The unpainted polyester sandwich panel deteriorated the most in 1 year's weathering, with both edgewise compressive and shear strengths being reduced 14 percent. Painting the other polyester panel with a MIL-E-7729 enamel of the type normally used on exterior reinforced plastic aircraft parts prevented loss in edgewise compressive strength and reduced the loss in shear strength to 6 percent. In comparison, unpainted polyester solid laminate panel 1/8-inch thick also had compressive strength reduced 14 percent by 1 year's weathering.

At 500°F the heat-resistant phenolic sandwich panel showed a 13 percent increase in edgewise compressive strength after a year's weathering, compared to the control value at 500°F.

Exposure to 100°F, 100 percent relative humidity for 16 months reduced edgewise compressive strength by 47 percent for the unpainted polyester sandwich panel, 51 percent for the painted polyester sandwich panel, 24 percent for the epoxy sandwich panel, and 29 percent for the heat-resistant phenolic sandwich panel. Exposure of 1/8-inch thick solid laminates to 100°F and 100 percent relative humidity for 1 month reduced compressive strengths 27 percent for polyester, 13 percent for epoxy, and 15 percent for phenolic laminates.

The Ohio State University. WADD 60-393. EVALUATION OF BRAZED HONEYCOMB STRUCTURES. Robert C. McMaster, Anthony T. D'Annessa, Henry W. Babel. AF 33(616)-6364. September 1960. PB 171408. Order from OTS $5.00.

The final report on U. S. Air Force Contract No. AF 33(616)-6364, administered by the Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division, summarizes the results of a one-year study of nondestructive test methods for the examination of brazed and welded joints in complex shapes. The investigation was directed, by mutual agreement, to commercially-available nondestructive test methods for evaluation of the quality and serviceability of brazed honeycomb structures. Applicable nondestructive test methods were found to include (1) penetrating radiation tests, (2) ultrasonic tests, and (3) thermal tests. Numerous experimental and production parts and brazed honeycomb assemblies provided by government and industrial facilities were examined by each test method and the results were correlated and evaluated. It was found that several commercially-available nondestructive test methods provide reliable and detailed indications of surface layer discontinuities such as core-to-skin filleting and metal-to-metal bonds in edge members. X-ray test methods alone provided reliable and detailed indications of internal conditions such as crushed core, node flow, vertical-tie braze areas, and repaired cells.

Penetrating radiation test methods evaluated included: (1) X-ray film radiography, (2) xeroradiography, (3) direct fluoroscopy, (4) fluoroscopy with television image amplifiers, (5) fluoroscopy with electrostatic image amplifiers, (6) large-area photoconductive screen television image amplifiers, and (7) small-area photoconductive screen television image enlargers. Under properly applied and controlled test conditions, each of these X-ray inspection systems was found usable in the inspection of brazed honeycomb structures.

Ultrasonic test methods evaluated included: (1) immersion ultrasonic tests with focussed transducers, (2) contact ultrasonic tests with resonance transducers, and (3) contact ultrasonic tests with pulse-reflection transducer systems. Detailed images of surface-layer conditions and discontinuities were obtained only with immersion ultrasonic tests with focussed transducers and high-resolution C-scan facsimile or memory-tube recorders. Large-area voids were detectable, under carefully-controlled test conditions, with contact ultrasonic tests with swept-frequency resonance transducer systems.

Thermal test methods evaluated included: (1) thermal-flash proof tests, (2) thermal-gradient tests with heat-repelled fluid coatings, (3) thermal-gradient tests with thermographic phosphor coatings, (4) thermal tests with temperature-sensitive color-changing paint coatings, and (5) thermal tests with remote infrared sensing systems. Reliable detailed images of surface-layer conditions and discontinuities were obtained with thermal gradient tests with transient front-side panel heating and heat-repelled fluid coatings that produced permanent images on the surfaces of the test specimens. Large-area voids were detected by bulging of the skin materials in thermal-flash proof tests. Other thermal test methods described are experimental in nature and deserve consideration for further development.

Complete detail indications have been confirmed for numerous test specimens whose surface-layer discontinuities were revealed by proper application and control of the following nondestructive test methods:

1. Film radiography
2. Xeroradiography
3. Direct fluoroscopy
4. Fluoroscopy with electrostatic image tubes
5. Fluoroscopy with photoconductive screen-television systems
6. Immersion ultrasonic testing with focussed transducers and C-scan facsimile recording systems
7. Thermal-gradient tests with front-side transient heating of panels coated with heat-repelled fluid media

Numerous specimens containing surface-layer discontinuities have been subjected to destructive peel tests or to metallographic sectioning and examination. The results of these destructive examinations confirm the nondestructive test indications produced by the methods listed above.

Further research and development are recommended for the following experimental nondestructive test methods:

1. Xeroradiography
2. Photoconductive-screen television systems, particularly those which permit image enlargement without deleterious loss of image detail
3. Thermal-gradient nondestructive tests with both direct coatings or remote sensing systems which respond to thermal gradients rather than to absolute temperatures.

Although brazed honeycomb assemblies can be inspected reliably and in detail by methods commercially available at present, the most effective test systems are presently slow and costly. Further developments are recommended for those systems which offer promise of adequate detail sensitivity and reliability, but with which the tests can be made faster and at lower costs.

Forest Products Laboratory. WADD TR 60-466. STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES AT ELEVATED TEMPERATURES (Epon 1031 Resin and 181-Volan A Glass Fabric). Kenneth H. Boller. DO 33(616)-58-1. September 1960. ASTIA Document No. AD 247437.

Several reinforced plastic laminates that show promise of having good strength properties at elevated temperatures are being tested to determine their strength and elastic properties. This report, the fourth of the series, presents results of mechanical tests on an epoxy resin laminate reinforced with glass fabric (Epon 1031 with 181-Volan A). Data were obtained after exposure periods from 2 minutes to 1,000 hours from such tests as weight loss, flexure, tension, compression, interlaminar shear, and bearing. Most of the tests were made parallel to the warp of the glass fabric to determine the effects of time and above-normal temperatures on the strength properties. Tension tests, however, were made at both 0° and 45° to the warp to obtain data from which edgewise shear strength and modulus of rigidity can be calculated. Creep and stress-rupture data were obtained under both tension and compression loads.

In general, the strength properties of this epoxy glass fabric laminate decrease with increase in temperature at constant periods of exposure. Specifically, however, the resin softens and the strength of the laminate decreases with the first application of heat, but then at constant temperature the strength properties often increase. Finally, after some critical time, additional exposure causes the properties to decrease. The specific magnitude of these strengths are presented in tables and curves.

WADD TR 60-314. THE THERMAL DIFFUSIVITY OF A STRUCTURAL PLASTIC LAMINATE AT ELEVATED TEMPERATURES. Rex W. Farmer. September 1960.

The applicability of previously developed relations for dynamically measuring the thermal diffusivity of structural plastic laminates is briefly examined. The thermal diffusivity was found to be an "apparent" or "effective" value for these materials above their degradation temperature with accompanying heat of reaction effects. Some exploratory experiments at temperatures up to 1,000°C are described for a phenolic resin/glass fabric laminate using a radiant heating source.

WADD TR 60-176.
FRACTURE STUDIES OF TRANSPARENT AIRCRAFT MATERIALS. Herschel L. Smith, William E. Anderson. AF 33(616)-57-32 and AF 33(616)-58-16. October 1960. ASTIA Document No. AD 251782.

This study was initiated to assess the relative shatter resistance of stretch oriented, as-cast, and laminated acrylics as a function of temperature and material thickness.

Simulated air frame enclosure conditions were used in the laboratory program. Formed semi-cylinders, internally pressurized, and under controlled temperatures were subjected to impact and penetration by high velocity projectiles.

The stretch oriented acrylic exhibited superior impact fracture resistance over the entire temperature range investigated. In addition, the results indicate that substantial weight savings are possible (35% to greater than 65%) without loss of structural integrity or reduced shatter resistance by using stretched acrylics in lieu of as-cast or laminated materials. Individual results are presented.

WADD TR 60-648.
ABLATIVE BEHAVIOR OF PLASTICS IN SUBSONIC AND SUPERSONIC HYPERTHERMAL FLOW. Rex W. Farmer. November 1960. ASTIA Document No. AD 252763.

Materials research was conducted on the ablative behavior of four plastic materials in subsonic and supersonic hyperthermal air/argon environments. Small hemisphere cylindrical models of the experimental materials were exposed to the very high temperature gas stream, which was generated by a stabilized electric arc. Environmental conditions used were: stagnation enthalpies from 750 to 2,600 Btu/lb, free stream Mach numbers of 0.9 and 1.8, and stagnation pressures of 1.8 and 4.0 atmospheres.

The material behavior was found to vary with certain environmental and materials parameters. Within the range of environmental conditions studied, inorganic fiber reinforced phenolic resin materials had the lowest erosion rates in supersonic flow while the lowest erosion rates in subsonic low mass flow were observed for a phenolic/nylon material.

Forest Products Laboratory. WADC TR 52-183, Sup 8.
ANNUAL REPORT ON RESEARCH IN ANC-17 HANDBOOK PLASTICS FOR FLIGHT VEHICLES. Donald G. Coleman. DO 33(616)-58-1. November 1960. ASTIA Document No. AD 251746.

Developments in the program of research in plastics for flight vehicles conducted by the U. S. Forest Products Laboratory during fiscal year 1960 are summarized. In general, the approach has been to derive criteria mathematically, and then to check by test. One technical report was issued during the fiscal year and is abstracted.

WADD TN 60-287.
PROPERTIES OF HIGH MODULUS REINFORCED PLASTICS. George P. Peterson. November 1960.

Properties of laminates made with a new high modulus glass fiber designated as YM31A glass, developed by the Owens-Corning Fiberglas Corporation under Materials Central contract, were determined. The work consisted of a determination of the mechanical and electrical properties under a variety of environmental conditions utilizing an optimum resin-finish combination.

The investigation demonstrated that YM31A laminates not only increased the modulus from 25% to 45% over duplicate "E" glass laminates but also had equivalent or higher strength properties. The electrical properties in the lower temperature ranges were approximately equivalent but showed a greater change in dielectric constant with increased temperature in the 200°-500°C range.

Factors which greatly increase the effectiveness of YM31A from both a strength and modulus aspect are minimizing yarn constraint (less interlacing in fabric pattern) and the use of resin systems, such as the epoxy type, which are not only more ductile but exhibit a high adhesive bond strength between resin and glass.

Stanford Research Institute. WADC TR 59-668, Part II.
A STUDY OF THE MECHANISM OF ABLATION OF REINFORCED PLASTICS. D. L. Chamberlain, Jr., D. E. VanSickle, C. W. Marynowski. AF 33(616)-5964. February 1961.

Nonreinforced polyethylene, poly(methyl methacrylate) (Lucite), Teflon, and Phenolic resins were subjected to r-f discharges produced in nitrogen, oxygen, and air atmospheres. The gaseous products of degradation were determined, and possible mechanisms of attack are discussed.

Analyses of gaseous products resulting from arc-image furnace exposures on selected reinforced resins are reported.

A technique for sampling the gaseous boundary layer of an ablating resin in an argon-stabilized plasma jet is described and results are discussed. Boundary layer emission spectra, both line and band, are reported for plasma jet exposures of reinforced resins as a function of both viewing position and period of exposure.

Polymers and Synthesis Studies

WADC TR 55-374.
POLYMERS AND COPOLYMERS OF N-1,1-DIHYDROFLUOROALKYL ACRYLAMIDES. Fred W. Knobloch. April 1954.

A series of six new fluorinated acrylamide monomers have been studied. These included 1,1-dihydrofluoroethyl acrylamide, 1,1-dihydroheptafluorobutyl acrylamide and the N-methyl, N-n-butyl, and N-isobutyl derivatives of the latter.

Homopolymerization of the fluoroacrylamide monomers readily proceeded both in bulk and in solution with benzoyl peroxide. Liquid monomers polymerized in emulsion with persulfate initiators. All homopolymers were thermoplastics which could be cast to yield clear transparent films. The electrical, thermal, and dilute solution behavior of some of these materials has been studied.

Copolymerisation proceeded with a number of comonomers which included alkyl acrylates, fluoroalkyl acrylates, vinyl ethers,and several dienes. Essential parameters such as monomer reactivity ratios,intrinsic viscosities and empirical slope constants k' were evaluated for selected copolymers.

Harvard University. WADC TR 54-102. ORGANOSILOXANE POLYMERS CONTAINING POLAR GROUPS IN THE SIDE CHAINS. Sanders D. Rosenberg, Eugene G. Rochow. AF 33(616)-479. July 1954. ASTIA Document No. AD 43063. PB 135450. Order from LC, Mi $3.00, Ph $6.30.

In accordance with the proposed objectives of this research a study of the possible synthesis of nitrogen and boron containing polymerizable silicon monomers of the p-$R_2MC_6H_4Si(CH_3)X_2$, (where M is either nitrogen and boron and X is a hydrolyzable group like chloro or alkoxy) was undertaken. Two nitrogen-containing silicon monomers methyl-p-dimethylaminophenyldichlorosilane and methyl-p-dimethylaminophenyldimethoxysilane were prepared by reacting p-dimethylaminophenyllithium with methyltrichloro-and methyltrimethoxysilane, respectively.

Various methods were attempted to prepare a suitable boron-containing silicon monomer without success. Incidental with these attempts it was found that p-bromophenyldichloroboron could be prepared by disproportionation of tri-p-bromophenylboron and boron trichloride. Symmetrical organoboron compounds were prepared by reacting the suitable Grignard reagent with boron trichloride or trimethyl borate. Di-n-butyl-and di-n-propylbromoboron were prepared by reacting the respective symmetrical compounds with bromine.

It was possible to prepare Grignard reagents of methyl-p-bromophenyldiphenoxysilane and methyl-p-bromophenyldi-o-cresoxysilane. Only the latter reagent proved to be of use in subsequent reactions.

Trimethylallylsilane and trichlorosilane were condensed, under the activation of benzoyl peroxide, to form trimethyl-3-trichlorosilylpropysilane. Under similar conditions, trimethylallylsilane did not condense with methyldichlorosilane. It is planned to attempt to condense trichlorosilane with triallylboron and di-p-propylallylboron.

Reference has been made to the difficulties involved in the analysis of organosilicon and organoboron compounds, and some suggestions have been made. From the infrared spectra of the organoboron compounds that were prepared a tentative assignment of the carbon-boron absorption frequency has been made at 1370-1390 cm^{-1}. Three tables containing the new compounds prepared during the course of this research are included.

Monomer-Polymer, Inc. WADC TR 54-264. SYNTHESIS OF MONOMERIC MATERIALS. B. David Halpern, Wolf Karo, Leonard Laskin, Philip Levine, Jack Zomlefer. AF 33(616)-252. December 1954.

In the course of this project, the possibility of preparing the following materials was investigated:

Vinylsilanes
Allyl Sulfides
Divinyl Sulfide
2-Methylthioethyl Vinyl Sulfide
Vinylidene Cyanide
N-(2, 2, 2, -Trifluoroethyl)-acrylamide
N-Alkyl-N-1, 1-Dihydroheptafluorobutylacrylamides
N-Alkyl-N-1, 1-Dihydroheptafluorobutylmethacrylamides
Methylene Silanes
1, 1-Dihydroheptafluoro-1-butyl alpha-(Trifluoromethyl)-acrylate
1, 3-Dicyano-1, 3-butadiene
Heptafluoropropylacrylamide

Prior to the attempted preparation of monomers, a comprehensive literature survey was undertaken.

The modification of Acrylon rubbers was investigated by use of copolymer and terpolymer systems. A preliminary evaluation of the more promising materials was performed.

Samples of monomers and intermediates have been submitted to the Wright Air Development Center.

Bjorksten Research Laboratories, Inc. WADC TR 54-525. RESEARCH ON HEAT-RESISTANT ACRYLATE-SILOXANE ELASTOMERS. Robert P. Cox, Ralph W. Buetow, Luther L. Yaeger. AF 33(616)-2135. February 1955.

The objective of this project was the synthesis of acrylate-siloxane elastomers with good thermal and solvent resistance. Of the many polymers investigated as gum rubber bases, vinyl triethoxy-silane-acrylonitrile-ethyl acrylate compositions were the best as measured by (1) swelling in 70-30 iso-octane-toluene and in Penola oil and (2) weight loss and flexibility after heating in air at 350°F.

The addition of either dibutyl phosphite or polychlorotrifluoro ethylene powder (Kel F) to these gum rubbers generally maintained their flexibility in air at 350°F for 250 to 1000 hours. As a reinforcing filler to provide good tensile strength, carbon black (PhilblackO) was superior to other fillers tested. Trimene Base was effective as a vulcanization accelerator throughout this work.

The vinyl triethoxysilane-ethyl acrylate gum rubbers could be vulcanized without additives through the formation of cross-linkages. Cross-linking probably took place through adjacent>SiOH groups formed in emulsion polymerization.

WADC TR 55-146. ORGANIC DERIVATIVES OF GERMANIUM ORTHO ESTERS OF 2-ALKOXYETHANOLS. Harold Rosenberg, Elizabeth J. Bartholomew, Donal F. Kippax. June 1955. PB 121067. Order from OTS $0.50.

An investigation was conducted to determine the applicability of germanium ortho esters, similar to the organosilicates, as base stock materials for high-temperature fluids and lubricants. A series of tetrakis(2-alkoxyethoxy) germanes was synthesized by the reaction of sodium 2-alkoxyethoxides with germanium tetrachloride after it was found that the germanium halide, unlike its silicon analog, failed to react with a 2-alkoxyethanol. Certain of the physical properties of the germanium ortho esters and their resemblance to those of the corresponding silicon derivatives are discussed. In the course of this work a new glycol ether, 2-cyclohexyloxyethanol, was prepared by the action of ethylene chlorohydrin on the sodium salt of cyclohexanol in the presence of xylene.

WADC TR 54-613 Part 1. ORGANOSILANES AND RELATED COMPOUNDS AS HIGH-TEMPERATURE LUBRICANTS. Part 1: Synthesis and Properties of Dodecyltrialkylsilanes. Harold Rosenberg, Elizabeth J. Bartholomew, James D. Groves, 1/Lt., USAF. July 1955. PB 121003. Order from OTS $0.75.

A series of studies was conducted to uncover new types of organosilicon compounds with suitable properties for extreme-temperature lubricant and hydraulic fluid applications. An introduction to these investigations in the form of a study of the synthesis and properties of one class of unsymmetrical tetraalkylsilanes is presented. A series of dodecyltrialkylsilanes was prepared and certain of the physical properties of these compounds correlated with molecular structure. These materials were found to be liquid over a wide temperature range and, in certain cases, to possess extremely high boiling points. On the basis of preliminary evaluation, these compounds appear to have promise as base stocks for certain extreme-temperature fluid and lubricant applications.

Peninsular CenResearch, Inc. WADC TR 55-220. DEVELOPMENT OF FLUORO-SILICONE ELASTOMERS. Paul Tarrant. AF 33(600)-26593. August 1955. ASTIA Document No. AD 93332. PB 132216. Order from LC, Mi $3.80, Ph $7.80.

The preparation of a homopolymer from 3,3,3-trifluoropropylmethyldichlorosilane has been accomplished on a small scale.

Experimental conditions have been studied for the reaction of trichlorosilane and methyldichlorosilane with olefins. For hydrocarbon olefins, the best yield of addition product was obtained at 250°C without a peroxide catalyst. The use of a platinum catalyst supported on charcoal allows the use of lower reaction temperatures and shorter reaction times in these additions.

The additions of silanes to a number of fluoroölefins have been carried out. Both methyldichlorosilane and trichlorosilane have been employed as addenda and CF_2 $CHCF_3$, CH_2 CHC_3F_7 and CH_2 $C(CF_3)CH_3$ as the fluoroolefins. The best yield by far was obtained using 3,3,3-trifluoropropene which gave a 72% yield of adduct, $CF_3CH_2CH_2$ $SiCl_3$, with trichlorosilane. The physical properties of the various compounds have been determined.

Vinylsilanes have been found to react with molecules such as CCl_3Br, CF_2Br_2, CF_3I, $CF_2BrCFClBr$ and $CF_2ClCFClI$ to give compounds containing halogen atoms on an alkyl side chain. The properties of these compounds have been studied.

Rutgers Univ. WADC TR 54-599. ELASTOMERIC POLYPHOSPHATES. R. A. Hubbard, II, U. P. Strauss. AF 33(616)-2059. August 1955. ASTIA Document No. AD 76871. PB 121516. Order from OTS $1.25.

The research on "Elastomeric Polyphosphates" covered in this report was carried out by R. A. Hubbard, II, and U. P. Strauss at Rutgers University from 1 July 1953 to November 1954. The problem is the preparation of an elastomer based on the high molecular weight polyphosphate chain. Since the known polyphosphates are essentially linear polyelectrolytes occurring in the glassy or crystalline form, the problem consists of two parts. First, the strong interionic forces which cause stiffness of the materials must be overcome; second, the chains must be cross-linked. The first of these problems is attacked by the use of large cations and plasticizers, the second by the use of polyvalent cations; possible chemical methods of tying the chains together are discussed. Some elastomeric products have been obtained, showing the essential soundness of this approach. However, none of the products prepared so far have the required thermal stability, even though the polymeric chain is stable. As a result of this research, we envision the need of a thermally stable cation with a built in plasticizer.

Hooker Electrochemical Co. WADC TR 55-221. INVESTIGATION OF CONDENSATION TYPE ELASTOMERS. George C. Schweiker, Rudolph N. Delco, Russell R. White. AF 33(616)-2421. August 1955. ASTIA Document No. AD 69978. PB 135194. Order from LC, Mi $3.60, Ph $9.30.

The ultimate goal of the exploratory investigations described is the development of a rubber for special Air Force applications. High thermal stability and resistance to fuels, synthetic oils and hydraulic fluids are major requirements for such elastomers.

Various fluorine- and sulfur-containing difunctional compounds have been prepared and high molecular weight polyesters have been synthesized. The linear polyesters made from fluorine-containing glycols exhibit rubber-like properties and are not brittle at low temperatures, in some cases. When fluorine is contained in these polyesters in adequate amounts, they are resistant to hydrocarbon solvents.

Preliminary cross-linking experiments have given a tough elastomer from hexafluoropentylene adipate, without added reinforcing fillers.

The materials described are the first examples of fluorine-containing polycondensation elastomers and appear to be of considerable interest.

WADC TR 55-25. POLYMERS DERIVED FROM DIHYDROPERFLUOROBUTYL ACRYLATE. Fred W. Knoblock, Horace C. Hamlin, Maj., USAF. September 1955. ASTIA Document No. AD 93171. PB 131998. Order from OTS $1.00.

Exploratory copolymerizations of 1,1-dihydroperfluorobutyl acrylate with various other monomers selected from such general classes as 1,3 dienes, unsaturated acids, acrylamides, haloacrylates, vinyl alkyl ethers, vinyl silanes and allyl esters of unsaturated acids are described. Preliminary compounding recipes and evaluation data are presented for those systems which appeared to be of definite interest in connection with the development of specialty elastomers for Air Force applications. Attempts to prepare a copolymer possessing a balance of properties superior to that which exists in poly 1,1-dihydroperfluorobutyl acrylate were successful. However, considerable information was obtained regarding the behavior of 1,1-dihydroperfluorobutyl acrylate in copolymerification reactions and the search for potentially valuable polymers based on this important new monomer was extended to include preliminary investigation of many copolymer compositions not previously studied.

Dow Corning Corp. WADC TR 55-193 Part 2. FLUORINE-CONTAINING POLYETHERS. Ogden R. Pierce, Donald D. Smith, Robert M. Murch. AF 33(616)-2417. February 1956. ASTIA Document No. AD 92587. PB 131227. Order from OTS $1.25.

This research was initiated to explore the preparation of fluorine-containing polyethers for possible use as thermally stable elastomers.

Investigations of several moners were conducted. Indications are that hypohalogenation of fluorine-containing olefins generally is unsuccessful.

The preparation of epoxides by reaction of fluorine-containing olefins with peracids has been briefly explored.

The synthesis of epoxides using fluorine-containing ketones as precursors was studied and the results indicate the approach warrants exploitation.

In the area of polymerization the use of free radical initiators was studied. In no instance was a satisfactory rubber obtained. The polymers prepared were quite solvent-resistant and could conceivably find applications where good rubbery character is not necessary.

Harvard Univ. WADC TR 54-102. Part 2. ORGANOSILOXANE POLYMERS CONTAINING POLAR GROUPS IN THE SIDE CHAINS. Victor D. Aftandilian, Eugene G. Rochow. AF 33(616)-479. February 1956. ASTIA Document No. AD 93800. PB 127902. Order from LC, Mi $2.40, Ph $3.30.

In accordance with the proposed objectives of this research a study of the synthesis of polymerizable organosilicon monomers containing organoboron groups was made. Such monomers are intended to be used in conjunction with nitrogen-containing monomers to increase interchain attraction in the polymer.

A high-vacuum system was built and by this means large quantities of dimethylboron bromide were prepared.

Side-chain chlorination of methylsilanes was carried out by photochemical reactions. In particular, methyl(chloromethyl)dichlorosilane was prepared by this method and was converted to methyl(chloromethyl)bis(o-cresexy)silane, a hitherto unknown compound.

A method was found to convert chlorosilanes to silanes, using lithium aluminum hydride, without reducing chloromethyl groups attached to silicon. Methyl (chloromethyl) silane was prepared for the first time by this process.

Attempts to prepare a Grignard reagent from methyl(chloromethyl)bis(o-cresexy)silane failed, probably because of steric hindrance of the two bulky o-cresexy groups. However, by replacing the o-cresexy groups with hydrogen atoms the preparation of the Grignard reagent was successful.

The reaction of dimethylboron bromide with the Grignard reagent of methyl(chloromethyl)silane appeared to proceed satisfactorily. The product was separated as an adduct of dimethylamine. Many attempts were made to find a suitable solvent for purification of this compound. Although the product contained boron, silicon, hydrogen, and nitrogen, the results of the analysis indicate presence of impurities. The synthesis of polymerizable silane monomers containing organoboron groups thus appears feasible by the methods developed under this project, but further improvements in separation are necessary in order readily to prepare considerable amounts of polymer for evaluation.

WADC TR 55-461.
GAMMA RAY INDUCED ADDITION OF BROMOTRICHLOROMETHANE TO OLEFINS. A. M. Loveless, 1/Lt. USAF, D. A. Rausch, 1/Lt. USAF. April 1956. PB 121279. Order from OTS $0.50.

A new technique in organic synthesis has been applied to the preparation of intermediates for monomers to be used in the polymerization of new useful fluid plastic and elastomeric material for high temperature applications in military aircraft. The addition of bromotrichloromethane to olefins has been effected with gamma radiation. The well-known free radical addition of bromotrichloromethane to olefins has been studied utilizing Cobalt^{60} as the energy source for the generation of free radicals. It has been found that additional reactions can be conducted in this manner resulting in good yields of the simple one to one addition products. No unexpected side reactions have been encountered and the adducts of bromotrichloromethane with 1-octene (85%), allyl chloride (51%), vinylacetate (70%), cyclohexene (65%) and vinyltrichlorosilane (47%) have been obtained in the yields shown after radiation dosage of 2.25×10^5 roentgens (30 minutes exposure). In all cases the resulting products were shown to be identical with those reported by other investigators using organic peroxides or ultra-violet light.

Using the system of bromotrichloromethane and 1-octene a study of the dependency of yield on radiation dosage was carried out. The variation in molar ratio of the reactants was shown to give a good yield (74%) of 1, 1, 1-trichloro-3-bromononane even when an equi-molar mixture of the methane and olefin was used.

The application to other reactions is being investigated.

Peninsular ChemResearch, Inc.
WADC TR 55-220 Part 2.
DEVELOPMENT OF FLUORO-SILICONE ELASTOMERS. Paul Tarrant, George W. Dyckes. AF 33(600)-26593. May 1956. PB 121394. Order from OTS $0.75.

A study has been made of the effect of various initiators or catalysts on the addition reaction involving a single olefin-silane system. The use of platinum supported on charcoal gives highest yields and allows shorter reaction times and lower temperatures for this reaction.

The preparation of the monomers, $CF_3CH_2CH_2Si(CH_3)Cl_2$, $CH_3CH(CF_3)CH_2Si(CH_3)Cl_2$, $CF_3CF_2CH_2CH_2Si(CH_3)Cl_2$, and $CF_3CF_2CF_2CH_2CH_2Si(Ch_3)Cl_2$ has been carried out on a larger scale and appreciable quantities have been made for polymerization studies.

Compounds resulting from the addition of CF_2Br_2 and CF_2-BrCFClBr to vinyltrimethylsilane have been made to undergo reactions involving loss of halogen. The properties of the new unsaturated compounds have been determined.

The B. F. Goodrich Company. WADC TR 55-453. DEVELOPMENT OF INORGANIC POLYMER SYSTEMS. Carlin F. Gibbs, Harold Tucker, George Shkapenko, John C. Park. AF 33(616)-2744. May 1956. PB 131935. Order from LC, Mi $3.90, Ph $10.80.

Following an initial investigation of modification of phosphonitrile chloride, which was then deemphasized to avoid contractual conflicts, a study of semi-inorganic polymers based upon an aluminum-oxygen-silicon system was begun. The basic reasons for studying such a system were founded upon the known thermal stability of aluminum silicates found in nature.

The most stable difunctional aluminum monomers found were quadricovalent chelates such as mono (1, 3-diphenyl-1, 3-propanediono) aluminum diisopropoxide which melted at 324°C without decomposition.

Reaction of the difunctional aluminum monomers with difunctional silicon monomers has not yet resulted in high molecular weight materials.

Rutgers Univ. WADC TR 54-599 Part 2.
ELASTOMERIC POLYPHOSPHATES. R. A. Hubbard II, U. P. Strauss. AF 33(616)-2059. May 1956. PB 121391. Order from OTS $1.00.

The purpose of the work was to prepare an elastomer whose back-bond is the polymetaphosphate chain. The part with which this report is concerned was the cross-linking of polyphosphate chains. We attempted to prepare the possible cross-linking agent neopentane tetraphosphonic acid and its derivatives. A mixture of acids was prepared by the Arbuzov rearrangement from penta-erythrityl bromide which appears to be primarily the tri-phosphonic acid. Attempts to prepare a cross-linked polyphosphate from this mixture by fusion of the phosphonic acid with KH_2PO_4 at 320°C were unsuccessful, due to the thermal instability of the neopentane carbon structure.

The molecular weights of several samples of potassium polyphosphate were determined viscometrically in order to obtain an idea of the degree of polymerization of the samples used in the cross-linking studies. It was found that the molecular weight of crystallized (KPO_3) x can reach 3×10^6.

Hooker Electrochemical Company
WADC TR 55-221 Part 2.
INVESTIGATION OF CONDENSATION TYPE ELASTOMERS. George C. Schweiker, Russell R. White, Rudolph N. Deleo. AF 33(616)-2421. May 1956.

The ultimate goal of the exploratory investigations described is the development of a rubber for special Air Force applications. High thermal stability (350°F or higher); resistance to aromatic fuels, synthetic ester-base oils and hydraulic fluids; resistance to ozone and to

weathering oxidation effects; resistance to acids, bases, and salts; resistance to abrasion; and satisfactory performance at -65°F or lower are major requirements for such an elastomer.

To this end, a number of linear polyesters have been prepared from fluorine-containing diols and various dicarboxylic acid chlorides, and their properties determined. Hexafluoropentylene adipate (made from adipyl chloride and 2, 2, 3, 3, 4, 4-hexafluoropentanediol) is a rubber-like gum when of sufficiently high molecular weight, and this polyester has been cross-linked to evaluate the properties of the elastomer in the light of Air Force requirements.

When reinforced with carbon black, the polymer exhibits very good physical properties (tensile strengths above 2600 psi with corresponding elongations above 300% at the present stage of development). Preliminary evaluations show that this elastomer is thermally stable (serviceable for extended periods to 350°F or higher in air); very resistant to aromatic fuels, synthetic ester-base oils and hydraulic fluids, and acid and salt solutions at room temperature; and not brittle at -65°F or lower. It is not long resistant at the present stage of development to diester fluids at 350°F, however, or to base solutions at room temperature.

It is believed that this rubber is the first example of a fuel resistant, thermally stable elastomer which does not become brittle at low temperatures.

Aerojet-General Corp. WADC TR 56-90. INVESTIGATION OF INORGANIC AND SEMI-ORGANIC POLYMERS. Samuel Witz. AF 33(616)-2739. May 1956.

A. The present investigation sought to determine whether a polymer containing a Si-O-P or a B-O-P linkage and possessing high-temperature stability and resistance to hydrocarbon fluids could be synthesized.

B. Several new polymeric materials containing a Si-O-P skeleton were synthesized. Most of these solids were low-melting, tacky, brittle resins of low molecular weight and were sensitive to moisture.

C. Fundamental information about the behavior of the Si-O-P linkage toward heat and hydrolysis was obtained through a study of some newly synthesized alkylsilyl phosphates $[R_3SiO]_3PO$ and phosphonates $[R_3SiO]_2P(O)R'$. The hydrolytic stability of the high-boiling phosphates was found to increase with increasing chain length of the silicon substituent. Both the phosphate and phosphonates undergo some decomposition on pyrolysis at 300°C (in vacuo).

D. In a study designed to test the effectiveness of dialkyl alkanephosphonates as heat-stabilizing additives, it was found that the addition of a small percentage (3.5%) of dibutyl butanephosphonate to a commercially available silicone resin improved its thermal properties.

E. Condensation of benzeneboronic and cyclohexanephosphonic acids produced an organoboron phosphate, melting above 300°C, possessing a limited solubility in organic solvents, but soluble in and hydrolyzed by water.

WADC TR 56-94. THE PREPARATION AND PROPERTIES OF SOME NEW FLUORINE-CONTAINING 1, 2-EPOXIDES. D. A. Rausch 1/Lt., A. M. Lovelace, 1/Lt. May 1956. PB 121529. Order from OTS $0.50.

The interest in fluorine-containing polyethers for possible application as thermally stable elastomers prompted research on the preparation of new epoxide monomers.

Six new epoxides were prepared and characterized. The utilization of these monomers in new polymer systems is being investigated.

Univ. of Southern California. WADC TR 56-82. PRINCIPLES OF INORGANIC POLYMERIZATION. Anton B. Burg, Peter J. Slota, Jr., Walter Mahler, Gordon L. Juvinall. AF 33(616)-2743. May 1956.

The object of this research is to discover new patterns of chemical bonding such as might be applicable to the invention of high polymers having unusual thermal stability. It is believed that a considerable number of structural arrangements of nonmetallic elements can be stronger than carbon chains, and more resistant to thermal degradation. Leads in this direction are found as phosphorus-boron bonding is subjected to further study, for the amplification of which the attachment of nonlabile electronegative groups to boron is regarded as useful. The P-N polymer system also seems promising, and methods are sought for placing new and nonlabile substituents on P in (PN)-n rings. Eventually such substituents should include cross-linking units, and this is one reason for studying a newly-discovered variety of fluorocarbon-phosphorus compounds wherein P$\overset{C}{\underset{C}{}}$P rings and $(-C-P-)_n$ chains are indicated.

As indicated in the summary, the work of the first year of this contract has developed a number of promising lines of endeavor, most of which cannot be fully understood and evaluated except through more detailed studies. Hence their utility is a matter for the future to decide.

Rutgers Univ. WADC TR 55-26 Part III. RESEARCH ON BORON POLYMERS. William L. Ruigh, Frank C. Gunderloy, Jr., Michael Sedlak, P. A. Van Der Meulen. AF 33(616)-2057. May 1956. PB 121374. Order from OTS $1.25.

A new, simple, and efficient synthesis of boron substituted borazoles has been developed. Butylboron dichloride reacts with ammonia to give a high yield of B-tributylborazole. This synthesis will be broadened by the employment of substituted amines. Some of the resulting B-N substituted borazoles may serve as the basis of thermally and hydrolytically stable semi-inorganic polymers and the liquid borazoles will be evaluated as lubricants and hydraulic fluids.

The preparation of benzeneboronic acid, tri-n-butylborine, butylboron dichloride and phenylboron dichloride as intermediates for our new borazole synthesis has been studied.

A new catalytic recirculating apparatus for preparing phenylboron dichloride from benezene and boron trichloride by Pace's method is described.

Stanford Research Institute. WADC TR 56-201. RESEARCH ON THE SYNTHESIS OF POLAR SILANE MONOMERS. Robert M. Silverstein, Leon Goodman, Allen Benitez. AF 33(616)-2998. May 1956. PB 121498. Order from OTS $0.75.

This report summarizes the work done between May 1955 and May 1956 on the synthesis of polymerizable polar silane monomers and the copolymerization of certain of these with dimethylsilicone.

The principal method used for the monomer synthesis was the addition of the Si-H linkage to vinyl and allyl monomers. Monomers containing halogens were prepared by the Grignard reaction, by Diel-Alder addition of hexachlorocyclopentadiene to unsaturated silanes, and by the addition of polyhalogenated molecules to unsaturated silanes.

Hydrolysates of certain of the polarsilane monomers strongly inhibited polymerization when mixed in 5 mole percent amounts with the hydrolysate of dimethyldichlorosilane. The polymerization could be forced but the products were then insoluble in benzene.

Rutgers Univ. WADC TR 55-26 Part IV. RESEARCH ON BORON POLYMERS. William L. Ruigh, Arthur D. Olin, Nathan G. Steinberg, Peter A. Van Der Meulen. AF 33(616)-2057. September 1956. ASTIA Document No. AD 110403.

Tri-B-β-chloroborazole has been prepared from β-chlorovinylboron dichloride and ammonia. It is unstable and in the presence of polar solvents forms either cyclic or long chain polymers.

Large samples of 100 grams or more of Tri-B-b-butylborazole, Hexabutylborazole, and Tri-N-Methyl-tri-B-n-butylborazole were prepared for evaluation.

The Pennsylvania State Univ. WADC TR-56-203. COORDINATION POLYMERS. W. Conrad Fernelius. AF 33(616)-2742. October 1956. ASTIA Document No. AD 110425. PB 121867.

● The purpose of the work is to provide research and development on organic metal-coordination polymers, which exhibit exceptional thermal stability.

The concept of producing polymers through coordination is presented, pertinent literature reviewed, and various possibilities of developing such polymers considered.

Chelate polymers of bis (β-diketones) were prepared under varying conditions and their physical properties determined. Molecular weights range up to 6000.

Attempts to form polymers from bis(Schiff bases) and to produce a truly inorganic polymer by the coordination of polyfunctional acids (anions) to complex cations have not yet been successful.

Monsanto Chemical Company. WADC TR 56-399. POLYMER EVALUATION HANDBOOK. C. H. Adams, R. J. Bourke, G. B. Jackson, J. R. Taylor. AF 33(616)-3034. November 1956. ASTIA Document No. AD 110557. PB 121870.

The Polymer Evaluation Handbook provides a system for obtaining a maximum amount of useful information on small quantities of experimental polymers. It also provides background information on principles of evaluation, properties and measurements and their significance, the nature and behavior of polymers and physical characteristics of plastics in relation to each other and to other classes of materials.

The Handbook describes a screening evaluation scheme for effectively and efficiently defining the physical and chemical behavior of small quantities of experimental polymeric materials. The soundness of the evaluation scheme was tested in a pilot laboratory. Examples of the results of these tests are given.

Interpretation concepts are discussed which pertain to the appraisal of evaluation information and data. It is noted that the effectiveness of screening evaluations will depend to a large extent on two major factors: (1) the scientific knowledge, experience, judgment, and perception of the person conducting the evaluation, and (2) a reasonable balance between research laboratory efforts on synthesis and evaluation work.

University of Florida. WADC TR 56-234. PREPARATION OF FLUORINE-CONTAINING COMPOUNDS. H. C. Brown, R. D. Dresdner, J. A. Wethington, Jr., J. A. Young. NONR 580(03)NR 356-333. November 1956. ASTIA Document No. AD 110493. PB 121818.

Part of the objective of this program is the preparation or attempted preparation of new fluorine-containing chemical species by the Simon's electrochemical process. The results of numerous operations in a variety of cells are summarized. Studies have been made with some of the resulting materials in order to find unique reactions of these materials and thereby new compounds exhibiting the chemical and thermal stability associated with fluorocarbons.

The preparation, pyrolysis or thermal reactions of certain simple fluorocarbon sulfides with fluorocarbon olefins are described. The sulphides are CF_3SF_5, C_2F_5SF, $(CF_3)_2SF_4$ and $(C_2F_5)_2SF_4$. The olefins involved were $CF_3CF{=}CF_2$, and $CF_3N{=}CF_2$.

The synthesis and properties of fluorocarbon derivatives having hetero-atoms such as oxygen or nitrogen in the principal carbon chain of the molecule have been studied. It has been shown that a perfluoro compound having an alternating chain C-O-C-N-C cannot be synthesized directly from the organic analogue. When prepared indirectly, however, such a chain is chemically and thermally stable. The compounds $(CF_3)_2NCOF$, $CF_3N\ CF_2$, and $(CF_3)_2NCF_2COOH$, which are the N-hetero analogues respectively of $(CF_3)_2CFCOF$, $CF_3CF\ CF_2$, and $(CF_3)_2\ CFCF_2COOH$, are discussed. The pyrolysis of a compound having C-C, C-O, and C-N bonds is described. Preparation of a new and unexplored class of compounds, the perfluoroamides, and the synthesis of a possible intermediate in the preparation of the difunctional acid $O(CF_2COOH)_2$ are mentioned.

Reaction of fluorocarbon olefins and chlorofluoroolefins has produced sulfur-containing fluorocarbon derivatives table to aqueous alkali. Preparations and reactions of perfluoroalkyl amidines, $R_FC(:NH)NH_2$, and perfluoro-alkyl triazines, $(R_FCN)_3$ have been studied. The reaction of polytetrafluoroethylene with sulfur or selenium under pyrolytic conditions produces a fusible polymer containing no sulfur.

Efforts to use potassium and sodium vapor as a reaction medium for fluorocarbon synthesis have not been successful. Fluorine exchange between metal fluorides and fluorocarbon olefins has been studied. Exchange was generally accompanied by decomposition. It was found that the reactor packing determined the nature of the products obtained from the oxidation of C_3F_6.

Polytechnic Institute of Brooklyn. WADC TR 56-326. CHLOROSULFONATION OF SILANES AND RELATED REACTIONS. C. G. Overberger, F. M. Beringer. AF 33(616)-2997. January 1957. ASTIA Document No. AD 110712. PB 121973.

The aim of this work was the synthesis of polar organo-silicon monomers capable of polymerization to synthetic rubber. The polar groups would be expected to decrease the solubility of the silicone rubber in organic solvents as well as increasing the transition temperature.

The synthesis of a suitable polar organosilicon monomer has not yet been effected. Our results, some of which represent progress toward the desired goal, are now summarized.

1. The direct photochemical chlorosulfonation of silanes with sulfuryl chloride was attempted without success. In one case a small amount of cleavage product, methanesulfonyl chloride, was formed.
2. The direct chlorocarbonylation of tetraethylsilane with oxalyl chloride was effected.
3. Three silanes probably containing the $-CH_2SCN$ group have been prepared. However, attempted conversions to compounds with $-CH_2SO_2Cl$ groups have failed.
4. Vinyltriethoxysilane with methylmagnesium iodide has given vinylmethyldiethoxysilane and vinyldimethylethoxysilane.
5. Benzyl and phenyl mercaptans have been added to the above vinylsilanes to give substituted beta-thioethylsilanes. Attempts to oxidize these sulfides to sulfones have led to mixtures of materials of

higher molecular weight.

6. Hydrolysis of various silane sulfides has given mixtures of silane sulfides, as yet incompletely characterized.

While silicones with polar substituents would still seem to be desirable and useful materials, the synthesis of suitable monomers has not been accomplished in the present work.

Stanford Research Institute. WADC TR 56-530. RESEARCH ON THE SYNTHESIS OF POLAR SILANE MONOMERS. Robert M. Silverstein, Leon Goodman, Allen Benitez. AF 33(616)-2998. January 1957. ASTIA Document No. AD 110717. PB 121887.

The prime objective of this research is to synthesize polymerizable, polar silane monomers for use as precursors or modifiers for improved silicone rubbers. A second important objective of the project is to prepare gums from these monomers, homopolymerized or copolymerized with dimethylsilicone oils, such that the copolymer can be compounded to produce suitable silicone elastomers.

The structures of the $MeSiCl_2H$ adducts with vinyl acetate and with methyl acrylate were shown, by nuclear magnetic resonance spectra, to be $MeSiCl_2CH_2CH_2OCOCH_3$ and $MeSiCl_2C^{CH_3}H\text{-}COOCH_3$, respectively. The latter compound represents the first reported example of non-terminal addition of Si-H compounds to vinyl double bonds.

Samples of dimethylsilicone copolymers containing the vinyl acetate adduct, the allyl acetate adduct, the vinyl ethyl ether adduct, and the allyl ethyl ether adduct were prepared. All of these were cross-linked and were unsuitable elastomer materials.

The reactions of the strong acids and strong bases used as polymerization catalysts, with the hydrolysates of the silane adducts, were shown to lead to C-Si bond cleavage in a number of cases. This cleavage provides a plausible explanation of the cross-linking that results when the adducts are used in copolymerization.

The use of β- and γ- radiation sources led, in preliminary studies, to surface grafts of β-cyanoethyl acrylate onto a commercial silicone elastomer which gave an improvement in resistance to swelling caused by 70:30 isooctane:toluene.

WADC TR 56-492. THE PREPARATION AND POLYMERIZATION OF PERFLUOROALKYL PROPENYL KETONES. D. A. Rausch, 1/Lt., L. E. Coleman, 1/Lt., A. M. Lovelace. March 1957. ASTIA Document No. AD 118158. PB 131055.

A study of the preparation of α,β unsaturated ketones containing fluorine has been undertaken in this laboratory for use as monomers in the preparation of new and useful plastic and elastomeric materials for high temperature applications in military aircraft.

The perfluoroalkyl propenyl ketones were found to copolymerize with a variety of vinyl monomers such as acrylonitrile, ethyl acrylate, styrene and vinyl acetate to give products ranging from powders to elastomers.

Naugatuck Chemical Company WADC TR 56-596. RESEARCH ON HIGH TEMPERATURE LAMINATING RESINS BASED ON MALEIMIDE AND ITS DERIVATIVES. Roger W. Amidon, Charles H. Alexander, Frank B. Root. AF 33(616)-3606. April 1957. ASTIA Document No. AD 118235.

The objective of this contract was the synthesis of laminating resins for service in the range of 600° to 700°F, based primarily on maleimide and on derivatives thereof.

Test laminates were prepared of various blends of maleimide with standard heat-resistant alkyds. Laminates from one such blend had a raw flexural strength of 67,100 psi, as compared to 37,600 and 42,100 psi for VIBRIN 135 and X 1068 laminates respectively. After 24 hours aging at 500°F, it retained a flexural strength of 61,200 psi as compared to 35,400 and 36,000 psi, and after 192 hours at 500°F, 34,500 psi as compared to 19,300 and 18,200 psi, measured at 500°F.

Copolymers of maleimide and methylol maleimide with several vinyl type monomers were prepared and studied as possible heat resistant resin components. None of the materials but one showed any promise of desirable properties, and further investigation along this line is not considered profitable.

Synthesis of three monomeric unsaturated compounds containing the maleimide nucleus was attempted. It appears the synthesis of N-Allyloxymethyl maleimide was successful; however, complete analysis of the reaction product is not yet available.

Midwest Research Institute. WADC TR 57-143. DEVELOPMENT OF THERMALLY STABLE SILICON-CONTAINING RESINS. L. W. Breed, Fred Baiocchi, Howard W. Christie. AF 33(616)-3675. May 1957. ASTIA Document No. AD 118308. PB 131190.

The purpose of this program is the development of thermally stable silicon-containing resins, and the parts of that program already completed are described in full. This program includes the synthesis of silane monomers which contain two silicon atoms connected by arylene groups and have sufficient functionality to form a thermosetting polymer when hydrolyzed and cured. It also includes finding methods of hydrolyzing and polymerizing these monomers in which the maximum use is made of their functionality.

Possible methods for synthesizing the monomers have been investigated. Those methods which were found suitable for the preparation of quantities of one pound or more were used to prepare the following compounds: 1,4-bis(methyldiethoxysilyl)benzene, 4,4'-bis(methyldiethoxysilyl)phenyl-ether, and phenyldichlorosilyl-trichlorosilylbenzene. Also, the description of a statistically designed series of experiments to study the variables effective in the formation of laminates from silicone resins and glass fiber fabric is included.

Hooker Electrochemical Co. WADC TR 55-221 Part III. INVESTIGATION OF CONDENSATION TYPE ELASTOMERS. George C. Schweiker, Burton S. Marks, Russell R. White, Rudolph N. Deleo. AF 33(616)-2421. May 1957. ASTIA Document No. AD 118294. PB 131178.

The ultimate goal of the exploratory investigation described is the development of a rubber for special Air Force applications. High thermal stability (350°F or higher); resistance to aromatic fuels, synthetic esterbase oils and hydraulic fluids; resistance to ozone and to weathering oxidation effects; resistance to acids, bases and salts; resistance to abrasion; and satisfactory performance at -65° F or lower are major requirements for such an elastomer.

Preliminary compounding and testing studies with an elastomer based on a fluorine-containing polyester, made from adipyl chloride and 2,2,3,3,4,4-hexafluoropentanediol, have given promising results. At the present stage of development, compounded and cross-linked specimens of hexafluoropentylene adipate retain useful mechanical properties after aging in air at 400°F (70 and 168 hour tests), and in diester oil at 350°F (70 hour test); are resistant to paraffinic and aromatic fuels; and exhibit a brittle termperature of -98°F (ASTM D-746).

This report describes the preparation, compounding, cross-linking and properties of hexafluoropentylene

adipate elastomer as well as syntheses and properties of other fluorine-containing condensation polymers and difunctional starting materials. Data indicating that increasing fluorine content of the polymers decreases their solubility in common solvents without regard to position of the fluorine in the polymer are extended. On the other hand, these data show that position of the fluorine, rather than total fluorine content of the polymer is of most importance in affecting brittle temperatures.

WADC TR 54-613 Part II
ORGANOSILICON COMPOUNDS. Part II - The Cyclopenthamethylenedialkylsilanes. Dr. Harold Rosenberg, Dr. Christ Tamborski, Marvin D. Rausch, 1/Lt. May 1957. ASTIA Document No. AD 130761. PB 131389.

An investigation into the use of cyclic tetra-substituted organo-silanes was conducted to determine their applicability as base materials for extreme-temperature lubricant and hydraulic fluid applications. A series of cyclopentamethylenedialkylsilanes was synthesized and certain of the physical properties correlated with molecular structure. In addition, one aryl derivative, cyclopentamethylenediphenylsilane, was prepared and evaluated. The cyclopentamethylenedialkylsilanes were found, similar to the unsymmetrical tetraalkylsilanes, to be liquid over a wide temperature range and, in the case of certain members, to be thermally stable at elevated temperatures. These compounds, on the basis of preliminary data, appear to show promise as base materials for certain wide-temperature hydraulic fluid and lubricant applications.

WADC TR 56-493.
PREPARATION AND POLYMERIZATION OF SOME FLUOROALKYLMETHYLCYCLOSILOXANES. Eugene C. Stump, 1/Lt. May 1957. ASTIA Document No. AD 118344. PB 131205.

Four fluoroalkylmethyldichlorosilanes were hydrolyzed and the resulting cyclosiloxanes separated and characterized.

Attempts to homopolymerize the fluoroalkylmethylcyclosiloxanes resulted in viscous fluids.

Gum copolymers with octamethylcyclotetrasiloxane were prepared by conventional methods.

Fluorine-containing cyclosiloxanes may also be prepared from the higher molecular weight siloxane residues of the hydrolysis reaction by passing through a hot tube at 470-520°C.

University of Southern California.
WADC TR 56-82 Part II.
PRINCIPLES OF INORGANIC POLYMERIZATION. Anton B. Burg, Gottfried Brendel, Aimery P. Caron, Gordon L. Juvinall, Walter Mahler, Kurt Modritzer, Peter J. Slota, Jr. AF 33(616)-2743. May 1957. ASTIA Document No. AD 118301. PB 131180.

This project continues to seek and to find new patterns of chemical bonding and to consider their possible usefulness in relation to the invention of new polymers having unusual thermal stability. Some of our aminophosphino-boron resins are stable at temperatures approaching 500°C, and the limit is not yet known; however, the problem of their poor mechanical properties remains. The phosphinoborine bonding principle withstands a great increase in the electronegativity of substituents on phosphorus. The stability of P-P bonding seems much enhanced by CF_3 groups on P, so that the new compounds $(PCF_3)_4$, and $(PCF_3)_5$ are decidedly stable. The previous tentative interpretations of the by-products of the fluorocarbonphosphorus iodide reactions with mercury are reconsidered in the light of new facts. Progress toward more electronegative substituents on boron is reported, and our recently initiated work on phosphinoaluminum compounds has produced some interesting materials. Nitrilophosphoric chloride is not depolymerized by pyridine, but it reacts with triethylamine in an interesting manner.

U. S. Naval Ordnance Laboratory
WADC TR 56-376.
DEVELOPMENT OF THERMALLY STABLE POLYMERIC MATERIALS. Charles P. Haber. AF 33(616)-56-41. June 1957. ASTIA Document No. AD 130798. PB 131253.

The work described within this report has as its objective the synthesis of polymeric materials having extreme chemical and thermal stability which are suitable for use in various aircraft applications such as elastomers, plastics, fluids, dielectrics, and adhesives. In most applications the needed materials must be able to resist the action of the atmosphere at the elevated temperatures; in some, resistance to corrosive fuels and fuel combustion products is required. The task of this program is to investigate promising inorganic bonding systems for thermal and chemical resistance and to incorporate these bonding systems into high polymer molecules with the required physical and mechanical properties.

Diphenylvinylsilane has been prepared in good yields. The polymerization of this material by peroxide-initiated reactions in bulk, solution, and emulsion has led only to low molecular weight, low melting solids of no apparent practical significance. The mechanism of polymerization has been established as the addition of the elements of Si-H bond of one molecule across the vinyl unsaturation of another molecule. Catalysis of the polymerization by a platinum-on-carbon catalysis led to a different type of reaction and a dissimular product tentatively assigned the structure $\phi_2C_2H_3\text{-Si-SiC}_2H_3\phi_2$.

In an attempt to prepare $(CF_3)_2PN$ polymers, a multistep synthesis giving an excellent overall yield of the desired starting material, $(CF_3)_2PCl_3$, was developed. The reaction of $(CF_3)_2PCl_3$ with ammonia and ammonium chloride under a wide variety of experimental conditions led to rearrangement, disproportionation, and ammonolysis of CF_3 groups. As a result of these side reactions, it has not been possible to prepare the desired nitride polymer.

Ford Motor Company. WADC TR 57-321.
SYNTHESIS OF POLYXYLYLYLDIMETHYLSILANE AND POLYXYLYLYLDIMETHYL SILOXANE. Glenn R. Wilson, Gretchen M. Hutzel, Louis B. Updegrove. AF 33(600)-32448. June 1957. ASTIA Document No. AD 130822.

The purpose of this program is to prepare a series of poly-p-xylylyldimethylsilanes and poly-p-xylylyldimethyl siloxanes for evaluation as high-temperature lubricants and/or hydraulic fluids. The major effort has been devoted to finding a suitable procedure for preparing one of the building blocks of the polymers, viz., bis(trimethylsilyl)-p-xylylene and suitable intermediates for synthesizing this latter compound. Many of the approaches for synthesizing the monomeric building block were unsuccessful and resulted in the formation of undesirable side reactions. Only recently was a successful procedure developed which involved the condensation of p-dibromobensene and chloromethyltrimethylsilane with metallic sodium. The application of this successful procedure towards synthesis of the polymeric silane and siloxane are now under investigation.

WADC TR 56-520.
COPOLYMERS OF 1, 1-DIHYDROPHERFLUOROBUTYL ACRYLATE WITH SOME VINYL SILANES. Eugene C. Stump, Jr., 1/Lt. August 1957. ASTIA Document No. AD 118247. PB 131375.

The purpose of this investigatigation was to prepare an elastomer with high temperature resistance as well as improved resistance to the deteriorating and swelling effects of fuels and oils.

Four silane monomers; vinyltriethoxy-, vinylethyldiethoxy-, vinyldimethylethoxy-, and vinyltrimethylsilane were copolymerized with 1, 1-dihydroperfluorobutyl acrylate. The resulting polymers were tested for thermal stability by determining percent weight loss in dry air at 350°F and 400°F. In most cases weight loss of the silane containing polymers was appreciably less than that of the homopolymer poly-1, 1-dihydroperfluorobutylacrylate. The enhanced thermal stability is attributed to a unique type of cross-linking, which is discussed theoretically.

Infrared spectra of the samples were recorded and discussed. Results of compounding, testing, and evaluation will be reported at a later date.

Peninsular Chemresearch, Inc.
WADC TR 55-220 Part III.
DEVELOPMENT OF FLUORO-SILICONE ELASTOMERS George W. Dyckes. AF 33(616)-3238. September 1957. ASTIA Document No. AD 131044. PB131425. Order from OTS $1.75.

Some promising fluoroalkyl silane monomers and polymers have been prepared for use as high temperature and aircraft fuel resistant elastomeric materials.

Appreciable quantities of the monomers, $CF_3CH_2CH_2Si(CH_3)Cl_2$, $CH_3CH(CF_3)CH_2Si(CH_3)Cl_2$, $CF_3CF_2CH_2CH_2Si(CH_3)Cl_2$ and $CF_3CF_2CF_2CH_2CH_2Si(CH_3)Cl_2$ were synthesized for polymerization studies. Hydrolysis reactions were carried out on the monomers and the constant boiling products have been characterized. Polymerization of the cyclic hydrolyses products to elastic homopolymers has been realized using the active metals in Group IA of the periodic table as polymerization catalysts. Copolymers with octamethylcyclotetrasiloxane were also prepared using potassium hydroxide as a catalyst. An attempt to prepare a high molecular weight ternary polymer from the trimer, $[CF_3CH_2CH_2Si(CH_3)O]_3$ and the cyclic tetramers, $[(CH_3)_2SiO]_4$ and $[CH_2=CHSi(CH_3)O]_4$, gave only a medium viscosity oil.

A study was made of the reaction of hydrogen fluoride with the compound, $CF_2BrCH_2CH_2Br$ in attempt to improve the yield of the fluorinated product, $CF_3CH_2CH_2Br$. Improved yields were obtained; however, percent conversion still remains relatively low.

In the preparation of new fluoroalkyl silane monomers, preliminary experiments were carried out on the fluorination of the beta carbon atom on the compounds $CCl_2=CHSi(CH_3)_3$ and $CHCl=CHSi(CH_3)_3$. Analytical results indicate the fluorinated compounds were obtained.

The B. F. Goodrich Company
WADC TR 55-453 Part II.
DEVELOPMENT OF INORGANIC POLYMER SYSTEMS. Carlin F. Gibbs, Harold Tucker, George Shkapenko, John C. Park. AF 33(616)-2744. September 1957. ASTIA Document No. AD 131036. PB 131427.

The purpose of this investigation was to prepare polymeric inorganic or semi-inorganic materials capable of withstanding elevated temperatures.

The investigation of the aluminum-oxygen-silicon system initiated last year has been continued through this year. Previous difficulties in formation of aluminum-oxygen-silicon bonds were traced to exchange reactions. Reaction of aluminum halides with sodium silanolates has been found to lead to formation of aluminum-oxygen-silicon bonds in high yields with no evidence of exchange reactions. By this means, tristriphenylsiloxyaluminum and tristrimethylsiloxyaluminum have been prepared. Attempts to extend this principle to the preparation of polymers using difunctional aluminum halides and silanol salts have not resulted in formation of high polymers, but only polymers very low in molecular weight.

Preparation of trimethylsiloxyaluminum diisopropoxide in high yield by reaction of aluminum isopropoxide with trimethylacetoxysilane indicates that this type of reaction might also be a good one for the establishment of aluminum-oxygen-silicon bonds.

A rubbery tin-silicon polymer containing 8-10% tin was stable upon being heated at 300°C for three hours.

Reaction Motors, Inc. WADC TR 57-45 Part I.
HIGH TEMPERATURE STABLE SEMIORGANIC FLUIDS. Daniel Grafstein, Rita Dudak, Murray S. Cohen, Joseph Green. AF 33(616)-3417. September 1957. ASTIA Document No. AD 131099.

Phenyldichlorofluorosilane and methyldichlorofluorosilane, monomeric materials suitable for the preparation of polyorganofluorosiloxane polymers, were prepared in good yield and good conversion by the reaction of gaseous boron trifluoride with phenyldichloroethoxysilane and methyldichloroethoxysilane. Phenylmethylchlorofluorosilane and phenylmethylfluoroethoxysilane were synthesized and evaluated as chain stopping agents for the organofluorosiloxane polymers. The polymerization of these monomers were studied and the liquid polymers obtained were evaluated for potential use as high temperature base stock fluids. The relationship of physical properties to structural features of the polymeric oils was established.

WADC TR 54-613 Part III.
ORGANOSILANES AND RELATED COMPOUNDS AS HIGH-TEMPERATURE LUBRICANTS. Part III: Mixed Symmetrical Tetraalkylsilanes. Dr. Harold Rosenberg, Dr. Christ Tamborski, James D. Groves, 1/Lt. September 1957. ASTIA Document No. AD 131066. PB 131424.

One class of mixed symmetrical tetraalkylsilanes was investigated to determine their applicability as base materials for Air Force high-temperature hydraulic fluids and lubricants. A series of di-n-dodecyldialkylsilanes was prepared and various physical properties of the fluids obtained were correlated with molecular structure. These materials were found to be thermally stable at elevated temperatures and liquid over a wide temperature range. In addition to the tetraalkyl compounds, one aralkyl derivative, di-n-dodecyldiphenylsilane, was also synthesized. On the basis of preliminary property data, this fluid and at least several mixed symmetrical tetraalkylsilanes offer considerable promise as base stocks for 700°F lubricant and hydraulic fluid applications in new advanced air weapon systems.

Univ. of Illinois. WADC TR 57-391.
POLYMERIZATION THROUGH COORDINATION. John C. Bailar, Jr., William C. Drinkard, Jr., Malcolm L. Judd. AF 33(616)-3209. September 1957. ASTIA Document No. AD 131100. PB 131517.

The purpose of this work is to study the preparation and properties of metal-containing polymers in which the metal ion is held in the polymer through coordinate bonds. It is expected that such polymers will have exceptional stability toward heat and chemical reagents.

The background for the formation of such polymers is presented and the principles involved in the preparation of plastic materials are discussed. Pertinent literature references are cited.

Chelate polymers containing bis-β-diketones, bis-β-hydroxyquinolines, β-hydroxyethylpolyamines, and bis-α-amino acids have been investigated, as well as polymeric phthalocyanines. The most promising results have been obtained with the polymeric phthalocyanines, which have molecular weights up to about 30,000.

Aerochem Research Laboratories, Inc.
WADC TR 57-189.
REACTIONS IN ELECTRODELESS DISCHARGES BETWEEN VOLATILE HALIDES AND ORGANIC COMPOUNDS. Costa C. Akerlof. AF 33(616)-3225. September 1957. ASTIA Document No. AD 131095. PB 131479.

The purpose of this program was to study the formation of new types of thermally stable fluids and solids by means of high voltage electrodeless discharges through mixtures of volatile halides, particularly of silicon, and organic compounds. The effect of temperature, pressure, flow rate, current, and voltage, on the product yield and discharge behavior was investigated for typical reactant combinations.

The produce properties were studied for forty different reactant systems and twenty samples were submitted to the Materials Laboratory, Wright Air Development Center, for more detailed evaluation. In addition, a 600 gram sample boiling above 400°C, from the reaction of methane with dimethyldichlorosilane in the electrodeless discharge, was submitted for evaluation. Fractionation of similar samples has given cuts boiling over 200°C without visible decomposition - these same fractions were liquid at -15°C. The reaction between methane and methylchlorosilanes produces substances with high boiling points which have excellent thermal stability, combined with low freezing points and relatively low viscosity over large temperature ranges.

WADC TR 57-503.
THE CHEMICAL REACTIONS OF SILICON ISOCYANATES. Joseph F. O'Brien, Capt. October 1957. ASTIA Document No. AD 142101. PB 131621.

The purpose of this research was to examine the chemical properties of silicon isocyanates to see if these isocyanates can be used as monomers for the preparation of silicon-containing polyurethane resins. A prototype silicon isocyanate, trimethylsilylisocyanate, was reacted with amines, Grignard reagents, alcohols and glycols. It was found that amines, Grignard reagents and alcohols cleave the isocyanate group from the silicon atom to form the corresponding alkyl urea, alkyl amide and alkyl biuret respectively. The glycols do not cleave the isocyanate group from the silicon, but add to trimethylsilylisocyanate to form the corresponding bis (trimethylsilyl) carbamate.

American Potash & Chemical Corporation
WADC TR 57-126 Part I.
PHOSPHINOBORINE POLYMERS. Ross I. Wagner, Earl M. Evleth, Levern D. Freeman, Robert D. Stewart, Ernest Levens, Leland W. Marple. AF 33(616)-3506. October 1957. ASTIA Document No. AD 142028.

One of the continuing objectives of this project is the accumulation of fundamental data relating the structure of phosphinoborines to their chemical and physical properties. To this end, ten new phosphinoborines have been synthesized and several of these, as well as some of the previously known derivatives, have been screened for oxidative, hydrolytic, and thermal stability. The synthesis of phosphine and borine intermediates by known methods has produced four new alkyl phosphines. Several approaches to the preparation of aryl phosphines and diphosphines have been attempted without success.

Battelle Memorial Institute. WADC TR 57-110.
POLYMERIZATION STUDIES ON MONOMERS AND EVALUATION OF DERIVATIVE POLYMERS. Faber B. Jones, Cherie A. Lichtenwalter, Palmer B. Stickney, Randall G. Heiligmann. AF 33(616)-3313. October 1957. ASTIA Document No. AD 142050. PB 131594.

This report describes research to determine the polymerization characteristics of a group of experimental fluorine-containing monomers and a preliminary evaluation of the potential of the resulting polymeric products as high-temperature and solvent-resistant rubbers and plastics.

The homopolymerization of a group of six fluorinated olefin oxides in mass and solution using numerous different polymerization catalysts yielded only low-molecular-weight liquid polymeric products.

Copolymerization of several fluorine-containing propenyl ketones with dienes yielded some promising elastomers. Copolymerization of some fluorinated pentadienes, butenes and pentenes was effected to yield resinous solids; homopolymerization of these monomers was unsuccessful. Some fluorine-containing cyclic olefins were homopolymerized and copolymerized.

WADC TR 57-502.
SILICON-OXYGEN-TIN POLYMERS. Joseph F. O'Brien, Capt. October 1957. ASTIA Document No. AD 142100. PB 131620.

The purpose of this work was to develop a method for preparing new types of polymeric materials having a silicon-oxygen-tin-oxygen backbone. It was found that polymers of this structure could be prepared by (1) the reaction of dialkyltin diacetates with dialkyldiethoxysilanes, and (2) the reaction of dialkyltin dialkoxides with dialkyldiacetoxysilanes. Both of these reactions are condensation reactions in which a polymer is formed upon elimination of a volatile acetate. In contrast to this condensation reaction, both dialkyltin diacetates and dialkyltin dialkoxides undergo an exchange reaction with dialkyldichlorosilanes. Dialkyltin dichlorides do not react with either dialkyldiacetoxysilanes or dialkyldialkoxysilanes.

Univ. of Colorado. WADC TR 56-590 Part I.
PHOTOCHEMICAL SYNTHESIS OF ORGANIC FLUORINE COMPOUNDS. Joseph D. Park, John R. Lacher. AF 33(616)-3266. November 1957. ASTIA Document No. AD 142171. PB 151275.

The purpose of this research work has been to synthesize organic and organo-metallic fluorine compounds with the major objective of obtaining monomers suitable for polymerization into elastomers, plastics, fluids and related material of high thermal and chemical stability. In conjunction with these aims, this Laboratory has synthesized two halogenated pentadiene.

Several important four-membered fluorinated ring compounds have been synthesized and their cyclic ethers. To furnish adequate proof of structure and determine the position of unsaturation, the compounds have been halogenated.

In the work on alcohols both 1, 2-difluoro-2-chloroallyl alcohol and 1, 1-dufluoro-2-bromoallyl alcohol have been synthesized, the first reported synthesis of a fluorinated allyl alcohol obtainable directly from a Grignard reagent. However, synthesis of trifluoroallyl alcohol has

not been achieved.

Attempts which have been made to synthesize monohydric and polyhydric alcohols indicate that the proposed routes are feasible. Work in this area has not been completed.

A variety of methods have been used in attempts to prepare alpha, beta-unsaturated ketones (Grignard, free-radical, preparation of alkyl lithium derivatives, Friedel-Crafts-type reactions, and preparation of an unsaturated acid) without success. Several of the ketones derived from the addition of a Grignard reagent to Kel-F Acids have been prepared. As intermediates to the preparation of ketones, two alkynes have been made.

Pennsalt Chemicals Corporation
WADC TR 57-436.
PREPARATION OF POLYMERS FROM HALOGEN-CONTAINING OLEFINS AND DIENE MONOMERS. H. Iserson, M. Hauptschein, F. E. Lawler, AF 33(616)-3137. November 1957. ASTIA Document No. AD 142116. PB 131615.

Synthetic work on this project included the preparation of fluoro- and fluorohalo-olefins and dienes. Among the monomers prepared were 3-chloro-1,1,2-trifluorobutadiene, 1,2-dichloro-3,4,4-trifluorobutadiene, 1,1,2-trichloro-3,4,4-trifluorobutadiene, 3-bromo-1,1,2-trifluorobutadiene, 1,1,2,3-tetrafluorobutadiene, 1,4-dichlorotetrafluorobutadiene, 1,1-difluoroisobutylene, 1-trifluorovinyl-2-chloro-2,3,3-trifluorocyclobutane, 1-vinyl-2-chloro-1,2,3,3,4,4-hexafluorocyclobutane, perfluoro-1-vinylcyclobutene-1. Polymerizations have been tried with these and with other olefins. Some of the polymers which appear of interest were prepared from: perfluoropropene and trifluoroethyl vinyl ether; 1,1,2,3-tetrafluorobutadiene; 1,1,2,3-tetrafluorobutadiene and trifluoroethyl vinyl ether; 1,1,2,3-tetrafluorobutadiene and ethylene oxide; trifluoroethylene and trifluoroethyl vinyl ether; tetrafluoroethylene and ethylene oxide; tetrafluoroethylene and trifluoroethyl vinyl ether. Terpolymers have been prepared from trifluoroethyl vinyl ether, ethylene oxide and one of the following: perfluoropropene, vinylidene fluoride, 1,4-dichlorotetrafluorobutadiene, 1-chloro-2,2-difluoroethylene, tetrafluoroethylene.

The Pennsylvania State Univ.
WADC TR 56-203 Part II.
COORDINATION POLYMERS. W. Conrad Fernelius, Maurice Shamma, Norman R. Garofano, David E. Goldberg, Dean E. Martin, Forrest D. Thomas, III. AF 33(616)-2742. December 1957. ASTIA Document No. AD 142191. PB 131649.

Various approaches towards the development of polymers through coördination have been investigated. Several bi(-diketones) have been prepared; this work will now be followed by attempts at their polymerization.

Attempts to prepare a silicon monomer by the reaction of dimethyldichlorosilane with the sodium salt of acetylacetone were unsuccessful, the product formed being unstable. On the other hand, a series of monomeric bis(acetylacetoneate)dialkoxy titanium compounds were synthesized.

Investigations on the coordination ability of organic phosphorus radicals have shown that they do not coordinate easily. Dithiophosphates, on the other hand, coordinate well. A series of salts of dithiophosphates has been prepared, and efforts have been made to prepare an analogous bifunctional ligand. Polymeric materials have been produced from what probably is a mixed ester of a dialcohol and monoalcohol. The dithio compounds have low thermal stability.

Investigations of similar compounds with other donor atoms led to the conclusion that compounds with various combinations of oxygen, sulfur, amido nitrogen, and substituted amido nitrogen do not coordinate well, except for the dithio compounds already mentioned. Because of little hope of producing high temperature polymers, this phase of the problem has been abandoned.

Efforts towards the formation of coordination polymers from bis(o-hydroxyazo) compounds did not yield promising results because of the insolubility of the products formed.

A series of o-hydroxyketones was prepared by means of the Fries Rearrangement preliminary to the synthesis of bis(o-hydroxyketones).

Naugatuck Chemical. WADC TR 56-596 Part II. RESEARCH ON HIGH TEMPERATURE LAMINATING RESINS BASED ON MALEIMIDE AND ITS DERIVATIVES. Roger W. Amidon, Charles H. Alexander. AF 33(616)-3606. December 1957. ASTIA Document No. AD 142212.

The object of this contract was the preparation of laminating resins for service in the range of 600° to 700°F based primarily on maleimide or derivatives thereof.

Resins were evaluated by preparing from them a Fiberglas cloth laminate and exposing cured test pieces to elevated temperature. Quality was judged by retention of flexural strength of exposed samples.

A VIBRIN 135 type system which contained 25% maleimide showed superior modulus under unaged and aged conditions. Flexural strength values after 192 hours exposure at 500°F have been as high as 38,000 psi.

New alkyds have been prepared and evaluated with triallyl cyanurate monomer. None investigated showed outstanding properties when compared to VIBRIN 135.

Several N-substituted maleimide compounds were investigated with a VIBRIN 135 alkyd and triallyl cyanurate. The strength of aged pieces did not compare well with maleimide resins.

Triallyl isocyanurate, triallyloxybenzene maleimidomethyl acrylate and N-allyloxymethyl maleimide were synthesized and screened as copolymerizable monomeric materials.

Effort was made to gain some insight into the cause and minimization of interlaminar blistering. A low acid number polyester reduced blistering somewhat. Blistering was not experienced with non-nitrogeneous monomers such as diallyl o-phthalate or styrene.

WADC TR 57-657.
CONFERENCE ON HIGH TEMPERATURE POLYMER AND FLUID SYNTHESIS. Calvin H. Schmid, Capt. January 1958. ASTIA Document No. AD 142285. PB 161663. Order from OTS $3.50.

This report has been prepared to make available all the technical papers presented at the conference on "High Temperature Polymer and Fluid Syntheses."

The purpose of the conference was to review the progress on a comprehensive research program sponsored by the Polymer Section Organic Materials Branch, toward the development of new polymeric and fluid materials to meet the high performance requirements of aircraft of advanced design, and to familiarize representatives of the chemical industry with the materials problems confronting the Air Force.

Reaction Motors, Inc. WADC TR 57-45 Part II. HIGH-TEMPERATURE STABLE SEMIORGANIC FLUIDS. Part II. Organophosphorus Compounds. Daniel Grafstein, Murray S. Cohen. AF 33(616)-3417. January 1958. ASTIA Document No. AD 142290.

Several alternate synthetic procedures were

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developed for the preparation of substituted aryldialkylphosphine oxides. Among the tertiary phosphine oxides prepared were diethylphenylphosphine oxide, p-chlorophenyldiethylphosphine oxide, triethylphosphine oxide, ethylmethylphenylphosphine oxide and 2, 5-dimethylphenyldiethylphosphine oxide. Other intermediates of some synthetic importance were prepared including o-tolydichlorophosphine, diethylphosphonyl chloride and ethylphenylphosphonyl chloride.

The inherent thermal and oxidative stability of the tertiary phosphine oxides has been experimentally verified at 285°C with both diethylphenylphosphine oxide and p-chlorophenyldiethylphosphine oxide. Principal efforts were devoted to improving the liquid range by introducing chosen substituents in certain positions of the aromatic ring of an aryldialkylphosphine oxide.

WADC TR 57-62 Part I.
METAL-CYCLOPENTADIENYL HIGH TEMPERATURE LUBRICANTS AND RELATED MATERIALS. Part I: Synthesis and Properties of Alkyl, Alkaryl, and Metalated Derivatives of Ferrocene. Marvin Rausch, 1/Lt., Martin Vogel, 1/Lt., Dr. Harold Rosenberg. January 1958. ASTIA Document No. AD 142326. PB 131701.

The purpose of this program was to uncover new types of cyclopentadienyl metals, or "ferrocene-like" compounds with properties suitable for extreme temperature lubricant and hydraulic fluid applications. A series of long-chain alkylferrocenes and arylalkyl-ferrocenes were prepared by the Friedel-Craft acylation of ferrocene and subsequent reduction. Furthermore, the metalation of ferrocene by n-butyl-lithium and by mercuric acetate was studied in detail, and some reactions of the metalated derivatives were investigated.

WADC TR 58-3
SYNTHESIS AND CHARACTERIZATION OF NEW VINYL POLYMERS. Lester E. Coleman, Jr., 1/Lt., William S. Durrell, 1/Lt. January 1958. ASTIA Document No. AD 142296. PB 131669.

This report describes research to determine the polymerization characteristics of four classes of experimental monomers and a preliminary evaluation of the potential of the resulting polymeric products as thermally stable, solvent-resistant plastics and elastomers.

The perfluoroalkyl propenyl ketones were found to copolymerize with a variety of comonomers such as vinyl acetate, styrene and butadiene to give products ranging from powders to elastomers.

The 1-alkyl-1-hydroperfluoroalkyl acrylates copolymerized readily and formed tough rubbery homopolymers which exhibited good thermal stability and low swell in diester-type lubricants.

Trans-cinnamoyl ferrocene formed copolymers with a variety of comonomers but low conversions, copolymers or copolymers containing small amounts of cinnamoyl ferrocene were obtained.

Reactivity ratio determinations of two trifluoromethyl-substituted styrenes indicated that substitution of the trifluoromethyl group on styrene increased its polymerization reactivity.

Midwest Research Institute.
WADC TR 57-143 Part II.
DEVELOPMENT OF THERMALLY STABLE SILICON-CONTAINING RESINS. L. W. Breed, Fred Baiocchi, Calvin C. Bolze. AF 33(616)-3675. February 1958. ASTIA Document No. AD 151002. PB 131715.

Additional work aimed at preparing thermally stable silicon containing resins is described and some information on the incorporation of these resins into glass fabric laminates is included. Other methods for synthesizing monomers were investigated, particularly the use of tetrahydrofuran as a solvent in the Grignard syntheses. The results of a statistically designed experiment to study the variables effective in the formation of laminates from silicone resins and glass fabric are given, and the results of a similar experiment to study the effect of treating finished silicone resins with catalyst solutions are also described. Use of phenyldichlorosilyl-trichlorosilylbenzene in resin compositions is reported in considerable detail and the properties of laminates prepared from these resins are described. Laminates are also obtained from resins prepared from 1, 4-bis(methyldiethoxysilyl)benzene, 4, 4'-bis(methyldiethoxysilyl)phenylether, m-bis(trichlorosilyl)benzene, p-bis(trichlorosilyl)-benzene and a mixture of the last two isomers.

WADC TR 57-764
FLUORINE-CONTAINING CHELATE COMPOUNDS. Glenn R. Buell, 1/Lt. February 1958. ASTIA Document No. AD 150984. PB 131809.

The purpose of this work was to study the feasibility of preparing liquid chelate compounds. The thermal stability of certain chelate compounds led to their study as possible liquid base stock materials. Several fluorinated diketones were chelated with metal ions and their melting points studied to determine the effect of the fluorine on the physical properties of the molecule. Only one liquid was obtained and all compounds exhibited high vapor pressures.

Hooker Electrochemical Company
WADC TR 55-221 Part IV.
INVESTIGATION OF CONDENSATION-TYPE ELASTOMERS. George C. Schweiker, Burton S. Marks, Carl J. Verbanic, Blaine L. Lucas, Edward V. Gouinlock. AF 33(616)-2421. February 1958. ASTIA Document No. AD 151009. PB 131800.

The ultimate goal of the exploratory investigations described is the development of a rubber for special Air Force applications. Major requirements for such a material include good mechanical properties, high thermal stability (originally 350°F or higher), satisfactory performance at -65°F or lower, and resistance to aromatic fuels, synthetic ester-base oils, and hydraulic fluids.

To this end, fluorine-containing condensation polymers are being investigated in an effort to discover and develop suitable new elastomers.

This report describes the preparation, compounding, cross-linking, and properties of certain fluorine-containing polyester elastomers which appear to meet the goals outlined above. The report also describes the research performed on the synthesis of fluorine-containing difunctional starting materials, polyesters and nitrogen-substituted polyamides therefrom, and their characterization.

WADC TR 57-62 Part II.
METAL-CYCLOPENTADIENYL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS. Part II: A. Synthesis and Properties of Aryl, Benzyl, and Silyl Derivatives of Ferrocen. B. Preliminary Studies on the Effect of Tetrahydrofuran on the Lithiation of Ferrocene. Marvin Rausch, 1/Lt., Martin Vogel, 1/Lt., Dr. Harold Rosenberg, Dana Mayo, 1/Lt., Paul Shaw, 1/Lt. February 1958. ASTIA Document No. 150979. PB 131714.

The purpose of this program was to uncover new types of ferrocene-containing materials with properties suitable for extreme-temperature lubricant and hydraulic fluid applications. A number of new aryl- and

benzylferrocenes have been prepared. A valuable modification of the lithiation of ferrocene by n-butyllithium has been devised using ethyl ether-tetrahydrofuran as the solvent system. This procedure has been employed for the preparation of both carboxy- and 1,1-dicarboxy-ferrocene in good yield, and for the synthesis of long-chain alkylsilylferrocenes.

Univ. of Colorado. WADC TR 56-590 Part II. PHOTOCHEMICAL SYNTHESIS OF ORGANIC FLUORINE COMPOUNDS. Joseph D. Park, John R. Lacher. AF 33(616)-3266. March 1958. ASTIA Document No. AD 151014. PB 131803.

The purpose of this research work has been to synthesize organic and organometallic fluorine compounds with the major objective of obtaining monomers suitable for polymerization into elastomers, plastics, fluids and related material of high thermal and chemical stability. In conjunction with these aims, this Laboratory has synthesized a number of diols and cyclic fluorosilanes. At the present time, however, the structures assigned to several of the synthesized fluorosilanes have not been verified.

Further work in the preparations of cyclic diols involving the dimerization reaction of fluoroolefins with alkenes containing functional groups has produced intermediates with interesting fluorescent properties.

Initial investigation has been made of obtaining a monomeric unit with fluorine substitution on the aromatic nucleus.

Battelle Memorial Institute.
WADC TR 57-110 Part II.
POLYMERIZATION STUDIES ON MONOMERS AND EVALUATION OF DERIVATIVE POLYMERS. Faber B. Jones, R. A. Markle, Palmer B. Stickney, Randall G. Heiligmann. AF 33(616)-3313. March 1958. ASTIA Document No. AD 151011. PB 131778.

This report describes research to determine the polymerization characteristics of a group of experimental monomeric compounds and a preliminary evaluation of the potential of the resulting polymeric products as thermally stable and solvent-resistant rubbers and plastics.

The homopolymerization of a group of six fluorine-containing olefin oxides in mass and solution using numerous different polymerization catalysts yielded only low-molecular-weight liquid polymeric products.

Copolymerization of perfluoromethylpropenyl ketone with butadiene at 5°C in aqueous emulsion by free-radical initiation yielded solid rubbery polymers. Copolymerization of perfluoropropylpropenyl ketone with 1,1,2-trifluoro-3-chlorobutadiene; 1,1,2-trifluorobutadiene; and normal butadiene by free-radical initiation in aqueous emulsion also yielded solid polymers with varying degrees of elastomeric character. Copolymerization of perfluoropropenyl ketone with 1,1,2-trifluoro-3,4,4-trichlorobutadiene or 1,1,2-trifluoro-3,4-dichlorobutadiene to solid polymeric products could not be accomplished by free radical or ionic initiation. Perfluoroheptylpropenyl ketone was observed to copolymerize with 1,3-dienes under conditions similar to those used for perfluoromethyl- and perfluoropropylpropenyl ketone but appeared to be less reactive.

The copolymers of perfluoromethylpropenyl ketone with butadiene and perfluoropropylpropenyl ketone with butadiene and 1,1,2-trifluoro-3-chlorobutadiene were briefly evaluated as rubber vulcanizates and showed some promise in high-temperature and solvent resistance.

The three perfluoroalkylpropenyl ketones appeared to homopolymerize in low yield to low-molecular-weight products by BF_3 catalysis at low temperatures. Generally, good yields of solid polymeric products were obtained when these perfluoroalkylpropenyl ketones were copolymerized with styrene or 1,1,1-trifluoroethyl vinyl ether by BF_3 catalysis under similar conditions.

Homopolymerization of perfluoro-1,4-pentadiene; 1,1,3,3,5,5-hexafluoro-1,4-pentadiene; 1,1,2,3,4,5,5 octafluoro-4,5-dichloro-1-pentene; and 1-ethoxyperfluorocyclobutene-1 could not be accomplished by free-radical or ionic polymerization techniques. Copolymerization of three monomers with such comonomers as vinyl acetate; acrylonitrile; butadiene, styrene, 1,1,1-trifluoroethyl vinyl ether; 1,1-dihydroperfluorobutylacrylate; and N-n-butyl-N-1,1-dihydroperfluorobutylacrylamide yielded polymeric products ranging from oils to elastomeric and resinous solids.

Homopolymerization of 1,2-diethoxyperfluorocyclobutene-1, 1-allyloxyperfluorocyclobutene-1; and 1,1,2,3,3,4,5,5-octafluoro-2-chloro-4-pentenylmethyl ketone by free radical and ionic polymerization methods yielded liquid and solid polymeric products. Copolymerization of these monomers with styrene and N-n-butyl-N-1,1-dihydroperfluorobutylacrylamide yielded resinous solid polymeric products.

Attempts were made to prepare high-molecular-weight homopolyesters by polycondensation of isomers of pyridine dicarboxylic acid with ethylene glycol. The products were brittle, low-molecular-weight, highly colored solids.

Transesterification of the dimethyl esters of the pyridine dicarboxylic acids with ethylene glycol did not give products of significantly improved properties. Replacement of ethylene glycol with neopentyl glycol, however, gave products of less coloration and apparently greater thermal stability.

Reaction of the diacid chlorides of some of the pyridine dicarboxylic acid isomers with ethylene and neopentyl glycol gave essentially the same results as transesterification reactions of the dimethyl esters, except that the reactions were more rapid and the products had, in general, greater clarity.

Thermosetting copolyesters were prepared by copolymerizing the pyridine dicarboxylic acids, their dimethyl esters or diacid chlorides, and an unsaturated dicarboxylic acid with ethylene glycol or neopentyl glycol. The products, translucent or opaque, dark brown semi-solids or solids at room temperature, could be modified with triallyl cyanurate and cured to give dark brown, opaque, hard, nontacky solids.

Thermosetting polyesters were obtained by reacting pyridine diacrylic acid or its dimethyl ester with ethylene or neopentylglycol. The uncured products were clear, dark brown, low-melting solids retaining double bond activity.

A polycarbonate of moderate molecular weight was prepared by reacting 4,4'-dioxydiphenyl-2,2-propane (Bisphenol A) with phosgene or with diphenyl carbonate. A molded film approximately 0.010 inch thick prepared from the product obtained by fusion with diphenyl carbonate was fairly stiff but could be bent double without cracking.

A low-molecular-weight water-soluble resin was prepared by the reaction of bis(hydroxymethane) phosphinic acid with dimethyl terephthalate.

Peninsular Chemresearch, Inc.
WADC TR 55-220 Pt IV.
DEVELOPMENT OF FLUORO-SILICONE ELASTOMERS. Byron H. Wise. AF 33(616)-3238. April 1958. ASTIA Document No. AD 151180.

Some promising fluoroalkylsilane monomers and polymers have been prepared for use as high temperature and aircraft fuel resistant elastomeric materials.

In addition to the halosilanes previously synthesized, the following new silanes were prepared:

Bis (trifluoropropyl)dichlorosilane

(Chlorohexafluorocyclobutylethyl)-methyldichlorosilane

(2-Trifluoromethyltrifluoropropyl)-methyldichlorosilane

Polymerization of the cyclic polymers of (pentafluorobutyl)-methylsiloxane and (heptafluoropentyl) methylsiloxane was attempted, but resulted in oils of lower viscosity than desired. Polymerization of the cyclic polymers of (trifluoropropyl)methylsiloxane yielded high viscosity oils and in one case an elastic gum was formed.

The University of Illinois. WADC TR 58-51.
HIGH POLYMERIC MATERIALS. Carl S. Marvel, Ludwig F. Audrieth and John C. Bailar, Jr. AF 33(616)-3772. April 1958. ASTIA Document No. 151177.

The purpose of the work reported here is to provide basic information concerning new types of polymeric materials which will be more resistant to high temperatures than are known polymeric materials.

The types of materials which have been investigated are: polymers which have the possibility of aromatization; copolymers of aromatic nuclei such as anthracene; polymers which contain metal coordination centers such as polyphthalocyanines, poly-Schiff base chelates, polybasic beryllium carboxylates; new derivatives of phosphonitrilic hydrosulfides; new polymers derived from benzene phosphonic amides; polymers containing coordination centers derived from bis-phenolic compounds.

Many new polymeric materials are described. One derived from dimethyl α, α-dimethylenapimelate shows some possibilities as a high temperature resistant replacement of polymethyl methacrylate.

American Potash & Chemical Corporation.
WADC TR 57-126 Pt II.
PHOSPHINOBORINE POLYMERS. Ross I. Wagner, Frederick F. Caserio, Jr., Earl M. Evleth, Levern D. Freeman, Ernest Levens and Kendrick R. Eilar. AF 33 (616)-3506. April 1958. ASTIA Document No. 151182.

One of the continuing objectives of this project is the accumulation of fundamental data relating the structures of phosphinoborines to their chemical and physical properties. To this end six new phosphinoborines have been synthesized and several of these, as well as some of the previously known derivatives, have been screened for oxidative, hydrolytic, and thermal stability. The synthesis of phosphine intermediates by known methods has produced one new alkylphosphine and six new organophosphine derivatives. The successful preparation of a primary aromatic diphosphine has been achieved.

University of Southern California.
WADC TR 56-82 Pt III.
PRINCIPLES OF INORGANIC POLYMERIZATION. Anton B. Burg, Gottfried Brendel, Aimery P. Caron, Peter J. Slota, Jr., Gordon L. Juvinall, Walter Mahler and Kurt Modritzer. AF 33(616)-2743. April 1958. ASTIA Document No. AD 151175.

The work of the past year has been devoted largely to the search for new substances which might seem promising as components of inorganic polymer systems, and to a study of their chemical bonding properties. The aminophosphine $(CH_3)_2NP(CH_3)_2$ has been converted to $(CH_3)_2PCl$ and to the new diphosphine $P_2(CH_3)_4$. The latter was used to make a $(CH_3)_2PBH_2$-chain polymer and a somewhat improved thermally stable resin. A new interpretation of such resins extends earlier ideas. The phosphino-aluminum system has been explored, indicating new-type compounds of some interest. A considerable further study of fluorocarbon-phosphorus compounds included a demonstration of CF_3P higher polymers, the $(CF_3PO_2)_n$ system, and $(CF_3P)_4$-nickel-carbonyl polymers. The synthesis of $(CF_3)_2PBH_2$ trimer and tetramer has been studied thoroughly, with recognition of interesting by-products. The $(CH_3)_3N$-$(NPCl_2)_3$ reaction has been shown to split the amine to form $(CH_3)_2N$-P derivatives. An acetylide derivative of $(NPCl_2)_3$ formed a higher polymer. Work with $(NPF_2)_3$ has begun. The interesting unstable product of the CF_3SCl-B_2H_6 reaction may prove to be CF_3BF_2.

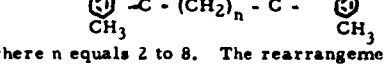
The Pennsylvania State University.
WADC TR 56-203 Part III.
COORDINATION POLYMERS. W. Conard Fernelius, Maurice Shamma, L. A. Davis, D. E. Goldberg, B. B. Martin, D. F. Martin and F. D. Thomas, II. AF 33(616)-2742. May 1958. ASTIA Document No. AD 155525.

As part of a study of the influence of structural variation on the properties of bis (β-diketones) and their metal derivatives, compounds of the following types have been prepared: (1) $RCOCH_2CO$-Y-$COCH_2COR$, (2) (R'CO)(RCO)-CH_2CHR'' and (3) (RCO)(R'CO)CH-Y-CH(COR')(COR). The methods of syntheses of these compounds have been studied. Compounds of types (2) and (3) offer the best promise of obtaining wide structural variation. Efforts to obtain bis-(β-diketones) containing fluorine atoms have failed, although some of the methods used have been successful when applied to other systems. The infrared and ultraviolet absorption spectra of various bis (β-diketones) have been obtained.

Various modifications of the polymerization techniques are being studied in an attempt to obtain high-molecular-weight polymers from the bis (β-diketones). Methods for fractionally separating polymers in order to get some idea of size distribution promise to be successful with the bis(β-diketone)-metal polymers.

The Fries rearrangement of di-p-tolyl esters has been studied as a means of preparing bis(o-hydroxyketones). As a result, di-p-tolyl esters have been prepared from succinic acid through and including sebacic acid, diglycolic acid, and phthalic, isophthalic and terephthalic acids.

The esters from succinic acid through and including sebacic acid were successfully rearranged (Fries rearrangement) to their corresponding bis-(o-hydroxyketone):



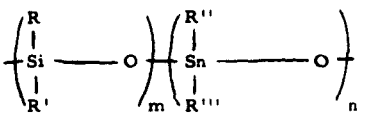
where n equals 2 to 8. The rearrangement was unsuccessful for di-p-tolyl-diglycolate. The rearrangement of the di-p-tolyl esters of isophthalic and terephthalic acids led to the formation of their corresponding bis(o-hydroxyketones). Attempted rearrangement of di-p-tolyl phthalate resulted in the formation of 1-hydroxy-5-methylanthraquinone. The infrared absorption spectra of the various bis(o-hydroxyketones) have been obtained.

In an attempt to prepare 5-acyl compounds of 8-hydroxyquinoline by the Fries rearrangement of 8-hydroxyquinoline esters, the following esters were prepared: 8-hydroxyquinolylacetate, propanoate, butanoate, benzoate, and adipate. The Fries rearrangement of the 8-hydroxyquinoline esters was unsuccessful.

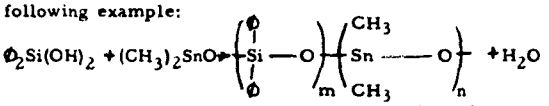
Preparation of 5-acyl derivatives of 8-hydroxyquinoline by means of the Friedel-Crafts reaction (8-hydroxyquinoline, acid chloride, aluminum chloride) appears quite promising. 5-Acetyl-8-hydroxyquinoline has been prepared in 27% yield. 5-Benzoyl-8-hydroxyquinoline and 5-propanoyl-8-hydroxyquinoline have also been prepared.

Ethyl Corporation. WADC TR 58-44 Pt I. SYNTHESIS OF TIN-CONTAINING POLYMERS. Paul E. Koenig and James H. Hutchinson. AF 33(616)-3848. May 1958. ASTIA Document No. AD 151197.

This investigation has been largely directed toward the preparation of stannosiloxane polymers, whose structure may be represented as followed:



These compounds have been prepared by various condensation polymerizations, including a novel reaction between silanediols and organotin oxides, typified by the following example:



A second excellent synthetic method involves cohydrolysis of mixtures of dichlorosilanes and organotin dihalides; this method is well-suited to the preparation of polymers containing alkyl groups bound to silicon.

An analogous cohydrolysis reaction employing different organotin dihalides but no silicon compounds leads to formation of copolymeric stannoxanes; these are interesting in that they represent the first thermoplastic metal-oxide type polymers other than the silicones.

The products obtained are viscous fluids or transparent glasses; their hydrolytic and thermal stability is good. Difficulty has been experienced in attempting to produce high polymers over the composition range examined; the molecular weights thus far observed have been of the order of 1,-5,000, and some properties of the products are therefore correspondingly deficient. Work is in progress to obtain higher molecular weight products, which may well be exceptionally stable and highly useful materials.

WADC TR 58-160. THE AIR FORCE INORGANIC POLYMER PROGRAM. Robert L. Rau, 1/Lt. June 1958. ASTIA Document No. AD 155675.

The purpose of this report is to critically review and evaluate the Air Force inorganic polymer program carried on by various organizations under contract to the Materials Laboratory, Wright Air Development Center, Air Research and Development Command.

Because of the thermal limitation of "organic polymers," efforts have been directed toward the preparation of polymeric materials having varying degrees of inorganic character. Various boron, nitrogen and phosphorus containing polymers have been investigated in addition to coordination polymers and those incorporating the silicon-oxygen-metal bond.

The results obtained have been partially successful. Boron-phosphorus polymers having stabilities as high as 450°C and coordination polymers stable in excess of 360°C in air have been prepared by contractors, although they did not have useful properties due to low molecular weights. Continued success is strongly dependent upon the development of methods for obtaining high molecular weights, as well as the development of analytical procedures for determining molecular weights for these types of polymers.

WADC TR 58-219. SYNTHESIS OF DIFUNCTIONAL FERROCENE DERIVATIVES. Paul D. Shaw, 1/Lt, Dana Mayo, 1/Lt, Marvin Rausch, 1/Lt, William S. Durrell, Myron Black, 2/Lt and Dr. A. M. Lovelace. June 1958. ASTIA Document No. AD 155658.

This program was undertaken to investigate the synthesis of difunctional ferrocene compounds for use as monomers. A number of ferrocene diols were prepared. Several methods for the synthesis of the ferrocene carboxylic acids were investigated. An improved synthesis of ferrocene dicarboxylic acid has been achieved using n-butyllithium in a solvent system consisting of diethyl ether and tetrahydrofuran.

Ford Motor Company. WADC TR 57-321 Pt II. SYNTHESIS OF POLYXYLYLYLDIMETHYLSILANE AND POLYXYLYLYLTETRAMETHYL SILOXANE. Glenn R. Wilson, Gretchen M. Hutzel, and Richard C. Hetu. AF 33(600)-32448. June 1958. ASTIA Document No. AD 155663.

The purpose of this project is the synthesis of a series of poly-p-xylylyldimethylsilanes and poly-p-xylylyltetramethylsiloxanes for evaluation as high temperature lubricants and/or hydraulic fluids.

The proposed program of syntheses was outlined and discussed previously in WADC TR 57-321 and was directed primarily at working out the details for synthesizing the basic building unit of poly-p-xylylyldimethylsilane before proceeding to the ultimate synthesis of the polymers. At that time we had obtained a very small quantity of a liquid material, whose elemental analysis agreed exactly with that calculated for bis(trimethylsilyl)-p-xylylene, from the coupling reaction of p-dibromobenzene and chloromethyltrimethylsilane with metallic sodium. Subsequent efforts to prepare larger quantities of this material by scaling up the reaction were unsuccessful and complex mixtures of difficult-to-separate materials were obtained.

Because of the multiplicity of functional groups involved in this particular coupling reaction, other routes of synthesis were investigated in an attempt to reduce the numbers of permutations and combinations of competing reactions. The results of these investigations indicate that an unequivocal route is yet to be found for preparing the desired compound.

As final efforts we are investigating a Friedel-Crafts type reaction described in the recent Russian literature and will also attempt to prepare the meta isomer of bis(trimethylsilyl) xylylene for which there is a much better chance of success.

Wyandotte Chemicals Corporation. University of Illinois. WADC TR 58-187 Part I. SYNTHESIS AND EVALUATION OF HIGH TEMPERATURE STABLE AND NUCLEAR RADIATION STABLE METAL-CYCLOPENTADIENYL FLUIDS. Robert L. Schaaf and Kenneth L. Rinehart, Jr. AF 33(616)-5053. July 1958. ASTIA Document No. AD 155737.

In a search for fluids suitable for use as Air Force high-temperature hydraulic fluids and lubricants, four 1, 1'-bis(3-substituted-1, 1, 3, 3-tetramethyldisiloxanyl)-ferrocenes were synthesized from cyclopentadiene via cyclopentadienyldimethylchlorosilane and a series of 1-cyclopentadienyl-3-substituted-1, 1, 3, 3-tetramethyldisiloxanes. The 3-phenyl compound possessed the desired thermal stability at 366°C (690°F). Other previously unreported ferrocenes and intermediates synthesized included 1, 3-bis(cyclopentadienyl)-1, 1, 3, 3-tetramethyldisiloxane, 1, 3-)1, 1'-ferrocenylene)-1, 1, 3, 3-tetramethyldisiloxane, 1-cyclopentadienyl-3-chloro-1, 1, 3, 3-tetramethyldisiloxane and 1-cyclopentadienyl-5-phenyl-1, 1, 3, 3, 5, 5-hexamethyldisiloxane.

Experiments designed to elucidate the structures of products from the condensation of ferrocene with formaldehyde and benzaldehyde were performed, and a

number of intermediates required for a study of the formation of bridged ferrocenes were synthesized.

Additional areas of investigation which were studied but largely without success included the syntheses of alkoxycyclopentadienes, the preparation of 3-methyl-1-ethylcyclopentadiene, and the identification of liquid ferrocenes by means of adducts of ferrocenes and tetranitrofluorenone.

Polytechnic Institute of Brooklyn. WADC TR 58-382 Part I.

SYNTHESIS AND EVALUATION OF NEW POLYMERS PREPARED BY STEREOSPECIFIC CATALYSIS. Charles C. Overberger. AF 33(601)-5253. November 1958. ASTIA Document No. AD 204215.

Pure cis or trans 3 and 4-methylvinylcyclohexane have been prepared. The polymer obtained from the triisobutyl aluminum-titanium tetrachloride catalyzed polymerization of 3-methylvinylcyclohexane was crystalline. Vinylcyclopentane has been synthesized and polymerized with a triisobutyl aluminum-titanium tetrachloride catalyst; and polyvinylcyclopentane was found to be crystalline. Methylenecyclohexane and methylenecyclopentane were made, and these olefins failed to polymerize with a Ziegler-Natta type catalyst. Methylenecyclobutane was synthesized and polymerized, apparently with some ring opening with a Ziegler-Natta type catalyst. 3, 3, 3, -Trifluoropropene was prepared and failed to polymerize with a triisobutyl aluminum-titanium tetrachloride catalyst. The preparation of 4, 4, 4-trifluorobutene-1 has been carried out. 1, 4-Pentadiene was obtained from the pyrolysis of 1, 5-pentamethylene diacetate.

A kinetic study of the polymerization of vinylcyclohexane with a triisobutyl aluminum-titanium trichloride catalyst showed that both oxygen and water affect the rate of polymerization. Unit cell dimensions were determined for crystalline polyvinylcyclohexane and crystalline polyvinylcyclopentane.

Denver Research Institute. WADC TR 58-377.

RELATIONS BETWEEN STRUCTURE AND RADIATION STABILITY OF VARIOUS ALKYL AROMATIC FLUIDS. Josef J. E. Schmidt, Don N. Gray, Francis S. Bonomo and James G. Pomonis. January 1959. ASTIA Document No. AD 207526. AF 33(616)-5317.

The effects of gamma radiation from a Cobalt-60 radiation source on liquid alkyl derivatives of biphenyl and terphenyl compounds have been investigated and the type and yield of gaseous decomposition products determined. The counter-current distribution method was found to be the most feasible means of separation of liquid irradiation products.

Alkyl substituted diphenyl and polyphenyl derivatives show a relatively high radiation stability. The influence of the type of alkyl substituent and its position on the molecule indicates an energy transfer mechanism. This mechanism is to be investigated in more detail in the remaining research period.

Midwest Research Institute. WADC TR 57-143 Part III.

DEVELOPMENT OF THERMALLY STABLE SILICON CONTAINING RESINS. L. W. Breed, William J. Haggerty, Jr. and Fred Baiocchi. AF 33(616)-3675. April 1959. ASTIA Document No. AD 212268.

The syntheses and evaluation of new monomers for incorporation into silicon containing resins are reported. The new monomers, which are mostly based on the arylenedisilane structure, are selected for use in establishing polymeric units capable of imparting good high temperature properties to silicone resin systems.

The new monomers include compounds which contain two or more silicon atoms separated by various space groups. Described in the report are the following compounds: $Me(EtO)_2SiYSi(EtO)_2Me$ where Y is $-C_6H_4C_6H_4-$, $-C_6H_4CH_2-$, and $-(CH_2)_6-$. Other monomers are prepared which have different substituents on the organic group not used as a spacer. These monomers include the following: $YC_6H_4(EtO)_2SiC_6H_4Si(EtO)_2C_6H_4Y$ where Y is H-, Cl-, MeO-, and Me_2N-. The method of synthesis and properties of each new monomer is described in detail.

Silicone-glass fabric laminates are described which contain disilylbenzene monomers and have improved flexural strengths at temperatures above 500°F. The results of screening new resins and investigating the composition and fabrication parameters in these new resin systems are included. These results are obtained by a procedure which requires relatively small amounts of experimental resin.

Hooker Chemical Corporation. WADC TR 55-221 Part 5.

FLUORINE-CONTAINING CONDENSATION POLYMERS AND RESINS. Carl J. Verbanic, David Knutson, John E. Wier, Edward V. Gouinlock and C. Thomas Bean. AF 33 (616)-5548. April 1959. ASTIA Document No. AD 212902.

The ultimate goal of the investigations described is the determination of the effect of fluorine and fluorine content on the thermal and oxidative stability of unsaturated polyester laminating resins. An additional function is to aid in the investigations of the perfluorinated amadine polymers currently of interest to Wright Air Development Center.

Polyesters and polyester resin laminates have been prepared using fluorinated glycols as a component of the polyester. The laminates have been aged at elevated temperatures and physical properties measured before and after aging. Comparison of these aging results with polyesters containing hydrocarbon glycol components of the same carbon chain length indicate a noticeable improvement in retention of physical properties of the fluorinated materials over the hydrocarbon analogues.

The report also describes the research performed on the preparation of certain fluorine-containing compounds for use as starting materials for the preparation of polyesters and cross-linking monomers.

A new and improved synthesis of perfluoroglutaronitrile is described and efforts to improve the yields of the dehydration of perfluoroglutaramide are given.

Research on the synthesis of certain novel or unusual fluoride-containing intermediates considered desirable for polymerization in this project was performed at Purdue University under sub-contract to Hooker Chemical Corporation. Details of the syntheses performed at Purdue during the contract year comprise Appendix I of this report.

University of Illinois. WADC TR 58-51, Part II.

HIGH POLYMERIC MATERIALS. Carl S. Marvel, Ludwig F. Audrieth, John C. Bailar, Jr. AF 33(616)-5486. May 1959.

C. S. MARVEL SECTION:

The work described herein is in part a continuation of that reported in WADC TR 58-51.

An intractable p-polyphenyl mixed with carbon black has been obtained but due to complete insolubility its molecular weight has not been established.

Poly-06 α'-dimethylenepimelonitrile which contains a cyclohexane recurring unit has been characterized and shown to have a better heat stability than does polyacrylonitrile. An interesting polymeric cyclic silicon compound has been prepared from diallyldimethylsilane.

Polymers containing pyridine rings along with vinyl ketone monomer units have been prepared but show disappointing heat stabilities.

It has not been possible to get polyphthalocyanines of high molecular weight. The products appear to be di- or trimeric rather than polymeric.

Other work which is in progress but not completed is recorded.

L. F. AUDRIETH SECTION:

Reaction of benzene phosphonic diamide with aqueous formaldehyde gives small yields of a dimethylol derivative. Side reactions necessitated a study of the hydrolysis of the diamide, both in water under varying pH conditions and in 100% acetic acid. Condensation reactions of benzene phosphonic amide with polyamines lead to elimination of ammonia and formation of low melting, glassy products which are presumably cyclic derivatives.

The hydrochlorides, phosphates and benzene phosphonates of hexamethylene diamine, diamine, diethylene triamine and ethylene diamine have been prepared and characterized.

The di- and hexa-substituted N-n-dibutyl phosphonitrilamides are less stable thermally than the hexa-N-phenyl derivative of triphosphonitrile.

Sodium-liquid ammonia is used to decompose chlorine-containing phosphonitrilic derivatives as the first step in the quantitative determination of chlorine in such compounds.

The trimeric and tetrameric phosphonitrilic isothiocyanates have been prepared by methesis in acetone solution. These substances polymerize to rubber-like products when heated above 150°, and undergo reactions characteristic of the -NCS group with ammonia, amines, alcohols, mercaptans and hydrazines.

J. C. BAILAR, JR. SECTION:

Several bis-aminophenols and bis-8-hydroxyquinolines have been prepared, and have been made to react with the divalent, tetracoordinate ions of copper and zinc. The substances so formed are undoubtedly polymeric, but are of low molecular weight. Attempts to form substances of higher molecular weight are planned.

Attempts have been made to prepare bis-catechol derivatives, and 2,2', 6,6'-tetrahydroxybiphenyl, but these have not yet been obtained in good yield. The work is continuing; when the materials are obtained, they will be made to react with silicon tetra-chloride. A short chain polymer has been prepared from methylenedisalicylic acid and silicon tetrachloride.

Studies of new methods of polymerization are being undertaken in the hope of learning more about the polymerization process.

Ford Motor Company. WADC TR 57-231 Part III. SYNTHESES OF POLYXYLYLYLDIMETHYLSILANE AND POLYXYLYLYLTETRAMETHYL SILOXANE. Glenn R. Wilson, Gretchen M. Hutzel, Richard C. Hetu and Arthur G. Smith. AF 33(600)-32448. May 1959. ASTIA Document No. AD 213605.

The purpose of this project is the synthesis of a series of poly-p-xylylyldimethylsilanes and poly-p-xylylyltetramethylsiloxanes for evaluation as high temperature lubricants and/or hydraulic fluids.

At the time of the last annual report (WADC TR 57-321, Part II), and after numerous unsuccessful attempts to prepare the basic building unit, bis (trimethylsilyl)-p-xylylene, of these proposed polymers, a successful procedure was developed while investigating the preparation of the meta isomer. As a result we were able to prepare the o-, m-, and p-isomers of bis (trimethylsilyl) xylylene and by the same technique a difunctional intermediate, bis (dimethylchlorosilyl)-p-xylylene that could be used for preparing the polysilane or polysiloxane.

Synthesis of the polysiloxane was selected as the first choice due to the fact that a homogeneous-type polymer was anticipated, whereas, a heteropolymeric chain was anticipated in preparing the polysilane due to the undesirable coupling of xylylene dihalides to bibenzyl moieties in the reaction.

Several small-scale polymerizations of bis (dimethylchlorosilyl)-p-xylylene, in the presence of variable quantities of trimethylchlorosilane, with water were carried out and the polymers obtained were of relatively low molecular weight, ca. 700-1100. Results of copolymerization with p-bromophenyldimethylchlorosilane, to provide analyzable end groups, indicated that the polymer contained a high percentage of cyclic polymer.

Since the project is being terminated, a series of runs for preparing bis (dimethylchlorosilyl)-p-xylylene were made and the yields combined to provide a sizable quantity. Some 600 grams of the intermediate has been prepared and polymerized with trimethylchlorosilane to provide a sufficient quantity of the polysiloxane for evaluation.

WADC TR 58-589.
THE SYNTHESIS OF SOME FLUORINE CONTAINING MONOMERS. William Durrell. May 1959. ASTIA Document No. AD 214766.

Various methods of synthesis of fluorine-containing olefins with different functional groups were studied. The methods involved additions to olefinic double bonds, both free radical- and base-catalyzed.

Attempts were made to prepare vinyl ethers, acrylonitrile derivatives, unsaturated olefin oxides, and vinyl ketones.

A new, polymerizable unconjugated pentadiene, $CF_2=Ch-CF_2-Ch=CF_2$ were prepared.

Several new free radical additional reactions were carried out and a novel mode dimerization of α,β -unsaturated fluoro-ketones discovered.

Midwest Research Institute. WADC TR 58-84, Part II.
DEVELOPMENT OF HIGH-TEMPERATURE STABLE LIQUID UREAS AND AMIDES. Cecil C. Chappelow, Jr. and Robert N. Clark. AF 33(616)-5129. June 1959. ASTIA Document No. AD 220082.

The program for the development of thermally stable liquid ureas was continued. This program consisted of the synthesis and evaluation of several classes of tetra-substituted ureas.

During the synthesis program, a total of 14 tetra-substituted ureas were prepared, 13 of which are new compositions of matter not previously reported in the literature. These compounds are representative of the following types of tetra-substituted ureas: aryltriphenylureas, dialkyldiarykyreasm alkyltriarylureas, symmetrical tetraarylureas, unsymmetrical methyltriarylureas and unsymmetrical tetraarylureas.

During the evaluation program, primary emphasis this year was placed upon the liquid properties of the ureas. By the proper use of n-alkyl and aryl groups, liquid ureas were prepared. However, due to the oxidative instability of n-alkyl groups, the research program was limited to the use of methyl and aryl groups. Attempts to obtain liquid ureas using these groups only were unsuccessful.

On the basis of two years' work, certain types of tetra-substituted ureas show promise as base stock materials for high temperature fluid and lubricant applications.

WADC TR 59-136.
EVALUATION OF EXPERIMENTAL POLYMERS. Charles D. Doyle. June 1959. ASTIA Document No. AD 216453.

Thermal stability is discussed in terms of limiting temperatures on the basis of thermodynamic, rigorous kinetic, non-rigorous kinetic, functional-environmental and empirical considerations. The desirability of rapid empirical methods in general prospecting is discussed, and the term "procedural decomposition temperature" (pdt) is used to emphasize the powerful effect of procedural details on the measured values in empirical tests. Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) are discussed in detail as rapid empirical methods for measuring pdt's. Several experimental materials are tentatively compared on the basis of TGA in inert atmosphere.

Ethyl Corporation. WADC TR 58-44, Part II.
ORGANOMETALLIC POLYMERS. Paul E. Koenig and Ronald D. Crain. AF 33(616)-3848. June 1959.

Continued investigation of stannosiloxane polymers has led to the conclusion that relatively large organic substituents must replace the methyl and phenyl groups thus far studied, if high thermal stability is to be obtained. The polyorganostanosiloxanes have been found to undergo extensive decomposition and disproportionation on prolonged heating above 300°C.

Reactions of organotin disodium derivatives with organic dihalides to prepare linear polymers containing tin-carbon chains are described.

A very convenient method is reported for the preparation of tin-sulfur polymers (stanthianes) by reaction of sulfur with divalent organotin compounds.

Explorations have been initiated to prepare aluminum oxide polymers. The thermal stability of the aluminum-oxygen bond should lead to polymers of improved heat and oxidative stability. The extreme reactivity of the aluminum-alkyl bond has been utilized in an effort to obtain high molecular weights.

Four methods of approach have been explored:

1. Trialkylaluminum compounds were reacted with dihydroxy monomers to produce products which are insoluble, infusible powders with good thermal stabilities.
2. Dialkylaluminum alkoxides were reacted with dihydroxy compounds to form similar products. In this instance, however, it was ascertained through molecular weight studies that the alkoxides were stable trimers, hence multifunctional, and the polymers obtained were therefore highly cross-linked.
3. Various "difunctional" aluminum chelates were prepared. The polymers obtained were infusible and insoluble powders except in one case where diphenylsilanediol was used.
4. Polymers obtained from aluminum salts such as sodium aluminum hydride gave brittle solids.

General Electric Research Laboratory.
WADC TR 59-61.
RESEARCH ON POLYMERIC BONDING SYSTEMS AND THEIR DIELECTRIC BEHAVIOR. R. P. Anderson and M. M. Spring. AF 33(616)-5535. June 1959. ASTIA Document No. AD 225910.

A literature review is given on boron polymers, with emphasis upon those that have boron-oxygen-silicon backbones. The literature on pyridyl and other nitrogen-substituted silanes is also reviewed.

The iodine catalyzed reaction of chlorosilanes with magnesium in tetrahydrofuran involves cleavage of the tetrahydrofuran ring. Thus 2,2-dimethyl-1-oxa-silacyclohexane can be prepared in good yield from dimethyldichlorosilane. Trimethylchlorosilane gives (3-buten-1-oxy)trimethylsilane and 2,2,8,8-tetramethyl-3-oxa-2,8-disilanonane.

Pyridylchlorosilanes can be prepared in reasonable yields if the cleavage reaction is avoided, via Grignard reactions or by interconversion reactions using n-butylithium and the corresponding chloro- and bromopyridines. (p(Dimethylamino)phenyl)trimethylsilane and (p(dimethylamino)-phenyl)methyldichlorosilane were also prepared by Grignard reactions in tetrahydrofuran. Convenient procedures for the preparation of 4-bromo-pyridine and r-chloropyridine are described. These compounds can be purified by distillation only if the proper precautions are observed.

Several synthetic routes to polyborosiloxanes have been examined. Benzeneboronic acid undergoes extensive dephenylation during reaction with acetoxysilanes; while silanols appear to undergo self-condensation in the presence of alkoxyboranes, and the water formed hydrolyzes the alkoxyborane. Cohydrolysis of alkoxysilanes and alkoxyboranes does not produce reasonable yields of co-condensates.

The acetoxysilane - alkoxyborane reaction has been studied in some detail. The reaction seems to hold considerable promise. However, dephenylation occurs to some extent whenever an ester of benzene-boronic acid is used; while esters of methaneboronic acid are much less reactive and tend to undergo oxidative side reactions. In the reverse reaction - that of an alkoxybornane - the presence of an amino group in the silane component greatly stabilizes the product against hydrolysis.

Wyandotte Chemicals Corporation.
WADC TR 58-187, Part II.
SYNTHESIS AND EVALUATION OF HIGH TEMPERATURE STABLE AND NUCLEAR RADIATION STABLE METAL-CYCLOPENTADIENYL FLUIDS. Robert L. Schaaf and Peter T. Kan. AF 33(616)-5053. June 1959. ASTIA Document No. AD 216451.

The objective of this work was the synthesis of materials suitable for use as high-temperature lubricants, hydraulic fluids and/or dielectric component potting materials. Seven previously unreported silox-anylferrocenes were prepared, and the thermal stability, viscosity and fluid range of these and two other siloxanylferrocenes were determined. Attempts to prepare polymeric siloxanyl-ferrocenes from difunctional silicon-substituted ferrocenes, from a cyclic silox-anylferrocene, and from bis (cyclopentadienyl)-siloxanes were unsuccessful. During this work fourteen silicon-substituted cyclopentadienes and seventeen silicon-substituted ferrocenes were synthesized.

The structure of the product from the condensation of ferrocene with formaldehyde was elucidated, and methods for the preparation of 1,1'-(γ-keto-trimethylene)-ferrocene and related compounds were investigated.

Monsanto Chemical Co. WADC TR 59-95.
SYNTHESIS OF 1000° STABLE BASE FLUIDS. James W. Dale, Elizabeth A. McElhill, Iral B. Johns and John Smith. AF 33(616)-5553. June 1959. ASTIA Document No. AD 215449.

This report presents the results of a fairly basic multipronged investigation of the preparation of thermally stable fluids liquid at room temperature and usable to 1000°F. The main emphasis of the work to date has been on the determination of the thermal stability of relatively simple basic molecules which may be linked together to form thermally stable fluids. The stability measurements have been carried out in both the vapor and liquid phases

using four types of apparatus. Approximately 50 compounds have been evaluated, and many of them synthesized.

In general, compounds appeared to be more stable in the vapor phase than in the liquid phase. In the vapor phase, the most stable compound tested was hexafluorobenzene which was stable about 1200°F, the limit of the apparatus. Benzene decomposed at 1100°F. Perfluorocyclohexane, quinoline, 2, 2'-bipyridine, pyridine, imidazole, pyramidine, naphthalene, and thiophene decomposed between 1100°F and 1200°F.

In the liquid or condensed phase, only two of the compounds tested appeared to be stable at 1000°F - naphthalene and diphenyl.

National Bureau of Standards. WADC TR 59-64. THERMAL DEGRADATION OF POLYMERS AT TEMPERATURES UP TO 850°C. Samuel L. Madorsky and Sidney Straus. AF 33(616)-58-8. June 1959. ASTIA Document No. AD 260123.

Work on thermal degradation of polymers has previously been carried out at temperatures up to about 500° C. In the present work the temperature has been extended to about 850°C. Polystyrene was pyrolyzed in a vacuum and also in helium at atmospheric pressure at 362° and 850°C. Analysis of the volatile products indicate that higher temperatures and higher pressures cause a greater fragmentation of the volatile products. Three polymers (poly(vinylidene fluoride), polyacrylonitrile, and poly (trivinylbenzene)) and 4 thermoset plastics (Vibrin, epoxy, phenolica, and silicone) were pyrolyzed in a vacuum at temperatures from 350° to 800°C and the products analyzed in the mass spectrometer. In all cases stabilization occurred in terms of a nonvolatile residue. Measurements were also made on rates of thermal degradation in a vacuum of poly(vinylidene fluoride), polyacrylonitrile, and poly(trivinylbenzene).

Wyandotte Chemicals Corp. WADC TR 59-129. MECHANISM OF CROSS-LINKING IN THE VULCANIZATION OF NEW ELASTOMERIC POLYMERS. Kay L. Paciorek, Raymond G. Spain, Eva P. Deck and Lawrence C. Mitchell. AF 33(616)-5642. July 1959. ASTIA Document No. AD 226506.

In an attempt to elucidate the mechanism of amine-induced cross-linking of the fluoroelastomers vinylidene fluoride-chlorotrifluoroelastomers vinylidene fluoride-chlorotrifluoro-ethylene (VF-CTFE) and vinylidene fluoride-perfluoropropene (VF-PFP), gelation studies, dehydrohalogenation reactions, pyrolysis, and model compound synthesis were employed.

Gelation and dehydrohalogenation studies indicated that cross-linking with primary diamines occurs readily; with tertiary monamines occurs less readily and only at elevated temperatures; was not noted in brief studies with primary and secondary monoamines. Treatment of polychlorotrifluoroethylene with hexamethylenediamine failed to afford gelation.

On the basis of these experiments, it was postulated that amine-induced cross-linking of fluoroelastomers occurs via dehydrohalogenation.

Vinylidene fluoride-perfluoropropene copolymer was treated with dimethylbenzylamine and magnesium oxide under nitrogen, and the reaction then repeated without magnesium oxide. In both instances benzaldehyde was formed.

Some pyrolysis reactions were attempted with vinylidene fluoride-perfluoropropene copolymer-hexamethylenediamine vulcanizate, but the results need to be clarified.

The multi-step synthesis of 2-trifluoromethyl-1, 1, -1, 2, 4, 4, 4-heptafluorobutane was initiated; four of the intermediate compounds have been prepared.

The literature survey was compiled on the cross-linking of selected elastomers.

Melpar, Incorporated. WADC TR 59-190, Part I. SYNTHESIS OF THERMALLY STABLE EPOXY RESINS FOR DIELECTRIC APPLICATIONS. Lee M. Kindley, Louis P. Glekas, Richard F. Marshall and Paul E. Ritt. AF 33(616)-5518. July 1959. ASTIA Document No. AD 226808.

Literature sources have not revealed any phosphorus epoxide polymers or organo-phosphorus compounds containing more than one epoxide group.

The characteristic thermal properties of commercial epoxy resins were obtained from Thermal Gravimetric Analysis (TGA). Differential Thermal Analysis (DTA), and Heat of Distortion values. Resins tested included Dow Epoxy Novalac X2638.3, Eccomold L 266, Stycast 2662, Epoxylike 813, Maraset 617, Sealcast 506, EPON X1310 laminate, EPON 828/PMDA-MA, EPON 828-EPON X1310/Diaminodiphenylsulfone, EPON 828-Triethanol Amine Titanate, and Permacel ST 3994.

It was found that Thermal Gravimetric Analysis (TGA) as compared with Differential Thermal Analysis (DTA) had an inherent advantage for comparing the thermal stabilities of polymers. The (TGA) curves share a common, well-defined "knee" in the temperature range wherein disruptive volatilization first becomes rapid, thus enabling clearer interpretation of degradation temperature.

A number of products resulting from the reaction of tatrakis (hydroxymethyl)phosphonium chloride and epichlorohydrin in the presence of aqueous sodium hydroxide yielded water insoluble foams when cured with anhydride type curing agents. Although positive identification of these products has been a formidable problem, the fact that polymers have been formed from the reaction of a quartenary phosphonium compound and epichlorohydrin is encouraging.

Even more promising results were observed in attempts to prepare a phosphorus containing epoxide by a two-step reaction of tris (hydroxymethyl)phosphine oxide and epichlorohydrin. Conditions have been worked out for obtaining satisfactory yields in the first or acid catalyzed step, but more development work is required to define conditions for the second or ring closing step.

Reaction Motors Division, Thiokol Chemical Corp. WADC TR 59-45, Part III. HIGH-TEMPERATURE STABLE SEMIORGANIC FLUIDS. PART III. PHOSPHINE OXIDES. Daniel Grafstein, Rita Dudak, Murray S. Cohen. AF 33(616)-5653. September 1959. ASTIA Document No. AD 227481. PB 161 110. Order from OTS $0.75.

The heretofore undescribed organophosphonyl fluorides were prepared and characterized. Unlike the corresponding chlorides, the organophosphonyl fluorides condense smoothly with organometallic reagents to give very high yields of the desired tertiary phosphine oxides. This represents a new one-step general synthesis of the phosphine oxides.

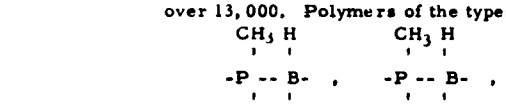
Benzenephosphophonyl difluoride, chloromethylphosphonyl difluoride and phenyl p-tolylphosphonyl fluoride were prepared for the first time and used as intermediates to the following phosphine oxides: triphenylphosphine oxide, phenyl-bis(p-biphenyl)-phosphine oxide, phenyl-bis (p-tolyl)-phosphine oxide, chloromethyldiphenyl-phosphine oxide, phenyl-o-tolylphosphine oxide and phenyl-dimethyl-phosphine oxide.

The thermal stabilities of the phosphine oxides were measured by an improved kinetic method. Phenyl-bis (p-biphenyl)-phosphine oxide was found to be thermally stable

at 400°C and to undergo rapid thermal decomposition at 448°C.

American Potash & Chemical Corp.
WADC TR 57-126, Part III.
PHOSPHINOBORINE POLYMERS. Ross I. Wagner, Frederick F. Caserio, Jr. and Levern D. Freeman. AF 33(616)-5435. September 1959. ASTIA Document No. AD 231654.

In continuing research toward the development of thermally stable polymers based on the boron-to-phosphorus bond, preliminary success has been achieved with the preparation of linear polymers of molecular weight over 13,000. Polymers of the types



have been prepared.

In addition, three new cyclic phosphinoborine trimer species have been synthesized. These, as well as some of the previously known derivatives, have been screened for oxidative, hydrolytic and thermal stability, broadening our knowledge of the relationships between the structure and the physical and chemical properties of phosphinoborines.

The synthesis of phosphine intermediates by known methods has produced four new secondary phosphines and six new organo-phosphine derivatives.

Work directed toward synthesis of borazene polymers has produced one new borazene derivative and demonstrated the principle of polymerization of borazene nuclei as well.

Merck Sharp & Dohme Research Laboratories.
WADC TR 59-151, Part I.
RESEARCH ON SYNTHESIS OF CHEMICAL INTERMEDIATES FOR HIGH TEMPERATURE FLUIDS AND POLYMERS. Robert E. Jones, John D. Garber, Samuel A. Robinson, Gustav A. Stein and George Gal. AF 33(616)-5329. September 1959.

This report covers the preparation of a wide variety of chemical intermediates required for the preparation of high temperature fluids and polymers. The intermediates prepared include organosilanes, chlorotriazines, haloalkanes, amino and nitroaryl ethers, an ester, a phosphonitrilic halide and a tetra-substituted urea. The experimental procedures used were adapted from published research on related materials. Reaction conditions and analysis of desired products are given.

University of Florida. WADC TR 59-272, Part I.
SYNTHESIS OF SEMI-INORGANIC FLUORINE POLYMERS. Henry C. Brown. AF 33(616)-5616. September 1959. ASTIA Document No. AD 229438.

Soluble thermally stable copolymers from perfluoroglutarimidine and perfluorobutyramidine have been prepared in solution and the solution viscosities studied. Although these polymers are of relatively low molecular weight, they incorporate the triazine structure as a basic unit and will serve for further cross-linking or chain extension studies.

A variety of catalysts have been shown to be effective in promoting polymerization of the perfluoroalkyl dinitriles. A copolymer of perfluoroglutarodinitrile and perfluorobutyronitrile has been formed in the presence of catalytic amounts of ammonia.

A new monomer nitrile, perfluoroethyladipodinitrile, has been prepared from perfluoroethylcyclohexene.

Irradiation of monomer nitriles and polymers containing the triazine structure by gamma rays at dosages of 1.5×10^7 r has no apparent effect on these materials.

E. I. duPont de Nemours & Co., Inc.
WADC TR 59-251.
SYNTHESIS OF THERMALLY STABLE INORGANIC AND SEMI-INORGANIC BASE FLUIDS. John A. Parkins. AF 33(616)-5843. September 1959.

The purpose of this study has been the synthesis and preliminary evaluation of inorganic, semiorganic, or organo-metallic compounds possessing extraordinary thermal stability and a wide liquid range as prototypes for base-stock fluids useful at 1000°F, (538°C) as lubricants, greases, and hydraulic fluids. A means of converting crystalline fractions of phosphorus nitrilic chloride to liquid fractions was developed. Fluoride was substituted for the chloride of the liquid fractions of the polymer, but the product boiled too low to be of interest. Attempts to prepare other derivatives of the liquid polymer were unsuccessful. Attempts to prepare the arsenic and antimony analogs of phosphorus nitrilic chloride were generally unsuccessful; however, a trace quantity of elastic polymeric material was obtained from the reaction of antimony pentachloride and ammonia, and tentative identification was made of an antimony-nitrogen structure in this material by infrared spectral studies. Antimony incorporated in phosphorus nitrilic chloride produced a significant reduction of the polymerization tendency of this polymer. A phenyl "stiboxane" was prepared from antimony oxide and partially characterized. Several phenyl stibine derivatives were prepared and given preliminary thermal screening. Initial attempts to prepare methyl cacodylate were unsuccessful. General relationships as guides for selection of low-melting eutectic mixtures were formulated and the zone-melting method was adapted for screening systems for low-melting compositions. A shear-modification method for reduction of the softening point and viscosity of glasses was proposed, but preliminary trials were hampered by mechanical difficulties.

WADC TR 59-268.
VACUUM VOLATILITY OF ORGANIC RESINS. George F. Matacek, 1/Lt, USAF. September 1959.

The effects of a high vacuum environment at various temperatures on seventeen different types of organic polymers commonly used in protective coatings were determined in this preliminary study. Weight losses and changes in physical appearances are reported. Also, the effects of film thickness, cure conditions, and exposure time on the volatility of some of the polymers were determined.

Four polymers (a silicone, a styrene-butadiene copolymer, a phenolic modified oil, and a copolymer of chlorotrifluoroethylene and vinylidene fluoride) withstood the exposures at 3×10^{-5} mm Hg pressure over the temperature range to 500°F with little or moderate change in appearance or weight. The silicone polymer was the most stable material of all those tested.

Most of the polymers tested, volatilised almost completely or were drastically charred during the course of the exposures.

WADC TR 59-453.
LINEAR POLYMERS CONTAINING THE TRIAZINE NUCLEUS. Herbert K. Reimschuessel, Edward Hagerman and Alan M. Lovelace. October 1959.

This report describes the significant aspects of the syntheses of novel linear polymers containing the

triazine nucleus within the repeating unit. The syntheses of the corresponding monomeric triazine derivatives are discussed, and the synthetic procedures and properties of a number of these new triazine derivatives are described. Furthermore, the syntheses of some prototype polymers are given. Considerable interest is being afforded to polymers of these types where the main polymer chain consists of alternating carbon and nitrogen atoms. These polymers may be generally referred to as "semi-inorganic polymers."

University of Florida. WADC TR 59-475, Part I.
THE PREPARATION OF CERTAIN HETEROCYCLIC POLYMERS BY AN ALTERNATING INTRAMOLECULAR-INTERMOLECULAR CHAIN PROPAGATION. George B. Butler, David L. Skinner, K. Darrell Berlin and Robert W. Stackman. AF 33(616)-5808. October 1959. ASTIA Document No. AD 230979.

Our research during the period indicated has led to the preparation of new organophosphorus compounds and organosilicon compounds several of which have been polymerized to give soluble polymers. All of these monomers contain two nonconjugated double bonds. The lack of residual unsaturation and their solubility characteristics indicate the polymers are linear; thus cyclization by an alternating intramolecular-intermolecular mechanism is implied for the polymerization process.

The new organophosphorus monomers which have been prepared are: diallylphenylphosphine oxide, dimethallylphenylphosphine oxide and diallylmethylphenylphosphonium bromide. Diallylhydrogenphenylphosphonium chloride and the corresponding bromide have been prepared also, but their purification has required elaborate procedures and a fairly high degree of purity has been attained only recently. Four compounds, dimethallylmethylphosphine oxide, diallylmethylphosphine oxide, diallylethylphosphine oxide, and diallylethylphenylphosphonium bromide have been prepared and tentatively identified.

Soluble polymers have been obtained from diallylphenylphosphine oxide, dimethallylphenylphosphine oxide, and diallylmethylphenylphosphonium bromide. Infrared analysis of these polymers indicates negligible unsaturation. Solubility properties and intrinsic viscosity data are included in the experimental section. Benzoyl peroxide was used as the initiator in the polymerizations of the phosphine oxides while t-butyl hydroperoxide caused the phosphonium bromide to polymerize.

Several commercially available organophosphorus esters, which conceivably could give cyclic polymers, were allowed to polymerize. Thus far, allylphenyl allylphosphonate has yielded a soluble polymer when treated in bulk with benzoyl peroxide. The infrared spectrum of this polymer shows very little unsaturation. Diallyl phosphite and diallyl phenyl phosphite are presently under investigation as possible monomers for the cyclization process.

Diallyldimethylsilane, diallyldiphenylsilane, diallylcyclotetramethylene-silane, diallylcyclopentamethylene-silane and dimethallyldimethylsilane have been prepared. The first four monomers have yielded soluble polymers when treated with the Ziegler catalyst; infrared data indicates very little unsaturation. The dimethallyldimethylsilane appears to be inert when treated with the Ziegler catalyst; this is an agreement with reported work (1). Attempts to use other catalyst systems to initiate polymerization of this monomer will be made.

Diallyldimethyl tin did not polymerize when treated with the Ziegler catalyst. Peroxide catalysts will be tried as possible initiators in the polymerization of this monomer.

Massachusetts Institute of Technology.
WADC TR 59-369.
THE PREPARATION OF ORGANOMETALLIC DERIVATIVES OF INORGANIC "BENZENOID" COMPLEXES. Dietmar Seyferth and Hubert P. Kogler. AF 33(616)-5582. October 1959. ASTIA Document No. AD 230 704.

All attempts to prepare monomeric compounds in which one or more triorganosilyl groups are linked directly to a ring atom in triazene, phosphinazine and borazene systems were unsuccessful.

The following new silicon-substituted borazene compounds have been prepared:

B-tris-(trimethylsilylmethyl)-N-trimethylborazene
B-tris-(ethyldimethylsilylmethyl)-N-trimethylborazene
B-tris-(n-butyldimethylsilylmethyl)-N-trimethylborazene
B-tris-(pentamethyldisiloxanylmethyl)-N-trimethyl-borazene
B-tris-(trimethylsiloxy)-N-trimethylborazene
N, N, N, N', N', N'-hexamethyl-B, B, B, (, B'-tetrakis-(trimethyl-silylmethyl)-B, B'-bis-borazene oxide)

B-tris-(trimethylsilylmethyl)-N-trimethylborazene seems to have good thermal stability.

Denver Research Institute. WADC TR 58-377, Part II.
RELATIONS BETWEEN STRUCTURE AND RADIATION STABILITY OF VARIOUS ALKYL AROMATIC FLUIDS. Josef J. E. Schmidt, George E. Bohner. AF 33(616)-5317. December 1959.

A knowledge of the effects of molecular structure on the radiolytic stability of certain classes of organic compounds is needed in order to synthesize more stable compounds and/or to permit more efficient equipment design. This research attempts to determine the effects of structure on the radiolytic stability of alkylpolyphenyl hydrocarbons.

Several classes of hydrocarbons were subject to gamma irradiation and where possible, quantitative measurements were made of the various degradation products. Based on the types of degradation products identified thus far, certain inferences are made concerning the structural relations of alkylpolyphenyl hydrocarbons to radiation stability.

The greatest portion of radiolytic degradation occurring in these compounds is attributed to reactions pertaining to the alkyl substituent. It appears that the degree of steric freedom of the alkyl group is related to the compound's stability.

Additional data must be obtained in order to define completely the mode of radiolytic decomposition in these hydrocarbons.

WADC TR 59-427.
CONFERENCE ON HIGH TEMPERATURE POLYMER AND FLUID RESEARCH. January 1960.

This report is the collection of papers presented at the Materials Laboratory, WADC Conference on "High Temperature Polymer and Fluid Research" held in Dayton, Ohio, on 26-28 May 1959.

The purpose of this conference was to review the recent progress in both contractual and internal research programs sponsored by the Polymer Branch of the Non-Metallic Materials Division in the area of synthesis of new polymers and fluids.

Midwest Research Institute. WADC TR 57-143, Part IV.

DEVELOPMENT OF THERMALLY STABLE SILICON CONTAINING RESINS. L. W. Breed, William J. Haggerty, Jr. AF 33(616)-3675. February 1960.

Continuing research on the preparation of silicon-containing polymers that are thermally stable and more rigid at elevated temperatures includes the preparation of both siloxane and nonsiloxane polymers as well as various intermediates and prototype compounds. Attempts to prepare certain bulky monomers, particularly silanes based on s-triazine, were unsuccessful, but siloxanes with bulky pendant groups were prepared. Siloxane polymerization reactions, hydrolysis and ester elimination, were also examined. Non-siloxanes that were studied include the following systems; silicon-oxygen-aluminum, silicon-oxygen-titanium, silane-pentaerythritol, and silane-piperazine. Whenever possible, monomers were selected to yield preordered polymers. None of the new systems have properties that equal those of the previously prepared siloxanes.

Iowa State University of Science and Technology. WADC TR 53-426, Part VIII.

ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS. H. Gilman, W. Trepka, B. J. Gaj, O. L. Marrs, G. Schwebke. AF 33(616)-6127. March 1960.

Reactions of triphenylsilyllithium with oxygen, di-, tri- and tetrasilanes, alkyl and aralkyl halides, olefins and various carbonyl compounds are described. A series of group IV-B elements analogues of 5-10-dihydroacridine has been synthesized.

U. S. Borax Research Corp. WADC TR 59-761.

RESEARCH ON INORGANIC POLYMER SYSTEMS. A. L. McCloskey, R. J. Brotherton, W. G. Woods, W. D. English, J. L. Boone, G. W. Campbell, Jr., H. Goldsmith, M. L. Iverson, H. C. Newsom, H. M. Manasevit, L. L. Petterson. AF 33(616)-5931. March 1960.

This report covers investigations by U. S. Borax Research Corporation and certain university subcontractors on the chemistry of thermally stable inorganic and semi-organic polymer systems showing promise of utility at 1000°F. Systems based on B-B, B-N, and Al-O bonding have been studied at U. S. Borax; sub-contractors have investigated polymers based on tin, on polyvalent metal salts, and pi-bonded and coordination polymers.

In the B-B system, several new monomeric and prototype compounds have been prepared. A facile new synthesis of tetra (dimethylamino) diboron has made many of these diboron compounds readily available. Polymers have been obtained by deamination of tetraaminodiborons and by the disproportionation of tetraalkoxydiborons. Thermal decompositions of prototype tetraaminodiborons and tetraalkoxydiborons were studied.

In the B-N system, thermally and chemically stable thermoplastic resins were obtained from organoborazoles at elevated temperatures. Thermal decomposition studies have provided an order of stability of various substituted borazoles. Product analyses in the decomposition study have provided some insight into the modes of decomposition of these prototypes.

In the Al-O system, a large number of prototype compounds and monomers were synthesized. Polymerization by various techniques generally gave low molecular weight materials or highly crosslinked, intractable solids stable near 400°C. Promising results have been obtained utilizing a phenyl-silicon cleavage reaction to give aluminum-oxygen-silicon resins. A detailed study has been made of the pyrolysis of aluminum alkoxides and an ionic mechanism was postulated.

National Bureau of Standards. WADC TR 59-64, Part II.

THERMAL DEGRADATION OF POLYMERS AT TEMPERATURES UP TO 1200°C. Samuel L. Madorsky & Sidney Straus. AF 33(616)-58-8. March 1960. ASTIA Document No. AD 235214.

Samples of three thermoset plastics, Vibrin, epoxy, and phenolic, and of three high-temperature polymers, poly(vinylidene fluoride), polytrivinylbenzene, and polyacrylonitrile, were pyrolyzed in a vacuum at 1200°C. The relative amounts of gaseous, liquid, wax-like, and solid products were determined. The more volatile products were analyzed in a mass spectrometer, while the less volatile ones were tested for their overall average molecular weight by microcryoscopy. Chemical analysis was made of the original materials, and also of the residues from pyrolysis at 500, 800, and 1200°C in the case of phenolic resin, poly (vinylidene fluoride), polytrivinylbenzene, and polyacrylonitrile. Pyrolysis of a high molecular weight linear polymethylene was conducted at 500, 800, and 1200°C in a vacuum and in helium, and the volatile products were analyzed by mass spectrometry and cryoscopy. A number of copolymers of styrene with di- or trivinylbenzene in various proportions were pyrolyzed in a vacuum, in the temperature range 346 to 456°C.

Measurements were made of rates of thermal degradation, in a vacuum at various temperatures, of all the polymers mentioned above, except polymethylene. The corresponding activation energies calculated from the rates were 36, 51, and 18 kcal/mole, for Vibrin, epoxy, and phenolic resins, respectively, and 48, 31, and 73 kcal/mole for poly(vinylidene fluoride), and polyacrylonitrile, and polytrivinylbenzene, respectively.

Hooker Chemical Corp. WADC TR 55-221, Part VI.

FLUORINE-CONTAINING CONDENSATION POLYMERS AND RESINS. David Knutson, John S. Kolano, John E. Wier, Edward V. Gouinlock. AF 33(616)-5548. April 1960.

The two principal objectives of this research are to determine the effect of fluorine and fluorine content on the thermal and oxidative stability of polyester laminating resins; and to investigate the preparation of perfluoroglutaronitrile, perfluoroadiponitrile, the corresponding imidine and amidine, and rigid polymers derived therefrom.

Polyesters have been synthesized from fluorinated glycols and the corresponding hydrocarbon glycols. Laminates prepared from these polyesters have been aged at elevated temperatures and their physical properties measured before and after aging. In general, the fluorinated polyester laminates exhibit much better retention of physical properties than their hydrocarbon analogs.

The report also contains a description of synthetic work directed toward the preparation of fluorine-containing compounds used in the preparation of polyesters and as cross-linking monomers.

The preparation and polymerization of perfluoroglutaronitrile is described. The polymerization of perfluoroglutarimidine and perfluoroadipamidine has been investigated.

Research on the synthesis of certain novel or unusual fluorine-containing intermediates considered desirable for polymerization in this project was performed at Purdue University under subcontract to Hooker Chemical Corporation. Details of this work performed at Purdue during the contract year comprises Appendix I of this report.

University of Denver. WADD TR 60-282, Part I.

DETERMINATION OF THE RELATIONS BETWEEN STRUCTURE AND RADIATION STABILITY OF ARYL ETHER FLUIDS. Josef J. Schmidt-Collerus, George E. Bohner. AF 33(616)-5317. May 1960.

The polyphenyl ethers are of interest to the Air Force as potential lubricants because of their demonstrated resistance to thermal and radiolytic degradation. A study of the degradation mechanisms involved in the radiolytic breakdown of diphenyl ether and higher homologs has been initiated during the current research period. A better understanding of the degradation mechanisms involved in the radiolysis of these ethers should permit a more realistic evaluation of the performance of these ethers in actual use.

It has been found that the radiolysis of diphenyl ether produces small amounts of gaseous products, mostly hydrogen, and other compounds such as benzene, phenol, p-phenylphenol, other phenolic materials, higher boiling three and four-phenylethers, and rather high molecular weight polymeric material. Although the research has not yet clearly defined a degradation mechanism for the diphenyl ether, certain postulations have been made which can plausibly explain the observed experimental data. Further research must be done in order to clearly elucidate the degradation mechanisms.

Stanford Research Institute. WADC TR 59-345.
HIGH-TEMPERATURE SYNTHESES OF NEW, THERMALLY-STABLE CHEMICAL COMPOUNDS. Russell C. Phillips, David L. Chamberlain, F. Alan Ferguson. AF 33(616)-5940. May 1960.

Studies of high-temperature reactions for synthesis of new, thermally-stable chemical compounds or precursors to such compounds, are described. The first part of the program was a survey of the literature for high-temperature reactions (above 500°C) which might result in such compounds. On the basis of this initial survey, and on information from continuing literature surveys, the following three studies were undertaken:

1. Synthesis of difluoroacetylene as a precursor to thermally stable, perfluorinated aliphatic or aromatic polymers.
2. Synthesis of stable organometallic compounds using arcs to ionize the metal constituent.
3. Synthesis of thermally-stable thiophosphonitrilic compounds.

Of three different approaches conceived for the synthesis of difluoroacetylene, only pyrolysis of the sodium salt of tetrafluorosuccinic acid appears to have yielded the desired product, although the analytical findings are not yet conclusive. Investigations of the reaction between atomic fluorine and graphite and of the pyrolysis of difluoromaleic anhydride are not yet completed.

The reactions between metals ionized from arc electrodes, and organic liquids circulated past the arc, have failed thus far to yield measurable quantities of organometallic compounds.

Reactions between vaporized phosphorus sesquisulfide (P_4S_3) and nitrogen in an electric arc have yielded several amorphous compounds containing sulfur, phosphorus and nitrogen. One such compound was heated to over 700°C in argon with no visible signs of decomposition.

Wayndotte Chemicals Corp., and University of Illinois. WADC TR 58-187, Part III.
SYNTHESIS AND EVALUATION OF HIGH TEMPERATURE STABLE AND NUCLEAR RADIATION STABLE METAL-CYCLOPENTADIENYL FLUIDS. Robert L. Schaaf, Peter T. Kan, Kenneth L. Rinehart, Jr. AF 33(616)-5053. May 1960.

In a search for liquids suitable for use as high-temperature hydraulic fluids and lubricants, six new siloxanylferrocenes were synthesized, and the thermal stability, viscosity and fluid range were determined. Efforts to find feasible routes to 1,1'-bis(aryloxyphenyl)-ferrocenes, polychloroferrocenes, and polymeric perfluoroalkylbenzimidazoles were unsuccessful.

From the lithiation and subsequent carbonation of dimethylferrocene, the enantiomorphic forms of 1,1'-dimethylferrocene- the enantiomorphic forms of 1,1'-dimethylferrocene-3-carboxylic acid was isolated, and 1,1'-dimethylferrocene-2-carboxylic acid and a mixture of di-acids were obtained. By reduction of the required esters with lithium aluminum hydride, mono- and bis-hydroxymethylferrocenes were prepared. Oxidation of desoxyferrocoin, methyl-, and ethylferrocene, respectively. The reduction of nitroferrocene, prepared from n-propyl nitrate and lithioferrocene, was investigated. Unsuccessful were attempts to prepare nitrosoferrocene, 1,1'-diaminoferrocene, and to unite hydroxymethylferrocene with methyl linolenate or ethyl linoleate.

Polytechnic Institute of Brooklyn. WADC TR 58-382, Part II.
SYNTHESIS AND EVALUATION OF NEW POLYMERS PREPARED BY STEREOSPECIFIC CATALYSIS. Charles G. Overberger. AF 33(616)-5253. May 1960.

3-Trifluoromethyl-1-butene and 4,4,4,-trifluoro-1-butene have been prepared. Trifluoromethylacrylonitrile was synthesized and polymerzed with several bases.

Methylenecyclobutane of high purity was obtained from 1,1-bis-(hydroxymethyl)-cyclobutane. Vinylcyclopropane of high purity was obtained from 1-cyclopropylethyl-S-methylxanthate while the pyrolysis of 1-cyclopropylethyl acetate yielded cis 1,3-pentadiene as the major product. Cyclo-butylethyl acetate was prepared and pyrolysis of cyclobutylethyl acetate yielded a mixture of olefins. Cyclobutanecarboxaldehyde was synthesized and found to trimerize to a cyclic ether which has been characterized. A lactone reduction product of 4-acetyl-3,3-dimethylcyclobutylacetic acid has been characterized and reduced to a diol which has also been characterized. A 2 step reduction of 4-acetyl-3,3-dimethylcyclobutylacetic acid yielded a cyclo-butylethanol, which has been characterized, and an unidentified ether. 3-Cyclo-hexenylmethyl acetate was prepared, and the pyrolysis of this ester yielded a mixture of methylenecyclohexene, toluene, methylenecyclohexane and several additional unidentified olefins. Methylenecyclohexene of high purity was obtained from 3-cyclohexenylmethyl-S-methylxanthate. Pure cis or trans 2-methyl-vinylcyclohexane has been synthesized, characterized and polymerized.

An isotope effect was observed in the polymerization of alphadeuterovinylcyclohexane in a comparison with the polymerization of vinylcyclohexane with a triethylaluminum-titanium tetrachloride catalyst.

The DP of polymethacrylonitrile from potassium in ammonia catalysis was approximately independent of both monomer and catalyst concentrations. Potassium amide catalysis produced an acetone insoluble polymer, and potassium hydroxide polymerized methacrylonitrile in ammonia. Water exerted an observable effect on potassium or potassium amide catalysis in the polymerization of methacrylonitrile in ammonia.

Polytechnic Institute of Brooklyn. WADC TR 58-382, Part III.
SYNTHESIS AND EVALUATION OF NEW POLYMERS PREPARED BY STEREOSPECIFIC CATALYSIS. Charles G. Overberger. AF 33(616)-5253. May 1960.

From the pyrolysis of 1-cyclopropylethyl acetate, polyvinylcyclopropane was prepared and the polymer modified by ring opening with hydrogen bromide, bromine, p-toluenesulfonic acid, and acetic-sulfuric acid mixture. 2-Trifluoromethylpropyl bromide was prepared and used to alkylate ethyl malonate. 3-Trifluoromethylbutene-1 was

prepared by pyrolysis of the primary acetate. Ethyl 3, 3-methylcyanobutyrate was prepared but could not be reduced to the corresponding cyanoalcohol. 1, 1, 1-Trichloro-3-bromopropane was converted to 1, 1, 1-trifluoro-3-bromopropane. Basic hydrolysis of 4, 4-methylcyanopentene-1 yielded 2, 2-dimethylpent-4-eneoic acid. 2-Trifluoropropane was prepared, but attempts at alkylation were not successful. 6, 6, 6-Trifluorohexene-1 was prepared but could not be obtained in a pure state. Homogeneous polymerization of trifluoromethacrylonitrile was effected. A methoxymercuriacetate derivative of 2-trifluoromethyl propene was prepared. A mixture of methylenecyclobutane, spiropentane, 2-methylbutene-1 was the product of the reduction of pentaerythrityl tetrabromide with zinc in various solvents. Methylenecyclobutane was purified by distillation, and also by regeneration from a silver nitrate solution. Methoxymercuration of methylenecyclobutane gave a solid derivative from which the monomer could be regenerated. The yield of polymethylenecyclobutane from polymerization with titanium tetrachloride-aluminum triisobutyl was found to increase with increasing temperature. Acid catalyzed rearrangement to 1, 1-bis-(hydroxymethyl) cyclopropane gave cyclobutanecarboxaldehyde. The corresponding cyclobutane gave cyclopentanecarboxaldehyde. It was found that the solvent strongly influences the reactivities of isobutylene, p-chlorostyrene when copolymerized with aluminum bromide or stannic chloride. The molecular weights of polystyrene polymerized in solvents of varying dielectric constants with aluminum bromide or boron trifluoride etherate as catalyst were determined. The xanthates of 1-cyclobutylethanol and 3-cyclohexenylmethanol were prepared. Methyl vinyl sulfone was prepared and a high vacuum manifold was constructed for its polymerization.

Melpar, Inc. WADC TR 59-190, Part II. SYNTHESIS OF THERMALLY STABLE EPOXIDE RESINS FOR DIELECTRIC APPLICATIONS. Lee M. Kindley. AF 33(616)-5518. May 1960.

The objective of the program was to prepare organophosphorus epoxides capable of being cured to thermally stable resins. To achieve this objective, several synthesis routes were explored.

Three new tertiary phosphine oxides were prepared and characterized. These compounds and several known organophosphorus compounds, to be used as intermediates, were prepared and analyzed.

The characterization of new phenolic tertiary phosphine oxides, which were to be reacted with epichlorohydrin to yield phosphorus epoxides, proved to be a difficult problem. After considerable development work, results indicated that the problem was one of analysis rather than preparation.

The preparation of a phosphorus-containing epoxide resin by condensing tris (hydroxymethyl) phosphine oxide with an epihalohydrin was investigated in some detail. Phosphorus-containing resins having a small amount of oxirane oxygen were isolated from some of the reactions. Attempts were made to cure these products with the hope of obtaining a useful product, but only thermoplastic or very viscous liquids were obtained.

General Electric Co. WADD TR 60-283. EVALUATION OF EXPERIMENTAL POLYMERS. Charles D. Doyle. AF 33(616)-5576. June 1960.

Thermogravimetric analysis (TGA) in dry N_2 is considered in detail as a method for comparing the intrinsic thermal stabilities of experimental polymers on both empirical and fundamental grounds. Two procedural decomposition temperatures are defined and discussed. One, the "differential procedural decomposition temperature" (dpdt), is based on the locations of recognizable curve features, while the second, the "integral procedural decomposition temperature" (ipdt), is based on areas under the curve. Kinetic analysis of volatilization data is discussed on the basis of both the Arrhenius rate equation and its integral. Two quasi-kinetic methods are discussed, one based on empirical time-temperature superposition; the other, on an empirical relationship between isothermal times and temperatures in TGA. Two corroborative test methods, differential thermal analysis and thermoparticulate analysis are discussed briefly.

Stanford Research Institute. WADC TR 59-345, Part II. HIGH-TEMPERATURE SYNTHESIS OF NEW, THERMALLY-STABLE CHEMICAL COMPOUNDS. Lars C. Bratt, David L. Chamberlain, F. Alan Ferguson. AF 33(616)-5940. June 1960.

Results of the continuing investigation of high-temperature reactions for the synthesis of new thermally-stable compounds is reported. The solid product obtained from the pyrolysis of disodium tetrafluorosuccinate is believed to possibly be a low molecular-weight polymer of difluoroacetylene whose presence, in addition to other carbon-fluorine groups, was indicated by mass spectral analysis of the volatile products.

Reaction of phosphorus sulfide (P_4S_3 or P_4S_{10}) vapors with nitrogen in an electric arc yielded inert, amorphous, thermally-stable solids containing sulfur, phosphorus and nitrogen. These as yet uncharacterized products were found to be insoluble in common organic solvents but hydrolyzed very slowly when extracted with boiling water.

The possibility of forming organic iron compounds by shock chilling a plasma-jet containing thermally-activated iron with benzene, dicyclopentadiene and cyclopentadiene was investigated. However, only an insignificant concentration of iron was found in any of the liquid products obtained from such reactions, indicating the absence of any organoiron derivatives.

Olin Mathieson Chemical Corp. WADD TR 60-315. SYNTHESIS OF NITROGEN-CONTAINING HETEROCYCLIC FLUID SYSTEMS. E. H. Kober, W. J. Schnable, H. A. Schroeder. AF 33(616)-6342. June 1960.

Melam, melem, cyameluric acid, and melonic acid form a class of solid and infusible compounds known for their extreme heat stability. Experiments, to modify these structures by attaching alkyl, benzyl, phenyl and substituted phenyl groups aiming to produce still thermally stable, but liquid derivatives, are reported. Numerous substituted melams, melems, phenylcyamelurines, and esters of cyameluric acid have been prepared. Many preparations show excellent heat stability and have melting points in the range of 90 to 120°C; liquids have not been obtained. Derivatives of melonic acid could not be isolated.

Monsanto Chemical Co. WADC TR 59-95, Part II. SYNTHESIS OF 1000°F STABLE BASE FLUIDS. James W. Dale, Iral B. Johns, Elizabeth A. McElhill, John O. Smith. AF 33(616)-5553. June 1960.

The results of tests for thermal stability directed at obtaining thermally-stable fluids which are liquid at room temperature and usable to 1000°F are presented. In this extension of the project an additional 50 compounds were tested in the vapor phase, or the liquid or condensed phase using four different types of apparatus. Many of the compounds were synthesized.

A number of fluoroorganic compounds were prepared for thermal stability evaluation. Several perfluorocyclic

compounds were prepared by fluorination of the corresponding aromatics, and in the case of $(CNF)_3$, by fluorination of $(CNCL)_3$ with SbF_3Cl_2. Other fluoroorganics, phenyl- or pyridyl-substituted perfluoroalkanes, were synthesized from SF_4 and the corresponding ketones or carboxylic acids. Evidence was obtained of a new reaction of SF_4 with aryl oxalates and other oxalic acid derivatives.

Compounds found stable over 1000°F included dibenzothiophene tested in the condensed phase, and tetrakis (perfluoromethyl) pyrazine and perfluorocyclobutane-perfluoroisobutylene tested in the vapor phase.

University of Florida. WADC TR 59-272, Part II.

SYNTHESIS OF SEMI-INORGANIC FLUORINE POLYMERS. Henry C. Brown. AF 33(616)-5616. June 1960.

The mechanism of the deammonation reaction of perfluoroalkyl amidines have been explored and the first step shown to be the formation of perfluoroalkyl imidines. These intermediate compounds readily form copper (II) and zinc chelates. Further steps in the deammonation reaction, which results finally in formation of a sym. triazine ring, are proposed.

The possible formation of a linear polymeric structure from the low temperature reaction of perfluoroglutaronitrile with perfluorobutyramidine is described. This structure is a possible precursor to a linear perfluoroalkyl-triazine polymer.

Copolymerization o. erfluoroglutaronitrile with perfluorobutyronitrile in the presence of basic catalysts other than ammonia is described. Copolymerization of perfluoroglutaronitrile with perfluoropropionitrile and perfluoroacetonitrile in the presence of catalytic amounts of ammonia appears to furnish copolymers of physical characteristics different from those previously reported but with high temperature stability.

Cotrimerization of perfluoroacetonitrile with benzonitrile is demonstrated and the resulting compounds, 2, 4-bis phenyl-6-trifluoromethyl-1, 3, 5-triazine and 2-phenyl-4, 6-bis trifluoromethyl-1, 3, 5-triazine are considered as possible monomers.

WADD TR 60-700.

CORRELATION BETWEEN STRUCTURE AND THERMAL STABILITY OF EPOXY RESINS. Gerhard F. L. Ehlers. July 1960. ASTIA Document No. AD 245270.

The objective of this study was to survey the relationship between the structure and thermal stability of epoxy resins.

A basic epoxy resin from Bisphenol A, as well as a number of other di- and poly-epoxy resins of defined structure, were cured with equivalent amounts of various anhydrides, amines, phenols and catalysts. Weight loss of these resins was determined from periods up to 200 hours at 230°C, also the Vicat heat distortion temperature was determined before and after several aging periods.

Thermal stability, indicated by the weight loss data, and heat softening, indicated by the maximum heat distortion temperature obtained during the aging period, were correlated with the structures of the synthesized resins.

Rigid (aromatic) structures as well as high functionality of the reactants, or dense crosslinking were found to contribute to a high heat distortion. Anhydrides as curing agents were found to be more favorable in this respect than phenols and amines, because the reactivity towards epoxy as well as secondary hydroxyl groups resulted in higher crosslinking density.

Comparison of the three types of curing agents indicated about equal stability of the -C-O-C- and the -C-NH-C- linkage. Both were somewhat more stable than the ester linkage -C-O-C-.

Unexpected high heat softening points were obtained by using additives with one epoxy group and a double bond, such as dipenteneoxide, or curing agents, containing a double bond, such as maleic anhydride. The results obtained under certain conditions indicate that the double bonds apparently are polymerized due to the presence of epoxy groups, resulting in additional crosslinking.

Wyandotte Chemicals Corp. WADC TR 59-129, Part II.

MECHANISM OF CROSS-LINKING IN THE VULCANIZATION OF NEWELASTOMERIC POLYMERS. Kay L. Paciorek, Lawrence C. Mitchell. AF 33(616)-5642. July 1960. ASTIA Document No. AD 245281.

The objective of this program was the elucidation of the mechanism of amine cross-linking of fluoroelastomers. Two approaches were undertaken: solution studies of the copolymers of vinylidene fluoride-chlorotrifluoroethylene (VF-CTFE) and vinylidene fluoride-perfluoropropene (VF-PFP), and investigations of certain model systems.

Solution studies revealed a great difference in the behavior of the two fluoroelastomers. Isolation of amine hydrohalides from room-temperature reactions of amines with fluoroelastomers in conjunction with the presence of unsaturation in the resulting polymers (shown by infrared examination), proved dehydrohalogenation to be the first step in amine induced cross-linking. Various mechanisms of cross-linking were postulated.

Several syntheses of model compounds were proposed. A synthesis utilizing 1, 1, 5-trihydroperfluoropentanol resulted in models suitable for amine studies. Parallel syntheses were also initiated. Preliminary model compound studies utilizing 1, 1, 3, 3, 5, 5-hexafluoro-1, 5-hexafluoro-1, 5-dibromopentane supported the findings of the solution studies.

University of Florida. WADD TR 60-478, Part I.

THE PREPARATION OF CERTAIN HETEROCYCLIC POLYMERS BY AN ALTERNATING INTRAMOLECULAR-INTERMOLECULAR CHAIN PROPAGATION. George B. Butler, K. Darrell Berlin, David L. Skinner, Robert W. Stackman. AF 33(616)-5616. July 1960.

A number of additional monomers containing phosphorus and silicon have been synthesized and their polymerization studied. These monomers contain two non-conjugated double bonds in the 1, 6-positions. All have been found to undergo polymerization by the intra-intermolecular chain propagation to produce soluble polymers. Some of the properties of the polymers are reported.

American Potash & Chemical Corp. WADC TR 57-126, Part IV.

PHOSPHINOBORINE POLYMERS. Ross I. Wagner. AF 33(616)-5435. August 1960.

A series of preparations of linear dimethyl-, methylethyl- and diethylphosphinoborine polymers have been made to optimize experimental conditions. N-Methylpiperazine when used as an end-group for linear dimethylphosphinoborine polymers provided a fair yield of product which, except for a large melting range, resembled polymers terminated with other end-groups. Triethylenediamine markedly reduced the rate of dimethylphosphine borine pyrolysis and failed to yield a linear polymeric product. Qualitative solubility data have been obtained for the linear methylethyl- and diethyl-derivatives. Attempts to prepare linear phosphinoborine polymers either by pyrolysis of trimethylphosphine trichloroborine or

dimethylphosphinoborine trimer with triethylamine or by alkylation of methylphosphinodimethylborine polymer were unsuccessful. Linear methylethylphosphinoborine polymer was only partially chlorinated on treatment with methyl chloride--aluminum chloride in contrast to the dimethyl-derivative. A further attempt to open the dimethylphosphinoborine trimer ring by heating with triethylenediamine resulted instead in a polymer formed by opening the ring structure of the diamine.

Dimethylphosphinoborine trimer was prepared in good yield from dimethylphosphonium chloride and lithium borohydride and in low yield by dehydrohalogenation of dimethylphosphine chloroborine. Dehydrohalogenation of dimethylphosphine diethylchloroborine and dimethylphosphine trichloroborine failed to give cyclic trimeric products but the latter appeared to yield low molecular weight linear phosphinoborine polymers. The stoichiometry of the fluorination of dimethylphosphinoborine trimer has been established experimentally but no success has been achieved in attempts to cyanogenate the trimer. An X-ray diffraction study of the dimethylphosphinoborine-methylphosphinoborine pentamer indicated the crystals to be of complex structure. Dimethylphosphinodichloroborine trimer appears to be less thermally stable than the parent compound.

The synthesis of phosphine intermediates by known methods has produced one new secondary phosphine and one new organophosphine derivative. Nuclear magnetic resonance spectra of the phosphine, C_2H_5P, prove it to be cycloethylenephosphine rather than the isomeric vinylphosphine.

N-dimethyl-B-trimethylborazene has been synthesized both by dehydrohalogenation of a mixture of the methyldibromoborine adducts of methylamine and ammonia and by pyrolysis of a mixture of the trimethylborine adducts of methylamine and ammonia. The isomeric N-trimethyl-B-dimethylborazene was prepared by reaction of N-trimethylborazene with a deficiency of methylmagnesium bromide. The attempt to prepare decamethylbiborazyl by pyrolytic dehydrogenation of the two isomeric pentamethylborazenes resulted in formation of by-product methane and a polymeric borazene derivative. The pseudoaromatic 8-bora-7, 9-diazaro-peri-naphthene has been prepared and studied briefly.

Dimethyl- and diphenylthiosiloxane polymers have been prepared by reaction of the corresponding dichlorosilanes with hydrogen sulfide. In the presence of trimethylchlorosilane the preparation of diphenylthiosiloxane polymers resulted in a high yield of a single crystalline solid, presumably the trimer or dimer.

General Electric Research Laboratory.
WADC TR 59-61, Part II.
RESEARCH ON POLYMERIC BONDING SYSTEMS AND THEIR DIELECTRIC BEHAVIOR. R. P. Anderson and M. M. Sprung. August 1960. ASTIA Document No. AD 244766.

Further work on the formation of borosiloxane polymers, particularly by the reaction between an acetoxysilane and an alkoxyborane or by the reaction between an alkoxysilane and an acetoxyborane, is described in this report. A variety of low polymers, bearing different substituents on silicon and on boron, were prepared. Several nonlinear, hydrocarbon-insoluble fractions were isolated, and it was shown that disproportionation sometimes, but not necessarily always, occurs. Some dephenylation of a phenylborane moiety almost always occurs when high temperatures are employed.

In the reaction of an aminoalkylalkoxysilane with boron acetate, the amino substituent enters the reaction by elimination of acetic acid. A primary amine will undergo group interchange with an acetoxy silane.

Dichloro-p-(dimethylaminopheny)methylsilane and dichloro-p-(dimethyl-aminophenyl)phenylsilane were prepared and converted to the corresponding diacetoxysilanes. Dichloro-p-(dimethylaminophenyl)phenylsilane was hydrolyzed nearly quantitatively to the corresponding diol, and the diol was reacted with benzeneboronic acid to produce a nitrogen-substituted borosiloxane.

The equilibrium of cyclic siloxanes and boroxanes were studied briefly. Here dephenylation is negligible at reasonable temperatures, but oxidation is still possible. Thermal equilibration, with the boron component acting as an auto-catalyst, is a slow process but gives better results than base catalyzed equilibration.

Several amine complexes of tributyl borate were prepared. Crystalline complexes of triphenylboroxole and amines were not obtained, but interesting, stable "adducts" were obtained with several polyethylene amines. A similar adduct was obtained from poly(triphenylborosiloxane) and tetraethylene pentamine.

Several aminoalkyl borates were prepared and their properties compared to simple alkyl borates. These data were then extended to linear polymers formed from alkyl borates with diols or aminodiols; and to crosslinked polymers formed from alkyl borates with diols, oxadiols, or aminodiols. It was observed that whenever five-membered rings can be formed through B-N dative bonding, this reaction route was favored almost exclusively over polymer formation.

WADD TR 60-357.
MOLECULAR WEIGHT DETERMINATION IN THE CHARACTERIZATION OF POLYMERS. Richard M. Lange. September 1960. PB 171 401. Order from OTS $0.75.

Preliminary results are given on the determination of the macrostructure of poly-(dimethyl-2, 2'-dimethylene pimelate) prepared by solution polymerization of 40% conversion. The physical methods used in this study include osomometry, light-scattering, viscometry and fractional precipitation.

Osmotic pressure measurements on a sample of the whole polymer in toluene solution yielded a number-average molecular weight of 3.36×10^5 grams/mole. Light-scattering measurements on the same system gave a weight-average molecular weight of 2.80×10^6 grams/mole. The heterogeneit index (M_w/M_n) of this polymer indicates a broad distribution of molecular weights.

A small sample of the polymer was subjected to fractional precipitation techniques with only partial success. Weight and number-average molecular weights were obtained on two fractions.

WADD TR 60-663, Part I.
DETERMINATION OF RELATIVE STABILITY OF UREA COMPLEXES FROM X-RAY POWDER DIFFRACTION DATA. Jack Radell and J. W. Connolly. October 1960. ASTIA Document No. AD 249 102.

Urea complexes consist of a channel made up of hydrogen-bonded urea host molecules into which a variety of guest molecules can fit to form a crystalline complex. The stability of the resulting complex is a function of the size and properties of the guest molecule. A procedure has been devised which produces pure complex free of any uncomplexed urea or host molecules in solution. The X-ray data and stability was determined for several members of each of the following classes of compounds: alkyl-silanes, partially fluoroinated esters, fumarates, malcates and acetylene derivatives.

TR 4860
PROPERTIES OF THE SYNTHETIC RUBBERS. P. J. Mahoney, J. I. Wittebort, C. E. Jaynes. November 1942.

TR 5891
INVESTIGATION OF RUBBER PLASTICIZERS FOR LOW TEMPERATURE APPLICATIONS. Earl R. Bartholomew. May 1949.

Washington Univ. TR 52-192.
INVESTIGATION OF THE SYNTHESIS AND TESTING OF VARIOUS POSSIBLE BORON-ORGANO AND OTHER SIMILAR POLYMERS. L. E. Stout, D. F. Chamberlain. AF 33(038)-23299. June 1952. PB 112048. Order from LC, Mi $2.75, Ph $7.50.

Exploratory work on the preparation of organo-titanium and organo-boron compounds is described. Initial efforts were directed toward the synthesis of compounds of the type $R_2Ti(OH)_2$ and $RB(OH)_2$. The work on titanium supports the evidence previously reported in the literature, that titanium-organo compounds, involving a Ti-C bond, cannot be formed.

Efforts to synthesize the boronic acids $RB(OH)_2$ were moderately successful; of the alkyl derivatives, the propyl, butyl, and hexyl were isolated, of the aryl type, only the phenyl derivative was obtained. All proved to be heat and moisture sensitive, and no condition of temperature, concentration, time atmosphere, or catalyst could be found which would induce condensation polymerization to $\left[-\underset{\displaystyle R}{\overset{|}{B}}-O-\right]_n$

Organo complexes of boron trifluoride and dihydroxyfluoboric acid were formed. Heating of these complexes produced various reactions, primarily decomposition, but did not result in polymer formation.

Condensation polymers were formed from boric acid and various glycols, but all were low melting and water solubie. Polymers were also formed from various mixtures of glycerol, boric acid, and phthalic anhydride (or maleic anhydride, or fumaric acid). Again the polymers were low melting and water sensitive.

It is concluded that the boronic acids do not undergo condensation polymerization similar to the dihydroxy silicones, but decompose to boric acid or the anhydride. Further, when polymeric esters or amides containing boron are formed, the presence of the boron introduces extreme water sensitivity.

TR 52-180.
INVESTIGATION OF SILICATE ESTERS AS RUBBER PLASTICIZERS. Donald L. Byerley, 2/Lt. August 1952. PB 133043. Order from LC, Mi $2.40, Ph $3.30.

A number of silicate esters, submitted by the John B. Pierce Foundation, Raritan, New Jersey, were evaluated as low-temperature rubber plasticizers. An attempt was also made to correlate structural variations of the esters with the properties imparted to the compounded rubber. It was found that, in general, as the ratio of the number of aliphatic carbon atoms/number of phenyl groups increased the ASTM brittle point was lowered. Variation of the structure of the silicate ester, on the other hand, appeared to have little effect on the physical properties (e.g. tensile strength, modulus, and elongation). Compatibility with common base polymers (Paracril 18, Paracril 35, and Neoprene W) decreased rather rapidly as the above ratio increased beyond a value of about 2.5. Extrapolation of these results indicates that a ratio value above 20 would not permit incorporation of a sufficient amount of the silicate ester to affect the low temperature properties of the compound stock significantly.

Minnesota Mining and Manufacturing Company
TR 52-197, Part 2.
SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS. A. M. Borders. AF 33(038)-515. August 1952. PB 116222. Order from LC, Mi $5.00, Ph $15.25.

Exploratory studies of the preparation and properties of fluorine-containing polymers are described. These syntheses and tests are directed to highly solvent resistant elastomers with the widest possible useful temperature range. New classes of materials include polymers of unsaturated and perfluoralkyl esters, fluorine-containing alkoxyalkyl acrylates, and vinyl 1,1-dihydroperfluoroalkyl ethers, and copolymers of perfluoroacrylonitrile, and of perfluorobutadiene.

Polymers and copolymers of 1,1-dihydroperfluoroalkyl acrylates continue to exhibit the best balance of low temperature flexibility and resistance to aromatic hydrocarbon fluids. Improved curing systems and reinforcement studies of these rubbery acrylate polymers have permitted comparison with commercially available synthetic rubbers. These compounds have adequate mechanical properties for gasket performance and have exceptional resistance to swelling by many fuels and fluids of interest to the Air Force. Although reinforcement of the fluoroacrylate homopolymers has not been possible, their butadiene copolymers have been reinforced to vulcanizates with tensile strengths in excess of 2,000 psi. Homopolymers and butadiene copolymers of the butyl and hexyl acrylates have been submitted or are being prepared for further evaluation by Wright Air Development Center.

The ultimate goal in low temperature flexibility has so far not been reached. At the request of the Wright Air Development Center increased emphasis will be given to fluorine-containing elastomers of improved high temperature resistance. Promising leads are described with perfluorobutadiene copolymers and with suitably vulcanized poly-1,1-dihydroperfluorobutyl acrylate.

Thiokol Corporation. TR 52-230.
FUEL CELL SEALANT COMPOUNDS. Earl H. Sorg, John F. McCarthy, Edward M. Fettes, Joseph S. Jorczak. September 1952.

This work was undertaken by the Thiokol Corporation to develop integral fuel tank sealant compounds with improved low and high temperature properties, increased toughness and adhesion, and better resistance to jet fuels.

Experimental integral fuel tank sealant compounds were prepared in two-package mixes from hexamethylene dichloride/triglycol dichloride/formal and pentamethylene dichloride/formal polymers. Compared to a 'Thiokol' LP-2 base sealant, these experimental compounds displayed improved low temperature properties, equivalent toughness and adhesion to aluminum, somewhat better resistance to heat aging at 212°F, and slightly poorer resistance to swell in aromatic fuels.

Compounding studies with 'Thiokol'LP-2 yielded formulations with improved adhesion properties and heat aging resistance.

Development work on clear sealant compounds disclosed the possibility of preparing sealants of this type from combinations of epoxide resins and 'Thiokol' liquid polymers; however, considerably more investigation of this aspect is required.

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Purdue Research Foundation. TR 52-191, Part 1.
FLUORINE CONTAINING ELASTOMERS. O. R. Pierce, E. T. McBee. AF 33(038)-20581. October 1952. PB 111284. Order from OTS $2.00.

The nature of the work described in this report embraced the synthesis of two types of materials: (a) fluorine-containing silicones, and (b) fluorine-containing styrenes copolymerized with butadiene. These materials are to be evaluated as low-temperature, fuel-resistant elastomers.

The fluorine-containing silicones were synthesized by routes employing the reaction of a fluorine-containing Grignard reagent with silicon tetrachloride or tetraethyl silicate followed by hydrolysis of the monomer silane obtained. In particular, the monomer, $(C_3F_7CH_2CH_2)_2SiX_2$(X=Cl or OEt) was prepared. Also, a perfluoroalkyl Grignard reagent, C_3F_7MgI, was synthesized and its utility in the preparation of perfluoroalkyl silicones studied. At the conclusion of the first year's work, the achievements indicate that the synthesis of highly fluorinated silicone elastomers is feasible and the research is being continued.

The fluorine-containing styrenes were prepared by known methods as follows:

$$C_6H_5CF_3 + Br_2 \rightarrow 3\text{-}BrC_6H_4CF_3 + Mg \rightarrow 3\text{-}(CF_3)C_6H_4MgBr \xrightarrow{CH_3CHO} 3\text{-}(CF_3)C_6H_4CHOHCH_3 \xrightarrow{P_2O_5} 3\text{-}(CF_3)C_6H_4CH{=}CH_2$$

Three monomers were obtained in this way: 3-(trifluoromethyl)styrene, 3,5-bis(trifluoromethyl)styrene, and 2,5-bis(trifluoromethyl)styrene. These materials were copolymerized with butadiene using an emulsion technique. After preparation of polymer samples was complete, the work was discontinued in this laboratory and no further investigations along this line are contemplated.

Minnesota Mining and Manufacturing Company
TR 52-197, Part 1.
SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS. W. H. Pearlson and N. W. Taylor. AF 33(038)-515. October 1952. PB 116221. Order from LC, Mi $5.00, Ph $15.25.

Exploratory investigation into the preparation and properties of fluorinated polymers is described. New polymeric compositions of matter have been synthesized and screened with respect to their potentialities as suitable elastomers for use under extreme conditions of temperature in contact with various fuels and oils. The esters of unsaturated hydrocarbons acids with 1,1-dihydroperfluoroalkyl alcohols represent the most promising class of monomers investigated so far; certain of their polymers have received considerable attention because of their rubbery properties and exceptional resistance to swelling by non-fluorinated solvents, although their low temperature flexibility will probably require extension by plasticization. Fluorinated condensation-type polymers such as polyesters and polyamides do not appear promising as elastomers but may have potentialities as fibers, films or structural plastics. Extensive structure versus property correlations are not attempted but it is concluded that either side-chain or backbone fluorine in polymeric structures promotes resistance to swelling by common solvents; the former does not appear to affect low temperature properties as adversely as sketal attachment. The possibility of constructing a better monomer than a fluoro-alkyl acrylate for development into a superior elastomer for specialized Air Force applications is still conceded for the field is relatively new and the number of known compounds small.

Phillips Petroleum Co. TR 52-80
INVESTIGATION OF PLASTICIZERS FOR OIL-RESISTANT RUBBER FOR SERVICE AT LOW TEMPERATURES IN CONTACT WITH HYDROCARBON FLUIDS. J. C. Hillyer, C. S. Imig. AF 33(038)-17201. November 1952. PB 123646. Order from LC, Mi $3.50, Ph $7.80.

TR 52-338.
USAF REQUIREMENTS FOR RUBBER MATERIALS. Capt. W. Postelnek. December 1952.

Both operational and environmental conditions profoundly affect the desirable properties of rubber materials used in military aircraft. Many requirements exist for improved elastomeric materials which will retain their rubbery properties over a wide temperature range in contact with various fuels, oils and corrosive chemicals, as well as on exposure to adverse atmospheric phenomena. Currently, a research and development program is geared to provide solutions to the more pressing problems.

Minnesota Mining and Manufacturing Co.
TR 52-197, Part 3.
SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS. F. A. Bovey. AF 33(038)-515. September 1953. ASTIA Document No. AD 62929. PB 129549. Order from LC, Mi $6.30, Ph $19.80.

This report describes the preparation and properties of fluorine-containing rubbers. The object of the work is the development of elastomeric materials which are resistant to the fuels, lubricants, and hydraulic fluids used in military aircraft and which are serviceable over the widest possible temperature range.

Of the materials under development, the following appear to be of chief interest:

A. Perfluorobutadiene Copolymers. Copolymers of perfluorobutadiene with 1,1-dihydroperfluoroalkyl vinyl ethers offer excellent high temperature resistance, low swelling in aircraft fluids, and high resistance to ozone.
B. Fluoroacrylates. In addition to the 1,1-dihydroperfluoroalkyl acrylates, which have been described in earlier reports and which have outstanding solvent resistance but limited low temperature flexibility, newer classes of fluoroacrylates have been synthesized. Of particular interest are the ω-(perfluoroalkoxy)-1,1-dihydroperfluoropropyl acrylates, which offer solvent resistance at least equal to that of the earlier series but are flexible at temperatures 25°C lower than the polymers of the 1,1-dihydroperfluoroalkyl acrylate.

In the compounding and vulcanization of fluoroacrylate polymers, the most striking accomplishment has been the development of polyamine curing systems which give greatly improved resistance to aircraft fluids such as diester lubricants at temperatures as high as 350°F, better compression set and other mechanical properties, and greatly improved resistance to nitric acid and to alkali.

Fundamental studies of the copolymerization behavior of the fluoroacrylates, of their swelling in organic liquids, and of their molecular weights are presented.

Purdue Research Foundation. TR 52-191, Part 2.
FLUORINE-CONTAINING ELASTOMERS. O. R. Pierce, E. T. McBee. AF 33(038)-20581. October 1953. PB 128531. Order from LC, Mi $3.60, Ph $9.30.

The nature of the work described in this report comprises investigations of the synthesis of three types of fluorine-containing alkylsilanes:

I. $(C_3F_7CH_2CH_2)_n SiX_{4-n}$
II. $(C_3F_7CH_2)_n SiX_{4-n}$

III. $(C_3F_7)_n SiX_{4-n}$
X=Cl or OEt

Type I. was prepared from the corresponding Grignard reagent and samples of two materials, $(C_3F_7CH_2CH_2)Si(OEt)_3$ and $(C_3F_7CH_2CH_2)_2Si(OEt)_2$ were submitted to the Materials Laboratory for polymerization studies.

The synthesis of type II. was planned from the reaction of the corresponding fluorine-containing Grignard reagent or lithium reagent with silicon tetrachloride or tetraethyl silicate. The organometallic could not be prepared in good yield and the desired silanes were not obtained. Experimental details of the attempted synthesis are given.

The synthesis of type III. was attempted from the reaction of either C_3F_7MgI or C_3F_7Li with silicon tetrachloride or tetraethyl silicate. The reaction products were found to be alkylsilanes but could not be identified conclusively.

TR 53-223.
DEVELOPMENT OF LOW TEMPERATURE FUNGUS RESISTANT VINYL COMPOUNDS. Wallace W. Jackson. January 1954. PB 111929. Order from OTS $0.50.

Efforts to develop a polyvinyl chloride plastic tubing conforming to and excelling the requirements of Specification MIL-I-7444 (Insulation, Sleeving, Electrical, Flexible) are described. This specification requires a combination of low temperature characteristics, fungus resistance and dielectric properties which commercial compounders have found difficult to obtain.

During this development a revised edition (MIL-I-7444-A) of this specification was made available. This revision requires flexibility at minus 90°F, but relaxes some of the other difficult requirements which were found to be unnecessary for most service applications.

Variation in compounding procedure and technique however, using a wide range of commercially available and experimental fungicides, plasticizers and stabilizers with a polyvinyl chloride/polyvinylidene chloride copolymer, resulted in a tubing compound exhibiting overall properties superior to those in the original version of Specification MIL-I-7444.

The Connecticut Hard Rubber Company.
TR 53-410.
INVESTIGATION AND DEVELOPMENT OF LOW-TEMPERATURE ADHESIVE TAPE. H. N. Homeyer, Jr., J. H. Preston. AF 33(600)-16213. January 1954.

This report describes work on the investigation and development of pressure-sensitive adhesives for application and use at sub-zero temperatures such as are encountered in certain aircraft and cold weather zone operations.

The research was carried out by personnel of the Connecticut Hard Rubber Company at New Haven, Connecticut during the period 9 April 1952 - 9 August 1953, under United States Air Force Research Contract AF 33(600)-16213.

The final product resulting from this work was twenty-five 60-yard rolls of two inch wide pressure-sensitive adhesive tape, meeting or exceeding all of the requirements of JAN-P-127 Type I, Grade A, and in addition being applicable and retaining adhesion down to -80°F. This tape has been forwarded to Wright Air Development Center.

The low temperature pressure-sensitive adhesive tape consists of silicone rubber adhesive compound, coated on a butyl rubber covered cotton print cloth backing.

Phillips Petroleum Company. TR 53-281.
PLASTICIZERS FOR OIL-RESISTANT RUBBER. AF 33(616)-59. March 1954. PB 121075. Order from OTS $0.75.

A number of experimental plasticizers were prepared and evaluated as non-extractable plasticizers in oil-resistant rubber for service at low temperatures. These included n-formyl morpholine; diethylene glycol bis (morpholinylformate); diethylene glycol dicarbonate esters of methyl, ethyl, and butyl Carbitol; dibutyl and diisobutyl sebacate; bis (Methoxy Polyethylene Glycol 350) adipate; bis (2-methoxy-2-bromoethyl) phosphonate; formals prepared from ethyl Carbitol, Ethoxy Triglycol, the monoethyl ether of Polyethylene Glycol 200 and the mixed formal from Methoxy Polyethylene Glycol 350 and n-hexyl Carbitol; and the product from the reaction of butadiene monoxide with phosphorus pentoxide. A number of these experimental plasticizers imparted fairly good low temperature properties to Paracril B. Di-(Ethoxy Triglycol) formal was superior to Thiokol Corporation's TP-90B. However, none of these plasticizers combined the ability to impart low temperature properties with non-extractability to the degree desired. Of the plasticizers tested only n-formyl morpholine imparted fair low temperature properties combined with fair oil-resistance.

Thiokol Corporation's liquid polysulfide, ZL-109, was evaluated in combination with TP-90B. In compounding studies this material exhibited a strong tendency to scorch, but it was found that sulfur prevented the scorching and Altax retarded the cure. Promising polymers resulted from the addition of ZL-109 to a 70/30 butadiene-acrylonitrile latex.

A number of 1,3-butadiene-1-cyano-1,3-butadiene copolymers were prepared and compared to Paracril B both with and without TP-90B and liquid polybutadiene as plasticizers. It was found that these experimental stocks were considerably inferior to Paracril B but had somewhat better low temperature properties. Temperature retraction and cold compression set data indicate a strong tendency for the cyanobutadiene stocks to crystallize.

United States Rubber Company. TR 54-68.
DEVELOPMENT OF AN IMPROVED IN-FLIGHT REFUELING HOSE. Richard J. Meisinger, Stanley L. Bertholf and Frank Lichtey. AF 33(616)-386. May 1954.

A laboratory study was made to determine what type of plastic material would be suitable as a seamless tube for Aircraft In-flight Refueling Hose.

Of the various materials evaluated, FM-6901 type Nylon was chosen as the most suitable. A sample length of hose was manufactured using this material as an inner liner. The hose carcass used is identical to that now being furnished by the United States Rubber Company in 2-1/4" MIL-H-4495-A (ASG), Rubber, In-flight Refueling Hose which this company is qualified to manufacture. A nylon tube was extruded and incorporated into the carcass using special techniques developed for this purpose.

The resultant hose sample was subjected to the low temperature test required by the contract and failed to meet requirements. The nylon liner cracked during bending.

It is concluded from this work that even though Nylon FM-6901 in strip specimens is flexible at -65°F it is unsatisfactory at this temperature when used in a hose.

Coast Pro-Seal and Manufacturing Company
TR 53-450.
DEVELOPMENT OF INTEGRAL FUEL TANK SEALANT COMPOUND. John M. Snider and Frank Hirosawa. AF 33(600)-19202. May 1954. PB 122475. Order from LC, Mi $7.20, Ph $22.80.

This work was undertaken to develop materials other than polysulfide polymers for use in fuel tank sealant applications. Materials for use in such applications preferably should be viscous liquid polymers which may be cured to an elastomeric state under ambient conditions after application.

Liquid polymers were prepared by the bulk copolymerization of acrylic esters and allyl glycidyl ether. These polymers could be cured to elastomeric products by the reaction of amine curing agents with epoxy groups. The cured products generally had good high temperature properties but poor low temperature flexibility. Resistance to aromatic fuels was not as good as would be desired.

Liquid polymers of a polyester type were prepared from mercaptoacetic acid. These polymers could be made sensitive to oxidation curing reactions by a process of heat treatment. The cured polymers have excellent solvent and water resistance. The curing reaction is so rapid that it is difficult to mix the curing agent with the polymer and it will be necessary to develop suitable methods for retarding the reaction.

General Tire & Rubber Company.
WADC TR 54-62.
BUTYL INNER TUBE COMPOUND FOR AIRCRAFT TIRES. Emmett B. Reinbold. AF 33(600)-22796. July 1954.

This report deals with the development of a butyl rubber compound with requisite physical properties for fabrication into inner tubes for aircraft tires.

A large number of plasticizers of widely divergent chemical characteristics were evaluated, with special emphasis placed on their low temperature properties in butyl rubber and including the technique of using high black, high plasticizer with a high viscosity elastomer. The effect of zinc oxide content in the formula was determined. Carbon blacks of all commercially available types were compared and the effect of the various blacks on physical properties, including low temperature characteristics were determined. On blacks producing highest tensile values, series of tests were made to determine the loading which produces the maximum physical properties. A comparison of low temperature properties is made of the available commercial butyl rubbers. An extensive investigation of an outstanding low temperature material, Silicone, was carried out.

On plasticizers which produced adequate low temperature flexibility in butyl rubber a study is made on volatility and migration of the plasticizer from cured compound and then low temperature properties determined following migration treatment of cured tensile sheets. In all cases, plasticizers which produced the target low temperature requirements showed poor low temperature properties following migration treatment.

The effect of inorganic acceleration and of the recently recommended processing technique of high temperature mixing was investigated.

WADC TR 54-213.
ELASTOMERIC FLUOROALKYL SILOXANE COPOLYMERS. Roy T. Clark, Jr. July 1954. ASTIA Document No. AD 47085. PB 129594. Order from LC, Mi $2.70, Ph $4.80.

Preparation of fluorine-containing silicone heteropolymers has been accomplished on an experimental scale. Specifically, elastomeric copolymers have been prepared of dimethyl dichlorosilane with bis 3,3,4,4,5,5,5 heptafluoropentyl diethoxysilane, 3,3,4,4,5,5,5, heptafluoropentyl methyl diethoxysilane and bis 3,3,3 trifluoropropyl dimethoxysilane. Copolymerization was realized both from the oil resulting from the cohydrolysis of these compounds and by polymerizing a blend of the separately hydrolyzed materials.

These copolymers all exhibited improved resistance to the swelling action by fuels and retained other desirable physical properties to a large degree when compared to methyl silicone rubber prepared by the same method. Although these elastomers, like commercial silicone rubber, had rather low tensile strength when in the swelled condition, they had considerably greater stability in diester oil at elevated temperature than either methyl silicone rubber prepared in the same manner or the commercially available polymers; the fluorinated elastomers retain over 50% of their tensile strength after 24 hours in diester fluid at 400°F, whereas the unfluorinated materials dissolve at approximately 350°F. Attempts to prepare fluorine-containing silicone homopolymers have been unsuccessful.

WADC TR 54-318.
DEVELOPMENT OF A RUBBER FOR HIGH TEMPERATURE SERVICE IN CONTACT WITH EXPERIMENTAL HYDRAULIC FLUIDS. Frederick G. Kitts. August 1954. PB 111766. Order from OTS $0.50.

The proposed operation of hydraulic systems at temperatures in excess of 500°F made necessary the development of a rubber for packings, gaskets, hose, etc., which would withstand newly developed fluids at system temperatures; the use of standard packings was proven to be impracticable.

Various commercial and experimental polymers were evaluated at elevated temperatures and ethyl acrylate copolymers were found to have the best inherent resistance to the fluids at high temperatures. Compounding studies were made on two such copolymers with particular emphasis on curing systems and fillers.

A compound was developed which laboratory tests indicate is satisfactory. "O" ring seals have been fabricated of this material and are now awaiting evaluation in actual hydraulic system components and mock-ups. It is anticipated that satisfactory hose can also be fabricated from this compound.

Thiokol Chemical Corporation. WADC TR 54-10.
FUEL CELL SEALANT COMPOUNDS. Earl H. Sorg, John P. Bowen, Edward M. Fettes, Joseph S. Jorczak. AF 33(038)-30523. October 1954.

This work was undertaken by the Thiokol Chemical Corporation to develop integral fuel tank sealant compounds with improved properties.

The hexamethylene terpolymer sealant compound, prepared and submitted to Wright Field at the end of the first year's work on this contract showed better heat resistance coupled with greatly improved low temperature properties than a comparable IP-2 compound. The 0.5 mole % of TCP and 2 mole % of TCF cross-linked pentamethylene formal copolymers showed better heat and aromatic fuel resistance than the 2 mole % of TCP cross-linked copolymers previously submitted.

Compounding studies showed that IP-32 should be more suitable than IP-2 compounds since lower moduli and higher ultimate elongation before and after heat aging and after immersion in aromatic fuel were attained.

Five two-package mix experimental translucent sealant compounds prepared from the liquid polysulfide/epoxy resin system and submitted to Wright Field were tough and resistant to jet fuel but did not pass all the Air Force specifications. However, the compounds displayed promising potentialities.

The IP-2 base experimental translucent sealant compound submitted to Wright Field for evaluation showed good potentialities as an improved sealant and was more

transparent during and after application and cure than an IP-2 base commercial sealant compound.

Many of the materials tested in this investigation were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of the material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

United States Rubber Company. WADC TR 53-372. HEAT-RESISTANT AIR HOSE, HEAT-RESISTANT FUEL AND OIL HOSE, AND HIGH-PRESSURE FUEL HOSE. Richard J. Meisinger, Theodore D. Ernst. AF 33(038)-22905. November 1954.

Two hoses, a braided type and a fabric type, were developed for use with compressor bleed air at an ambient temperature of 500°F. These hoses are designed to operate at 100 psi and meet the collapse resistance requirements set forth in Specification MIL-H-6000 and flexibility requirements equivalent to Specification MIL-H-5511.

A heat resistant fuel and oil hose equivalent to Specification MIL-H-5511 was developed. This hose is designed to handle MIL-L-7808 type oil at an ambient temperature of 350°F.

Experimental work was conducted to develop a fuel hose equivalent to Specification MIL-H-5511 hose except to meet higher operating pressure requirements. No hose was developed to meet these requirements.

Phillips Petroleum Company.
WADC TR 53-281 Part 2.
PLASTICIZERS FOR OIL-RESISTANT RUBBER. Charles S. Imig. AF 33(616)-59. November 1954. PB 121076. Order from OTS. $1.25.

Various compounds either purchased or synthesized were evaluated as low temperature, non-extractable plasticizers for oil-resistant polymers such as Paracril B. These compounds including various carbamates, dithiocarbamates, amides, imides, esters, and several liquid polymers were generally unsatisfactory as low temperature plasticizers for Paracril B. While several of these compounds, especially the polymers, were non-extractable in 70/30 isooctane-toluene and others, particularly N,N-dibutylformamide and N,N-dibutylacetamide, imparted good low temperature properties, none was found which combined the desired characteristics of being non-extractable and imparting good low temperature properties.

Attempts to improve the low temperature properties of 70/30 butadieneacrylonitrile copolymer by the addition of ethyl, butyl or hexyl mercaptans was unsuccessful. It appeared that higher mercaptans might improve the low temperature properties but only at some sacrifice in oil resistance.

It was found that a mixture of Thiokol Corporation liquid polysulfude ZL-109 and TP-90B is an effective plasticizer for Paracril B and imparts fair low temperature properties combined with reduced extractability. However, with stocks containing ZL-109, the scorch time is reduced, the stress-strain properties are impaired, and the cure is retarded. Compounding studies were made to circumvent these deficiencies.

The Connecticut Hard Rubber Company
WADC TR 54-407
RESEARCH AND DEVELOPMENT OF NONMETALLIC ACID-RESISTANT HOSE. Donald C. Dougall, Stephen Casapulla. AF 33(616)-425. November 1954.

This report describes the development and fabrication of a flexible nonmetallic acid-resistant hose for use on type B-2 servicing semi-trailers. The final product of this research was the fabrication of two fifty-foot lengths of hose, consisting of a polytrifluorochloroethylene helical bellows liner, encased in a synthetic rubber tube and a stainless steel abrasion-resistant cover. The hose is capable of resisting the corrosive effects of red and white fuming nitric acid. It will withstand a pressure of 125 psi, will operate at temperatures from -65° to 160°F, and can be used as a suction hose.

The development and research were carried out by The Connecticut Hard Rubber Company of New Haven, Connecticut.

WADC TR 54-458.
DEVELOPMENT OF A RUBBER FOR SERVICE IN CONTACT WITH EXPERIMENTAL HYDRAULIC FLUIDS AT 400°F. Frederick G. Kitts. December 1954. PB 11598. Order from OTS. $0.50.

The operation of hydraulic systems of piloted supersonic aircraft at temperatures in the neighborhood of 400°F will necessitate the replacement of standard AN seals and hose with a more heat and fluid resistant compound. Earlier work had proved Buna N (Butadiene-Acrylonitrile Copolymers) unsatisfactory over the temperature range - 65° to 400°F in the silicate ester type fluids so Neoprene WRT was chosen as the best overall compromise of original and aged physical properties.

A compound of Neoprene WRT was developed which was marginally satisfactory after aging 168 hours in MLO 8200 at 400°F in the absence of air. If air were not at least partially excluded the rubber would reach an unsatisfactory condition in less than 70 hours. It was found that a blend of 85 parts of MLO 8200 and 15 parts of di(2 ethyl hexyl) sebacate with anti-oxidant added to bring the concentration in the blend up to the original concentration in MLO 8200 gave the best balance of aged properties with Neoprene WRT with and without air above the fluid surface.

The compounds of Neoprene WRT are believed to be processable on industrial equipment such as mills, extruders, etc. "O" rings have been fabricated in the Materials Laboratory and are undergoing evaluation testing.

Purdue Univ. WADC TR 52-191 Part 3.
FLUORINE-CONTAINING ELASTOMERS. Earl T. McBee, Carleton W. Roberts. AF 33(038)-20581. December 1954.

An improved stepwise synthesis of bis(3, 3, 4, 4, 5, 5, 5-heptafluoropentyl)diethoxysilane has been developed to give the product an overall yield of 55 percent based on the 3, 3, 4, 4, 5, 5, 5-heptafluoropentylmagnesium bromide.

The one step synthesis of bis(3, 3, 4, 4, 5, 5, 5-heptafluoropentyl)diethoxysilane from 3, 3, 4, 4, 5, 5, 5-heptafluoropentylmagnesium bromide and diethoxydichlorosilane gave less than 48 percent of crude product.

3, 3, 4, 4, 5, 5, 5-heptafluoropentylmethyldiethoxysilane was prepared by the reaction of 3, 3, 4, 4, 5, 5, 5-heptafluoropentylmagnesium bromide with methyltriethoxysilane and by the reaction of methylmagnesium bromide with 3, 3, 4, 4, 5, 5, 5-heptafluoropentyltriethoxysilane.

3, 3, 4, 4, 5, 5, 5-heptafluoro-1-bromopentane was obtained in 39 percent overall yield from heptafluorobutyraldehyde.

Three synthetic routes were investigated for the synthesis of 3, 3, 3-trifluoropropyl halides, the hydrobromination of 3, 3, 3-trifluoropropene, chlorination of 1, 1, 1-trifluoropropane obtained by catalytic reduction of

3, 3, 3-trifluoro-1, 1, 2-trichloropropane, and the best method, the liquid phase chlorination of 1, 1, 1-trifluoropropane from fluorination of 1, 1-dichloro-1-propene. The latter method gave the desired product in an overall yield of 42 percent.

Both 3, 3, 3-trifluoropropylmagnesium bromide and chloride were converted in a stepwise manner in bis (3, 3, 3-trifluoropropyl)dimethoxysilane; the best overall yield based on 3, 3, 3-trifluoropropyl halide was 57 percent.

3, 3, 3-trifluoropropylmethyldimethoxysilane was prepared in 62 percent yield.

Several attempts were made to obtain evidence for the formation of a Grignard reagent or a lithium compound from 1, 1, 1, 2, 2, 3, 3-heptafluoro-4-iodopentane; these were unsuccessful.

Attempts to prepare 2, 2, 3, 3, 4, 4, 4-heptafluoro-1-methylbutylsilanes were unsuccessful.

Synthetic steps leading to 2-methyl-3, 3, 4, 4, 5, 5, 5-heptafluoro-2-iodopentane were stopped at the p-toluenesulfonyl ester of the corresponding alcohol.

Extensive studies have been completed on the synthesis of perfluoropropylsilanes. The exchange reactions between perfluoropropyl iodide and methyllithium and perfluoropropyl iodide and phenylmagnesium bromide have been used to prepare perfluoropropyllithium and perfluoropropylmagnesium bromide. These organo-metallics have been caused to react in ether solvents at varying concentrations and temperatures with silanes containing replaceable halogen or alkoxy groups.

Perfluoropropylsilanes have been successfully prepared using perfluoropropyllithium and several silanes. Perfluoropropylmethylethoxychlorosilane has been prepared in 10 percent yield.

The decomposition of perfluoropropyllithium to give high percentages of perfluoropropene, the azeotroping tendency of the silanes, and the wide spectrum of products obtained from the reactions of perfluoropropyllithium with silanes has made isolation of desired products a demanding research undertaking.

Battelle Memorial Institute. WADC TR 54-190. DEVELOPMENT OF HIGH-TEMPERATURE OIL-RESISTANT RUBBER. William H. Gillen, William J. Mueller, Richard A. Clark, Randall G. Heiligmann. AF 33(616)-476. January 1955. PB 111693. Order from OTS. $3.50.

Research toward the development of rubber compounds, to be used in connection with a diester-type lubricating oil (Turbo Oil-15) for long term exposure at 350° to 550°F, is described in this report. The evaluation of experimental compounds was confined to one temperature (350°F). At this temperature, the most promising results were obtained with compounds prepared from an acrylate polymer, Hycar 4021, compounded with Silene EF. The best composition of this type fell short of the target requirements only because of about 6 percent excessive swelling. Another acrylate-type rubber, Acrylon EA-5, shows about equal promise for this application.

Compounds of a butadiene-acrylonitrile copolymer (Hycar 1001) showed promise, except that they cracked badly when aged in Turbo Oil-15 at 350°F. ELC Magnesia was the best reinforcing agent used with this polymer. Variations in the antic .ant and vulcanizing system provided only slight improvement in crack resistance. The chief weakness of this polymer is the vulnerability of its double bonds to oxidation.

The emphasis of future research on this project will be directed toward compounding acrylate-type rubbers, including poly-1, 1-dihydroperfluorobutyl acrylate, and in seeking methods for protecting the double bonds of acrylonitrile-type rubber.

Minnesota Mining and Manufacturing Company
WADC TR 52-197 Part 4.
SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS. F. A. Bovey. AF 33(038)-515. January 1955.

This report describes the preparation and properties of fluorine-containing rubbers. The object of the work is the development of elastomeric materials which are resistant to the fuels, lubricants and hydraulic fluids used in military aircraft and which are serviceable over the widest possible temperature range.

Of the materials under development, the following appear to be of chief interest:

A. Perfluorobutadiene Copolymers. Copolymers of perfluorobutadiene with 1, 1-dihydroperfluoroalkyl vinyl ethers offer promising high temperature resistance, low swelling in aircraft fluids, and high rsistance to ozone. Difficulties have been experienced in preparing products of consistent quality, and ineffectively vulcanizing and stabilizing the copolymers.

B. Fluoroacrylates. In addition to the 1, 1-dihydroperfluoroalkyl acrylates, which have been described in earlier reports and which have outstanding solvent resistance but limited low temperature flexibility, newer classes of fluoroacrylates have been synthesized. Of particular interest are the ω-(perfluoroalkoxy)-1, 1-dihydroperfluoropropyl acrylates, which offer solvent resistance at least equal to that of the earlier series but are flexible at temperatures 25°C lower than the polymers of the 1, 1-dihydroperfluoroalkyl acrylates.

The mechanism of the polyamine vulcanization reaction has been shown to be amide formation. Extensive testing of vulcanizates of poly-1, 1-dihydroperfluorobutyl acrylate (poly-FBA) prepared in poly-amine curing recipes has indicated excellent resistance to a variety of liquids at high temperature.

A thorough study of the molecular weight and fundamental solution properties of poly-FBA has been carried out.

Rutgers Univ. WADC TR 55-26 Part 1. RESEARCH ON BORON POLYMERS LITERATURE SURVEY. William L. Ruigh, Charles E. Erickson. AF 33(616)2057. March 1955. PB 111689. Order from OTS. $1.50.

A literature search has been made in the field of boron polymers. Polymers derived from boric acid esters are all sensitive to hydrolysis by water as are borazole and its derivatives. Boronamides and N-boro-ureas have been claimed to yield strong fibre and film-forming polymers.

The role of boron compounds in the technology of silicone and other synthetic rubbers is important. The patent literature does not satisfactorily reveal the chemical functions played by boron compounds.

Research has been initiated on the preparation of polymeric boronamides derived from stabilised boronic acids and bifunctional isocyanates.

Battelle Memorial Institute.
WADC TR 54-190 Part 2.
DEVELOPMENT OF HIGH TEMPERATURE OIL RESISTANT RUBBER. William J. Mueller, Louis E. Novy, Richard A. Clark, Randall G. Heiligmann. AF 33(616)-476. April 1955. ASTIA Document No. AD 75797.

This report describes additional research directed toward the development of rubber compounds that can be used in diester-type lubricating oil (Turbo Oil-15) for long

exposures at 350 to 550°F. The evaluation of experimental compounds was confined largely to 350°F, although a few tests were made at 400°F. Studies at Battelle showed that poly-FBA (poly-1,1-dihydroperfluorobutyl acrylate) can be compounded to meet the minimum target requirements of this project. Further work with the acrylate-type rubbers, Hycar 4021 and Acrylon EA-5, showed no substantial gain for this class of rubbers. Hycar 4021 still misses the target requirements only because of a swell that is 6 percent above the 30 percent maximum desired. Acrylon EA-5 appears inferior to Hycar 4021 for this application because of its tendency to crack during hot-oil aging tests. This cracking tendency is unusual, inasmuch as the material is a saturated polymer.

Further attempts have been made to overcome the only real shortcoming of high-nitrile rubbers (such as Hycar 1001) for this application -- cracking after hot-oil aging. Although some short-term improvements have been effected by employing thiols and amines as antioxidants, all such gains have been lost by the end of 500 hours' aging. Methods for eliminating a portion of the polymer double bonds by hydrogenation are under consideration. The double bonds of hydrogen atoms on carbons alpha to the double bond are presumed to be focal points for oxidative degradation.

Two aluminum-block heaters for test-tube aging of rubber samples in oil at temperatures up to 500° and 800°F, respectively, have been designed for WADC under this contract.

In future work on this basic problem, the emphasis will be shifted toward developing rubber compositions for use in synthetic hydraulic oils, such as the silicate esters.

WADC TR 55-57
VULCANIZATION OF RUBBER WITH HIGH-INTENSITY GAMMA RADIATION. Wallace W. Jackson, Denver Hale. April 1955. PB 111675. Order from OTS. $1.00.

Techniques of vulcanizing both natural and synthetic rubber polymers, using high intensity gamma radiation are described.

This process utilizes the gamma radiation from Cobalt 60 or spent uranium reactor fuel elements and is accomplished without heat or chemical vulcanizing agents. Reinforcing agents, anti-oxidants and other additives are still required however, to obtain an optimum balance of physical properties.

After compounding the elastomers in the conventional manner and subjecting them to various dosages of gamma radiation, the resultant physical properties were determined.

Evaluation of this technique shows considerable promise in developing elastomers for specialized uses, such as producing high temperature oil resistant rubber compounds and for improving the compression set of specialized rubber compounds.

This study further emphasizes the practical usefulness of atomic energy in providing the rubber technologist a new experimental variable or tool for determining fundamental properties of elastomeric compounds.

Rutgers Univ. WADC TR 55-26 Part 2.
RESEARCH ON BORON POLYMERS. William L. Ruigh, Frank Gunderloy, Charles E. Erickson, Michael Sedlak. AF 33(616)-2057. May 1955. PB 111892. Order from OTS $2.25.

An exploratory study has been made of certain boron compounds and polymers in connection with the development of plastics and elastomers, which are thermally stable as well as oil and fuel resistant.

The stable polymeric "boronamides" derived from boronic acids and bi-functional isocyanates and claimed in a recent patent have been shown to be essentially polyureas admixed with soluble boron compounds.

Esters and polymers based on tricovalent boron are hydrolytically unstable and therefore this project was directed toward the synthesis of quadricovalent chelate and quasi-chelate boronic acids and borates. The diethanolamine ester of benzene-boronic acid has been prepared. It was stable to hydrolysis, and could be recrystallized from water. Allylboronic acid and its esters have been synthesized and studied primarily as intermediates for o-dimethylaminopropane boronic acid and related compounds which as yet have not been prepared. This work is being continued.

WADC TR 55-117.
ELASTOMERIC DITHIOPOLYESTERS. F. W. Knobloch. June 1955. PB 111949. Order from OTS $0.75.

Elastomers have been prepared from an experimental liquid polymer formulation derived from a condensation product of mercaptoacetic acid and triethylene glycol. Two general types of these elastomers are discussed; one was obtained by reaction of the liquid polymer with organic and inorganic peroxides, the other by reaction of terminal mercapto groups with unsaturated aldehydes.

Selected specimens of each type of elastomer were successfully compounded and vulcanized using conventional rubber processing equipment. The behaviour of these vulcanizates in fuels and synthetic ester base oils has been studied along with the effects of aging at elevated temperatures. The vulcanized dithiopolyesters showed excellent resistance to the swelling action of 70/30 iso-octane/toluene fuel mixtures. Ester base oils of the MIL-L-7808A type were found to cause shrinkage. Elevated temperatures 350°F) consistently brought about rapid deterioration of both vulcanized and raw polymer stocks.

Dow Corning Corporation. WADC TR 55-193.
FLUORINE-CONTAINING POLYETHERS. Ogden R. Pierce, Donald D. Smith, Robert M. Murch. AF 33(616) 2417. June 1955. PB 111986. Order from OTS $1.50.

Appreciable quantities of the two monomers, $CF_3\overbrace{CH\text{-}CH_2O}$ and $CF_3\overbrace{C(CH_3)CH_2O}$ have been prepared by reactions involving the dehydrohalogenation of the corresponding halohydrins.

Dehydrohalogenation studies were conducted using the following halohydrins: $CClF_2CH_2OH$, CCl_2FCH_2OH, $CClF_2CHOHCCl_2F$, $CClF_2CHOHCClF_2$, and $CCl_2FCHOHCCl_2F$. The corresponding epoxides were not obtained but rather extensive decomposition of the organic reactants was observed.

An investigation of the hypochlorination of the olefins, $CH_2{=}CF_2$, $CF_3(CH3)$ a CH_2 and $C_3F_7CH{=}CH_2$, is in progress. The results are inconclusive at this time.

The preparation of a polyglycol formal was accomplished by reaction of a fluorine-containing glycol with dibutyl formal. The material was found to be thermally unstable.

The polymerization of both $CF_3\overbrace{CHCH_2O}$ and $CF_3\overbrace{C(CH_3)CH_2O}$ was accomplished using ferric chloride as a catalyst. In addition, copolymers of these two monomers were prepared in a similar manner.

WADC TR 55-377.
A STUDY AND EVALUATION OF KEL-F ELASTOMER. R. E. Headrick. October 1955. PB 111984. Order from OTS $1.25.

The bulk of the work reported herein is a summary of experimental compounding while trying for low compression set and good chemical resistance.

Initial compounding of Kel-F Elastomer revealed the most promising compounds for low compression set and chemical resistance were those cured with benzoyl peroxide. The compound having the lowest set in this effort was 266-62-1. This compound has a set of 40 percent when compressed 30 percent at 250°F for 70 hours.

Immersion tests in experimental hydraulic fluids composed of silicate esters or "silicone oils" indicate that Kel-F Elastomer may prove useful for aircraft hydraulic system applications up to 400°F. Tests also indicate that this elastomer when properly compounded has exceptional resistance to potential rocket fuels such as fuming nitric acid and may prove useful for hose, seals, protective clothing and other items for contact with these fluids.

Of particular importance to the rubber compounder is the discovery that prolonged milling is required during compounding to obtain uniform physical properties from the compounded elastomer. Variation in milling in time can change final physical properties of the cured elastomer as much as 100 percent, at least on the polymer produced to date.

WADC TR 55-381.
POLY FBA - A FLUORINATED ACRYLIC ELASTOMER FOR HIGH TEMPERATURE SERVICE IN THE PRESENCE OF AIRCRAFT FUELS AND LUBRICANTS. Horace C. Hamlin, Maj. USAF. November 1955. PB 111946. Order from OTS $1.50.

A large number of fluorine containing polymeric systems have been investigated by the Minnesota Mining & Manufacturing Company under Air Force Contract No. 33(038)-515. One of the most successful developments, poly 1, 1 dihydroperfluorobutyl acrylate (poly FBA) exhibits good rubbery characteristics, excellent resistance to many fuels, lubricants, solvents, chemicals, and ozone, plus very good stability at elevated temperatures. Compounding and processing studies have been made, and tests conducted in various media at temperatures up to 550°F. Results of laboratory and simulated service tests show poly FBA to be vulcanizable through the action of certain metal oxides or organic amines, reinforcible with carbon blacks and some inorganic fillers, and readily handled on standard rubber processing equipment. Tensile strength ranges from 1000 to 1400 pounds and elongation from 200 to 400%. It is highly resistant to the effects of hydrocarbon fuels, even at elevated temperatures, and has been successfully tested in the presence of synthetic lubricants for up to 300 hours at 400°F, and for shorter periods at higher temperatures.

Poly FBA is also highly resistant to the chemical action of fuming nitric acid, although quite permeable to it. It is completely resistant to ozone. Low temperature properties are limited to about 0°F unplasticized, although it retains good elasticity almost down to the brittle point.

Battelle Memorial Institute. WADC TR 54-190 Part 3.
DEVELOPMENT OF HIGH-TEMPERATURE OIL-RESISTANT RUBBER. William J. Mueller, Louis E. Novy, Richard A. Clark, Randall G. Heiligmann. AF 33(616)-476. December 1955. PB 121195. Order from OTS $2.50.

This report describes research to develop two types of rubber composition. These were (1) a composition that will withstand 500 to 1000 hours of immersion in diester-type lubricating oil (Turbo Oil-15) at 350 to 400°F, and (2) a composition that will withstand immersion for the same time period in silicate ester-type hydraulic fluids (OS-45 or MLO-8200) at 400°F.

The best compositions for use in hydraulic fluids for an aging time of 168 hours at 400°F were prepared from Neoprene WRT. For this time period, these compositions met nearly all of the WADC minimum target specifications. For longer aging periods, stocks made with Acrylon BA-12 and with Philprene VP-A appeared to be the most promising, although only limited work has been done with these polymers. Hycar 1014 and Philprene VP-25 were less promising polymers than Neoprene WRT, and Hycar 1072 and butyl rubber were completely unsuitable.

Compositions to be used in Turbo Oil-15 were evaluated at both 350 F and 400 F. At 350 F, poly-FBA compositions were able to meet all of the minimum target specifications of this project, and at 400°F they failed only because of low tensile strength after aging (300 to 350 psi, compared with a minimum target of 800 psi). At 350°F, Hycar 4021 stocks missed the minimum target requirements because they swelled about 6 per cent above the 30 per cent maximum. At 400°F, Hycar 4021 stocks had about 25 per cent excessive swell, fell down in stress-strain properties, and cracked after 500 hours of aging. Further efforts to overcome the tendency of Acrylon EA-5 stocks to crack, even when aged at 350°F, were not successful.

Connecticut Hard Rubber Company. WADC TR 55-351.
TESTING OF SILICONE RUBBER AT ELEVATED TEMPERATURES. Aldo J. DeFrancesco. AF 33(616)-2542. January 1956. PB 121192. Order from OTS $1.25.

An apparatus to measure tensile strength, elongation, and tear strength of silicone rubber at elevated temperatures was designed and constructed. Fifteen different silicone compounds were tested at room temperature, 212°F and 400°F.

Although it is known that physical properties of silicone rubber are only slightly affected after the rubber is aged at high temperatures, it was found that they are greatly reduced when measured at high temperatures.

The best tensile strength, elongation and tear strength at 400°F were displayed by Cohrlastic HT Heat Stable (CHR 5908A), a compound containing Valron (DuPont) as the filler. CHR Compound 5901, composed of Dow Corning 6-128 with glass wool as an additional filler, also exhibited good tensile and tear strengths at 400°F.

The least reduction in these properties at 400°F, as compared with results obtained on the silicone compounds at room temperature, was displayed by CHR 5901. Dow Corning Silastic 152, postcured at 600°F, showed the least change in elongation.

Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

Connecticut Hard Rubber Company. WADC TR 55-439.
REINFORCEMENT OF SILICONE RUBBER WITH CARBON BLACK. Aldo J. DeFrancesco. AF 33(616)-2542. February 1956. PB 121231. Order from OTS $1.00.

The silicone polymer, Linde W-96, when filled with various carbon blacks, was successfully cured to form a firm, well-reinforced rubber. Not all carbon blacks, however, are satisfactory. In general, an optimum loading of most acetylene and furnace blacks can be used.

Minnesota Mining and Manufacturing Company. WADC TR 52-197 Pt 6.

SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS. Carl A. Dahlquist. AF 33(038)-515. September 1956. ASTIA Document No. AD 97330. PB 121 762.

This report describes the preparation and properties of fluorine-containing rubbers. The object of the work is the development of elastomeric materials which are resistant to the fuels, lubricants and hydraulic fluids used in military aircraft and which are serviceable over the widest possible temperature range.

Certain fluorine-containing alkoxy acrylate rubbers are superior to poly-FBA in serviceability at low temperature, and equal to poly-FBA in resistance to fuels and hydraulic fluids. The alkoxy acrylate rubbers which have been investigated are poly-FMFPA (poly-3 perfluoromethoxy-1,1-dihydroperfluoropropyl acrylate), and poly-FEFPA (poly-3-perfluoroethoxy, -1,1-dihydroperfluoropropyl acrylate), and poly-H-FEFPA (poly-3(α -hydroperfluoroethoxy)-1,1-dihydroperfluoropropyl acrylate). Poly-H-FEFPA is more sensitive to ketone and ester solvents than the other rubbers mentioned above.

A copolymer of perfluorobutadiene and vinyl 1,1-dihydroperfluorobutyl ether continues to be of interest for its resistance to hot air aging. Good vulcanizates have not been obtained because the copolymer crosslinks badly during polymerization.

Process changes which facilitate production of poly-FBA have been approved.

The amine curing recipe for poly-FBA is still the preferred recipe for best mechanical properties. Attempts to develop latent curatives for compounds with better bin life than the TETA recipe have been unsuccessful.

Vulcanizates of blends of poly-FBA and Linde silicone rubber W-96 containing 50% or less poly-FBA exclusive of fillers and curatives have excellent low temperature flexibility, low volume swell in phosphate ester hydraulic fluids, and good resistance to hot air aging.

Surface esterified silica (Valron) has given excellent reinforcement of poly-FBA, but is no longer commercially available. Valron filled stock is far less permeable to Freon 22 than black filled stock.

Boeing Airplane Company. WADC TR 56-272.

DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS. Frank W. Tipton. AF 33(616)-2867. November 1956. ASTIA Document No. AD 110598. PB 121 898.

In order to fully utilize materials for seal design, a knowledge of the relationship between the physical properties of the materials and sealing efficiency is required. This report includes a literature survey on O-rings and seal design and functional tests of O-rings prepared from rubber compounded to have various physical properties. No definite relationship between seal life and physical properties was found. The inability of the O-ring to maintain sufficient internal pressure and to withstand mechanical conditions are believed to be the two reasons for seal failure.

Connecticut Hard Rubber Company. WADC TR 56-351.

EFFECT OF POTENTIAL ROCKET FUELS AND OXIDIZERS ON ELASTOMERS AND DEVELOPMENT OF ELASTOMERIC COMPOUNDS SUITABLE FOR RETENTION OF THESE MATERIALS. C. J. Maloney and A. S. Kidwell. AF 33(616)-2962. November 1956. ASTIA Document No. AD 110511.

Kel-F Elastomer was selected on the basis of screening tests as the elastomeric material showing the best overall resistance to red fuming nitric acid and hydrocarbon rocket fuels. Blends of polyisobutylene and polyethylene, while not true elastomers, showed excellent resistance to immersion in and penetration of red fuming nitric acid; these blends also showed good resistance to n-propyl nitrate, ethylene oxide, and uns-dimethylhydrazine, but were not suitable for exposure to JP-4 fuel. Poly-FBA was fairly resistant to attack by the acid, although it swelled considerably in the acid and was relatively permeable to it. Both Kel-F Elastomer and Poly-FBA were little affected by JP-4 fuel at room temperature or at 160°F.

Kel-F Elastomer was selected for further work under the second phase of the program. Compounds cured with benzoyl peroxide or p-chlorobenzoyl peroxide far exceeded those cured with amines or isocyanates in resistance to red fuming nitric acid; optimum peroxide concentrations found were considerably lower than those normally used, reducing undesirable by-products and minimizing blistering and delamination problems in fabrication. Of the fillers tested, LM-3 silicone-coated Hi-Sil C showed the best reinforcement; others showed promise as extenders. Kel-F Elastomer was not suitable for use with n-propyl nitrate, ethylene oxide, or uns-dimethylhydrazine.

A modification of the standard H-cell permeability test showed good correlation between the water-pH values and actual metal corrosion.

RFNA with hydrogen fluoride additive showed noticeably greater effect on the various elastomers than straight RFNA.

Firestone Tire & Rubber Company. WADC TR 56-331.

DEVELOPMENT OF HIGH TEMPERATURE RESISTANT RUBBER COMPOUNDS. Floyd M. Smith, Thomas F. Lavery, Robert A. Hayes, Leland J. Kitchen and Sydney Smith. AF 33(616)-3108. December 1956. ASTIA Document No. AD 110643. PB 131 923. Order from OTS $4.00.

Commercially available elastomers were compounded for high temperature performance and tested for resistance to 8-hr heat-aging up to 550°F, with the object of selecting elastomers that might prove suitable at 350-550°F for use in tires and other aircraft component parts.

Silicone rubber (Linde W-96 Silicone) and Kel-F Elastomer vulcanizates were outstanding in heat-aging resistance, requiring temperatures of 480°F and 450°F, respectively, to produce a 25% reduction in room-temperature tensile strength after 8 hrs. of aging. The aging temperatures at which other elastomers retained 75% of their strengths when tested at room temperature after the 8-hr. aging are as follows: Acrylon EA-5, 425°F; Hycar 4021, 395°F; Fluoro-Rubber 1F4, 385°F; Hypalon and polyurethane, 350°F; Butaprene NL (nitrile rubber), 340°F; butyl, 335°F; Neoprene Type WRT, 325°F; Thiokol ST, 285°F; GR-S, 275°F; carboxy rubber, 220°F; and Hevea, 215°F.

The elevated temperatures at which some unaged elastomers retained 50% of their original room-temperature strengths are: butyl, 330°F; DC-401 Silicone, 323°F; Hevea and Acrylon EA-5, 277°F; Hycar 4021 and Thiokol ST, 255°F.

To obtain the above tensile strength information it was necessary to modify an L-6 Scott Tester, develop a suitable source of heat and temperature controls and determine the optimum techniques for obtaining reproducible results. A special permeameter was developed for determining the permeability to air and other gases at elevated temperatures, and air permeabilities were measured at 176°F and 250°F. High-temperature hardness and resilience measurements also were made.

New polymers of high thermal stability were synthesized by extending polyester chains with EPON resin, and also with 1,2,3,4-butanetetracarboxylic acid

dianhydride. A special polyurethane rubber was developed which was thermally stable up to 350-400°F. This is about 50°F higher than for conventional polyurethane-type rubbers.

Coast Pro-Seal and Manufacturing Company.
WADC TR 56-155.
HIGH TEMPERATURE RESISTANT SEALANT MATERIALS. Leonard C. Boller, John M. Snider, John H. Emigh, Wendell Olson and Frank Hirosawa. AF 33(616)-2767. December 1956. ASTIA Document No. AD 110633. PB 121 911. Order from OTS $1.75.

This project was undertaken by the Coast Pro-Seal and Manufacturing Company to develop fuel tank sealant compounds capable of withstanding fuel vapor temperatures of 540°F and liquid fuel temperatures of 380°F for a limited period of time.

A formula was developed for a sealant compound which was unaffected by JP-5 jet fuel under these conditions, retained flexibility and adhesion and withstood proof testing under flexing and pressure at the liquid and vapor fuel temperatures required.

A study was made of the thermal stability of the various commercially available types of butadiene-acrylonitrile polymers and combinations of them with various commercial phenolic resins. Studies were made with added antioxidants and in inert atmospheres.

Some preliminary work was started with coatings based on aqueous dispersions of polytetrafluoroethylene.

Test procedures were developed using the Parr high pressure apparatus for liquid fuel at 380°F and fuel vapor at 540°F. Procedures and apparatus were developed for proof testing coatings under conditions of flexing under pressure at these temperatures.

Wyandotte Chemicals Corporation.
WADC TR 55-492.
DEVELOPMENT AND PHYSICAL TESTING OF ELASTOMERIC COMPOUNDS RESISTANT TO PETROLEUM BASE FUELS AT ELEVATED TEMPERATURES. Edward J. Fujiwara, Theodore L. Eriksson, Loren C. Smith, Wayne G. Lajines and Anthony P. Tochman. AF 33(616)-2779. March 1957. ASTIA Document No. AD 118145. PB 131 029.

This report describes research directed toward the development of rubber compounds resistant to various petroleum base fuels for long exposures (500 hours) at elevated temperatures (250 to 400°F.). At the beginning of the program, no apparatus was available which was capable of performing tests of tensile strength and elongation in the presence of the test fuels at the elevated temperatures. Consequently, a screening test was adapted to test candidate materials after exposure to the test fuels at elevated temperatures. This work was performed concurrently with the design of high temperature-total immersion test apparatus.

Initial screening tests on elastomer samples submitted by suppliers showed that target physical properties after exposure in test fuels for 500 hours at 400°F. could not be met. However, the initial data indicated that compounded materials based on fluorinated raw gums could possibly yield satisfactory products. Initial compounding and curing studies with polymers of Hycars 1001 and 4021 and subsequent screening tests on the resulting materials, to date, indicate that only marginal improvements can be made.

Two apparatuses capable of performing tests of tensile strength and elongation on elastomer samples, in O-ring form, in the presence of test fuels at elevated temperatures, have been designed. A single sample tester (designated Type I) has been constructed. The design and necessary modification designs and the engineering and detail drawings of a multi-sample tester (designated Type II) have been completed. This tester is ready for immediate construction.

In future work on this problem initial major effort will be directed to the construction of the Type II Tester and necessary refinement work on both types of testers. Concurrently, studies on the development of rubber compositions based on fluorinated raw gums will be initiated with eventual shift of emphasis to this work. Other materials, both polymers and test liquids, will be considered as indicated and as requested by Wright Air Development Center.

B. F. Goodrich Company Research Center.
WADC TR 55-58 Pt III.
A STUDY OF THE EFFECTS OF NUCLEAR RADIATION ON ELASTOMERIC COMPOUNDS AND COMPOUNDING MATERIALS. John W. Born, Dwain E. Diller and Eugene H. Rowe. AF 33(616)-2308. June 1957. ASTIA Document No. AD 130803. PB 136 237.

The effort to define, understand, and prevent radiation damage to rubber continued during 1956. Research followed four main lines: the development of Anti-Rads to prevent radiation damage; infrared and mass spectral analyses to relate radiation stability to molecular structures of elastomers; stress relaxation studies to define the mechanisms of radiation damage; and measurements of dynamic mechanical properties of representative rubber compounds after irradiation. Emphasis upon applied testing of end-item materials increased.

The best Anti-Rad extended the retention of tensile strength and ultimate elongation by natural rubber tread stock more than tenfold. The screening study is providing criteria for the selection of superior Anti-Rads. A new technique of mass spectral analysis promises to aid greatly in determining what molecular structural features impart radiation stability. Stress relaxation measurements detail the important role of oxygen in radiation damage. Dynamic test data are given for eight representative rubber compounds.

WADC TR 57-247.
THE MICRO-COMPOUNDING AND EVALUATION OF RUBBER-LIKE POLYMERS. E. G. Schwarz, Lt. July 1957. ASTIA Document No. AD 130899. PB 131 393.

A micro-compounding technique has been developed to process the "research sized" samples resulting from Air Force and Industrial polymer development programs.

Scaled down equipment and testing procedures have also been developed successfully to evaluate the small gram-size samples of vulcanized elastomers. Much of the insight to compounding and vulcanizing these experimental polymers is based upon the chemistry of the system, supplemented by experience and common sense.

Using the technique described in this report, experimental polymers can be evaluated and the decision made to reject or further develop the polymer system--with good assurance that a promising polymer has not been cast aside and that further effort on others is justified.

Boeing Airplane Company. WADC TR 56-272 Pt II. DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS. George E. Trepus. AF 33(616)-2867. September 1957. ASTIA Document No. AD 131094. PB 131 510.

This study is to gain knowledge of the relationship between the physical properties of seal materials and sealing efficiency so that materials may be fully utilized in seal design. The literature survey on O-rings and

seal design has been continued. Commercially available polymers have been compounded with various physical properties. Static annulus (with varying groove configuration), rotating shaft, and reciprocating shaft functional test jigs have been designed and manufactured. Seal tests, under various environmental and mechanical conditions, have been run in these jigs.

No definite relationship between physical properties and seal efficiency was found. A general relationship, however, was evident between seal life, and compression modulus and compression relaxation.

Coast Pro-Seal and Manufacturing Company. WADC TR 56-155 Pt II.
HIGH TEMPERATURE RESISTANT SEALANT MATERIALS. Leonard C. Boller, Gene M. LeFave, Edward O'Brien, Arthur Milner and John H. Emigh. Sept. 1957. AF 33 (616)-2767. ASTIA Document No. AD 131091. PB 131 478.

This project was undertaken by the Coast Pro-Seal and Manufacturing Company originally to develop fuel tank sealant compounds capable of withstanding fuel vapor temperatures of 540°F and liquid fuel temperatures of 380°F for a limited period of time. These requirements have been essentially fulfilled and the results have been presented in WADC Technical Report No. 56-155.

Under an extension of the above contract, No. S1 (56-773) covered in this report, the previous temperature requirements were made inapplicable and a higher temperature (700°F) has been substituted for the 380°F and 540°F temperatures.

While the 700°F requirement was not attained, a substantial increase has been accomplished. A formula was developed for a sealant compound which was unaffected by JP-5 jet fuel in the 600°F to 625°F range It retained flexibility and adhesion and withatood proof testing under flexing and pressure in the temperature range indicated. It is clear from the test data that operation in the presence of fuel vapor is possible in the neighborhood of 650°F.

A thorough formulation study was made of the butadieneacrylonitrile rubber and phenolic resin system in order to develop the optimum thermal stability potential. The effect of added antioxidants, leafing pigments, plasticizers and certain other materials was determined.

Several other coating systems were briefly evaluated, such as the liquid polybutadiene modifications.

Wyandotte Chemicals Corporation. WADC TR 55-492 Pt II.
DEVELOPMENT AND PHYSICAL TESTING OF ELASTOMERIC COMPOUNDS RESISTANT TO PETROLEUM BASE FUELS AT ELEVATED TEMPERATURES. Loren C. Smith, Theodore L. Eriksson, Wayne G. Lajiness and Brock G. Peacock. AF 33(616)-2779. October 1957. ASTIA Document No. AD 142024.

This report describes continued research directed toward the development of rubber compounds resistant to various petroleum base fuels for long exposures (500 hours) at elevated temperatures (up to 400°F.).

The test tube portion of the Type I Tester was redesigned and this tester was used to evaluate several candidate elastomers in the presence of test petroleum base fuels under simulated use conditions of elevated temperature and positive pressure.

The fabrication and construction of various parts of the multisample tester (Type II) was completed and its assembly initiated.

New selected candidate elastomers of the perfluorinated type were screened.

Compound and curing studies with the Kel-F elastomers were initiated and are continuing.

Southern Research Institute. WADC TR 57-364.
RESEARCH ON MATERIALS SUITABLE FOR FABRICATION INTO V51R DESIGN EAR PLUGS. Edgar A. Verchot and Arthur E. Raeuber. AF 33(616)-3488. October 1957. ASTIA Document No. AD 142051. PB 131 656.

This report describes research and development work directed toward finding an acceptable formulation for V51R design ear plugs and toward setting up test methods so that future lots of purchased ear plugs will equal the acceptable formulation.

A test procedure to measure the collapsibility of a medium-size ear plug has been developed. Collapsibility measurements were used to define the properties of ear plugs acceptable to those experienced in the art of using and fitting ear plugs to military personnel.

The ear plug formulations studied were also evaluated with respect to tensile strength, resistance to low temperatures, resistance to lanolin and to detergents, color, surface finish , and resistance to accelerated aging.

Three ear plug formulations were selected as being the most acceptable according to the results of the tests made. The properties of these formulations have been used as the basis for suggested changes in the specifications given in Military Medical Purchase Description Number 4, dated 16 August 1954.

It is recommended that ear plugs based on these formulations be manufactured and tested in field use before further minor adjustments in formulation are attempted.

Minnesota Mining and Manufacturing Company. WADC TR 52-197 Pt VII.
SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS. Carl A. Dahlquist. AF 33(038)-515. October 1957. ASTIA Document No. AD 142027.

Twenty pounds of the polymer of perfluoromethoxy 1, 1 dihydroperfluoropropyl acrylate were prepared for Wright Air Development Center.

A copolymer of 3(ω-chloroperfluoroethoxy) 1, 1 dihydroperfluoropropyl acrylate and its ω-hydro analog has shown some promise as a heat, solvent, and low temperature resistant rubber.

Fluorine-containing silanes ($R_fCH_2CH_2Si(CH_3)(OC_2H_5)_2$) were polymerized to low molecular weight oils (probably the cyclic tetramer), but attempts to polymerize the oils to high molecular weight polymers did not succeed. Rubbery copolymers of each of these silanes and dimethyl dichlorosilane have been prepared, but no useful vulcanizates have been obtained. Fluorine-containing polysiloxane oils having $-Si(CH_3)_3$ end groups have been prepared.

Flexonics Corporation. WADC TR 57-346.
THE DEVELOPMENT OF A HOSE AND HOSE END FITTING FOR POLY-FBA SYNTHETIC RUBBER. John P. Thomas. AF 33(600)-29001. November 1957. ASTIA Document No. AD 142156.

Poly-FBA can be processed with conventional rubber equipment modified to suit its peculiar properties in processing.

Poly-FBA is limited in its use as a hose material to lip seal fittings, because of its cold flow properties even at temperatures of 300°F.

Minnesota Mining and Manufacturing Company. WADC TR 52-197 Pt VIII.
SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS. Carl L. Sandberg. AF 33(038)-515. November 1957. ASTIA Document No. AD 142114.

Further evaluation of 3(ω-chloroperfluoroethoxy)-1,1-dihydroperfluoropropyl acrylate has shown it to be comparable to other perfluoro alkoxy acrylates in low temperature properties and resistance to aromatic solvents and hot air aging, but somewhat poorer in resistance to oxygenated solvents and to diester lubricants.

Attempts to homopolymerize 1,2-epoxyperfluoroheptane with peroxide catalysts or with a ferric chloride: propylene oxide catalyst have given mainly reaction products and little evidence of polymerization. Attempts to copolymerize 1,2-epoxyperfluoroheptane with propylene oxide, using a ferric chloride:propylene oxide catalyst, have given viscous liquid products which may be copolymers.

Exploratory copolymerizations of perfluoro-2-azapropene with vinyl-type monomers have given products containing small amounts of the azapropene.

Connecticut Hard Rubber Company.
WADC TR 57-651.
DEVELOPMENT OF RUBBERLIKE MATERIALS FOR APPLICATIONS INVOLVING CONTACT WITH LIQUID ROCKET PROPELLANTS. John H. Baldrige. AF 33 (616)-5931. December 1957. ASTIA Document No. AD 142205.

A survey of available knowledge concerning the resistance of elastomers and plastics to a number of propellants has been prepared and will appear as WADC TR 57-472.

Elastomeric materials showing satisfactory resistance during an immersion period of seven days at 160°F have been developed for the following propellants: unsymmetrical dimethyl hydrazine, ethylene oxide, n-propyl nitrate, and anhydrous ammonia. Compounds were tested which were resistant to 90 percent hydrogen peroxide at room temperature.

No elastomeric materials were found with satisfactory room temperature resistance to inhibited red fuming nitric acid or to JP-X fuel mixture.

United States Rubber Company. WADC TR 57-553.
DEVELOPMENT OF REFUELING HOSE & COUPLINGS. Stanley L. Bertholf, Leonard Castelbaum and Dante E. Piccoli. AF 33(600)-27745. January 1958. ASTIA Document No. AD 142281.

Development of two basic types of 3" I.D. inflight refueling hose, wire woven type and wire braided type, along with suitable end fittings was the object of this development contract. All work pertaining to hose was performed by United States Rubber Company, at Passaic, New Jersey. Coupling design work was performed by The Weatherhead Company of Cleveland, Ohio, and the Flex-O-Tube Division of Flexonics, Inc., of Detroit, Michigan.

Three different constructions of wire braid hose were developed. The wire woven type hose was eliminated because this type of hose exhibited excessive elongation under load. A total of 35 lengths of wire braided hose assemblies coupled with swaged fittings were furnished under the contract.

Firestone Tire & Rubber Company.
WADC TR 56-331 Pt II.
DEVELOPMENT OF HIGH TEMPERATURE RESISTANT RUBBER COMPOUNDS. Robert A. Hayes, Floyd M. Smith, Ward A. Smith and Leland J. Kitchen. AF 33(616)-3953. February 1958. ASTIA Document No. AD 151003. PB 131 924.

Elastomers of potential interest for high-temperature aircraft tires -- including butyl, silicone rubber, "heat-stable" polyurethane, and Viton A -- were studied to determine tensile, wire-cord adhesion, and air-permeability properties at temperatures ranging up to 400° or 500°F, and properties of interest in tire development, such as internal friction and running temperature. Most promising wire adhesions were obtained with silicone rubber, which retained 63% of the 75°-F adhesive strength at 450°F, compared with 9% for Hevea. Silicone rubber is the most highly permeable to air; Thiokol ST, Vyram and butyl have the lowest air permeabilities. Nitrogen appears to be the most promising of the unreactive gases to use for inflation of high-temperature tires.

Type of sulfur crosslink (mono-, di-, or tetrasulfide) had little effect on high-temperature properties of Hevea. Gamma-irradiation of vulcanized Hevea improved the high-temperature tensile strength. Balata had lower tensile strength than Hevea over the entire range of 73°F - 400°F. Vyram N-7700 was about equivalent to Vyram N-5400. Polyether-type polyurethane had 400°-F tensile strength of 60 psi vs. 515 for the "heat-stable" polyester type. Teflon resin had a tensile strength of 3825 psi at 73°F and 980 psi at 600°F.

Studies on effects of a 4-hour heat-aging at 400°F upon burst pressures of a nylon aircraft tire and of a wire tire are described. It is concluded that a 400°-F tire will require wire tirecord in place of organic fabrics used in present tires.

A prototype aircraft tire successfully was constructed from silicone rubber and steel wire tirecord. Coating the wire tirecord and the bead wire with a thin layer of partially cured silicone rubber facilitated fabrication of the beads and plies.

Experimental butadiene and acrylic polymers containing functional groups were prepared and their high-temperature properties were determined. Carboxyl as the functional group gave better results than hydroxyl or epoxy. Best properties at 400°F resulted from using a metal oxide -- particularly magnesium oxide or cadmium oxide -- with epoxy resin to cure the carboxy polymers, enabling 400°-F tensile strengths as high as 1400 psi to be obtained.

The advantages of the metal oxide cure were obtained with methacrylate rubber (copolymer of butadiene and methyl methacrylate) by curing with barium hydroxide octahydrate, which gave tensile strengths of 4025 psi at 73°F and 960 psi at 400°F when used in combination with bis (α,α'-dimethylbenzyl) peroxide. In this manner the advantageous high-temperature properties of the metal oxide cure were realized with improved processability compared with the carboxy polymers.

It was found that butadiene copolymers can be vulcanized by heat alone at 500°F, a GR-S - carbon black mix giving a tensile of 2450 psi; but this crosslinking could only partially be inhibited. Salicylate esters were the most effective inhibitors.

Wyandotte Chemicals Corporation.
WADC TR 55-492 Pt III.
DEVELOPMENT AND PHYSICAL TESTING OF ELASTOMERIC COMPOUNDS RESISTANT TO PETROLEUM BASE FUELS AT ELEVATED TEMPERATURES. Raymond G. Spain, Wayne G. Lajiness, Eva Deck and Arnold L. McMaster. AF 33(616)-2779. February 1958. ASTIA Document No. 150978.

This report describes continued research toward the development of elastomeric compounds resistant to petroleum base fuels at temperatures up to 400°F for periods up to 500 hours.

Candidate elastomer compounds were screened using conventional fuel aging techniques. On the basis of these screening tests, compounds of interest were further tested under conditions of simulated use using the interim Type I Tester.

The multiple sample Type II Tester was completed during this report period.

The design and the major portion of assembly was completed for the Micro Tester which permits testing under more severe conditions than the two previous testers.

Aging studies were concentrated on compounds based on fluorinated elastomers.

WADC TR 58-19.
AN EVALUATION OF FLUORO-RUBBER 2F4. Roger E. Headrick. February 1958. ASTIA Document No. AD 150983.

Fluoro-Rubber 2F4 was developed by the Minnesota Mining and Manufacturing Company under Air Force Contract AF 33(038)515 to overcome the low temperature limitations of its precursor Fluoro-Rubber 1F4 (poly FBA).

This elastomer has a 30° to 40°F improvement in low temperature properties (-35°F Brittle Point) over Fluoro-Rubber 1F4 and has excellent resistance to jet fuels, lubricants and solvents and has a fair retention of physical properties after aging at elevated temperatures.

Boeing Airplane Company. WADC TR 56-272 Part III.
DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS. Frank W. Tipton, George E. Trepus, James J. Hill, Ethel L. Schiavon and Chester J. Dezeih. AF 33 (616)-2867. April 1958. ASTIA Document No. 151181.

This is a continuation of a study to gain knowledge of design data for O-rings and similar elastomeric seals. The literature survey on O-rings and seal design has been continued and enlarged to include the effects of irradiation and extreme low temperature on seal design. Low temperature tests, relaxation and volume change tests, screening tests, and functional tests have been conducted. The functional tests include pulsed annulus tests with various groove configurations and reciprocating shaft tests with and without different types of back-ups at room temperature and at elevated temperatures.

No overall relationship between physical property values and seal efficiency has been found. It has been noted, however, that certain physical properties appear to be important to seal life for specific seal conditions.

WADC TR 58-18.
INVESTIGATION OF OIL AGING PROCEDURES FOR ELASTOMERIC MATERIALS. K. Murray. May 1958. ASTIA Document No. AD 151196.

Several oil aging procedures utilizing various venting methods, and an oven or an aluminum block heater as heat sources, were compared to determine the most suitable, reproducible test procedure for evaluating potential oil-resistant elastomers at elevated temperatures.

The "chimneyed-stoppered-tube method" as described in this report provides for better reproducible results, and a better, more reliable evaluation of potential oil-resistant elastomers at elevated temperatures.

B. F. Goodrich Company. WADC TR 58-114.
A DESIGN MANUAL FOR ELASTOMERS USED IN NUCLEAR ENVIRONMENTS. Richard G. Bauman. AF 33 (616)-2308. June 1958. ASTIA Document No. AD 155631.

A design manual has been prepared which assembles the research information gathered by B. F. Goodrich scientists during the years 1954-1957. Only that material of interest to airframe design engineers has been included.

The primary topics included are the radiation resistance of a large group of rubber compounds to tensile changes; the dynamic property changes of a group of selected compounds when irradiated; the radiation resistance of practical aircraft compounds used in tires, hose, fuel cells, gaskets and packing; and the protection afforded by anti-rads.

Products Research Company. WADC TR 58-89.
DEVELOPMENT OF HIGH TEMPERATURE SEALANTS. Irvin P. Seegman, William Cheorvas, Francis H. Ingham, R. Loren Varner, Sarkis H. Kalfayan, Paul A. Mallard and Ron E. Thompson. AF 33(616)-3976. June 1958. ASTIA Document No. AD 155606.

A fill and drain sealant based on Viton A had been developed which has excellent resistance to fuels at 450° F. Good adhesion to stainless steel and titanium was obtained by use of a primer and was retained after exposure to fuel for 42 hours at 450°F, oven again 336 hours at 450°F and immersion in salt water for 168 hours at 140° F. The fill and drain system consists of a dilute solution of Viton A and phenolic resins as a primer, a Viton A sealant compounded with silicone coated silica and magnesium oxide at 28% solids in methyl isobutyl ketone and an amine curing solution.

An 80% solids filleting sealant with excellent resistance to fuel and dry heat at 450°F and adequate low temperature properties has been developed from Viton A cured with hexamethylene diamine. A phenolic primer provided satisfactory adhesion to stainless steel, while a dual primer system was required for titanium. The sealant can be cured adequately in one hour at 250°F but requires an additional 2 hours at 350°F to develop adhesion.

A 100% solids filleting formulation based on PAH-1 was cured with benzoyl peroxide to an elastomeric material which had excellent resistance to Type III and JP-5 fuels at 350°F, but its initial tensile strength properties were not of a very high order. After these exposure conditions good adhesion was retained to stainless steel primed with either DC-A-4014 or a phenolic-triallyl cyanurate primer. Improved tensile strength properties were obtained with peroxide cured filleting formulations based on Viton A, LS-53 or 1F4 plasticized with either PAH-1 or reverted LS-53U, but good adhesion to stainless steel has not been obtained.

A groove sealant was developed which meets the 350°F and 450°F requirements of this contract. This sealant formulated from LS-53U and 1F4 maintained its seal and was capable of reinjection after 6 days immersion in Type II reference fluid or JP-4 fluid at 275°F followed by 7 days at 350°F or 2 days at 450°F with equally satisfactory results. The compound met the major requirements of the proposed 250°F Military Groove Sealant Specification and the sealing and reinjection requirements of this specification when tested at 350°F and 450°F.

Compounds have been developed which should be suitable for use as nonfuel resistant pressurization sealants for high temperature exposures to 700°F. Formulations are based on room temperature vulcanizing silicone compounds. For elevated temperature service a stepwise post cure up to the anticipated operating temperature is required. Higher temperature properties were outstanding. Physical properties were reasonable and adhesion was obtained at all temperatures with use of the proper primer.

An inorganic polymer, phosphonitrilic chloride, has been polymerized to a rubbery mass. Several objectionable characteristics including its highly corrosive nature made it evident that this material was not suitable for sealants.

TR 4620.
PHYSICAL PROPERTIES OF NYLON MATERIALS DEVELOPED FOR AIR CORPS USE. J. Edward Gill. March 1941.

TR 5590.
TREATMENT OF NYLON WEBBING TO INCREASE RESISTANCE TO ABRASION. C. A. Willis. June 1947.

TR 5665.
POROSITY CHARACTERISTICS OF PARACHUTE MATERIALS. H. G. Battles. January 1948.

TR 5699.
TREATMENT OF NYLON WEBBING TO INCREASE RESISTANCE TO ABRASION. C. A. Willis. May 1948.

New Mexico State College of Agriculture and Mechanic Arts. TR 5894.
TEXTILES; ACCELERATED WEATHERING VERSUS OUTDOOR EXPOSURE TESTS. Joyce C. McGrath. AF 33(038)-16956. October 1950.

Georgia Institute of Technology. TR 52-55.
A STUDY OF THE EFFECT OF TWIST ON THE PROPERTIES OF SYNTHETIC FILAMENT YARNS. J. C. Taylor, C. Y. Widequist, M. M. Christie, and V. R. Thompson. AF 33(038)-10179. March 1952. PB 111241. Order from OTS $2.00.

As synthetic textile fibers have varied uses in today's ideas and materials, a need has been felt for an investigation into the effect of twist on synthetic yarns. It was decided that in the study of synthetic yarns, those used most in USAF materials would be evaluated.

Yarns studied included five deniers of Viscose Rayon from three producers; ten deniers of nylon, four deniers of Orlon; and three deniers of Dacron. The main objective of the program was the study of the effect of twist on the following properties of the yarn.

a. Denier
b. Diameter
c. Breaking strength
d. Elongation
e. Energy Absorption
f. Elastic Recovery

Increments of twist were added to the yarns until the breaking strength had decreased approximately 40% from the original values, or until creeping or shearing occurred.

The twisting of the yarns and testing were conducted in laboratories conditioned to 70° plus or minus 2°F and a Relative Humidity of 65 plus or minus 2%.

Each yarn has been considered separately in the evaluation, but with rayon yarns no separation was made for each different manufacturer's yarn. Generally the results show that the effect of twist on any characteristic of yarn is greater for the higher denier yarns than for the low denier yarns. Detailed data are given in both tabular and graphic form, for the effect of twist on the properties of the individual yarns.

TR 52-112.
DEVELOPMENT OF FABRICS FOR USAF SUMMER UNIFORMS USING SNYTHETIC FIBERS. Rama Maxwell Ellis, Jr., 2/Lt. July 1952. PB 11763 Order from OTS $0.75.

This project was undertaken as a result of a letter from Headquarters USAF, dated 29 March 1951, entitled "Conservation of Wool", and because it was believed that synthetic fibers possessed an untapped potential adaptable to the needs of the USAF for uniform purposes.

"Dacron", a polyester fiber, was chosen as the principal constituent in this investigation because of its unusual properties. The manner in which this fiber would react when used in a uniform fabric and when blended with other fibers could not be determined by laboratory methods; hence the service wear test.

This service wear test was conducted during the months of July, August, September and October of the year 1951 at Wright-Patterson Air Force Base, Ohio, by the Textile Branch of the Materials Laboratory, Research Division, Wright Air Development Center. Forty-eight men participated in the test.

The results of this test indicate that Dacron, when blended with wool or viscose rayon in a fifty-to-fifty weight ration, will produce a fabric superior to either the presently used 100% wool or the experimental 100% Dacron fabric.

Georgia Institute of Technology. TR 52-283. Part I.
AIR PERMEABILITY OF PARACHUTE CLOTHS. M. J. Goglia. AF 33(038)-15624. November 1952. PB 111242. Order from OTS $2.00.

The air permeability of eight standard nylon parachute cloths was determined using a sample 6.05 inches in diameter in a wind tunnel whose capacity permitted obtaining static pressure differentials across the cloth as high as 55 inches of water. Fifty-nine experimental nylon cloths manufactured by the Bally Ribbon Mills were subjected to the same test procedure, as were two experimental fabrics of orlon and dacron, respectively.

Upon assuming that the presure gradient in the flow through a parachute fabric is proportional to the arithmetic sum of an inertial ($\beta\rho v^2$) and the viscous contribution ($\eta_\mu v$), one is able to infer the existence of a parameter, β/α, whose measure is length. This length can be employed to characterize the geometry of the cloth. Experimental work to date in the case of the eight standard cloths and the orlon and dacron fabrics has indicated a verification of the assumption; a high-pressure tunnel employing pressure differentials across the cloth approximately ten pounds per square inch was used for this purpose.

Employing the characteristic length so determined permits writing a single relation common to all cloths between a "flow-through-drag coefficient," C_f, and a Reynolds number based on the characteristic length; viz., $C_f = 2\phi \frac{2}{N_{Re}}$.

Georgia Institute of Technology. TR 52-283. Part 2.
AIR PERMEABILITY OF PARACHUTE CLOTHS. H. W. S. Lavier. AF 33(038)-15624. December 1952.

The air permeability of special, woven, nylon, orlon, and dacron, parachute-type fabrics was determined using a sample 6.05 inches in diameter. The permeometer used in this program permitted testing the fabric samples at pressure differentials across the cloth as high as 55 inches of water. The sixty-one experimental cloths woven

in the Laboratories of the Georgia Institute of Technology Textile School were subject to this test procedure.

Air permeability data for the Georgia Tech-woven fabrics, Bally Ribbon cloths, and the ten Air Force-furnished fabrics are presented here in graphical form as volumetric flow (cubic feet per minute) versus static pressure differential across the cloth.

The number of ends per inch in warp, and picks per inch in the filling, and denier of yarns was found to affect air permeability of these fabrics. The finishing of the fabrics was found to affect the permeability more than any other of the many variables involved. The tests to determine the effect of variation of the weave pattern failed to show significant differences on this account.

Pioneer Parachute Company. TR 52-57. INVESTIGATION OF IMPACT LOAD ABSORPTION THROUGH SUSPENSION LINE ELONGATION. E. A. Gimalouski. AF 33(038)-10401. December 1952. PB 111239. Order from OTS $2.50.

The purpose of this investigation was the study of suspension lines under actual operating conditions, having varying degrees of elongation, energy absorption and elasticity. A secondary purpose of the investigation was the study of fabric porosity, shape and type of canopy, and method of parachute deployment.

The shock absorbing capacity of five different types of parachute suspension line cord were first evaluated for basic physical properties, then were tested by assembling into parachutes and opening the parachutes at various speeds - first at 100 mph, then 150 mph, finally 200 mph - on a whirling test tower. The parachutes were evaluated first in standard back type, then in deployment bag openings. Two types of parachutes were used; one being a twenty four foot standard parachute, the other being a 30 foot parachute, with an extended skirt.

The loads were measured and the cords evaluated on the basis of shock absorbing capacity, strength, and durability. Final results of the work indicates that the presently used 550 lb tensile strnegth cord, Specification MIL-C-5040, Type III, is the best of the cords evaluated. A good correlation was established between line energy absorption and snatch force, but opening shock loads appear to be little affected by line characteristics.

Fabric Research Laboratories. TR 53-21. A STUDY OF THE EFFECT OF TEMPERATURE ON TEXTILE MATERIALS. Myron J. Coplan. AF 33(038)-22932. March 1953. PB 111240. Order from LC, Mi $7.00, Ph $24.00.

Temperature requirements are of prime importance in the development of materials and equipment for the USAF. The use of parachutes to decelerate aircraft and recover missiles from high altitudes, brings forth the requirements for heat resistance, while operation in the Arctic and Antarctic regions sets the requirements for low temperature. With these requirements in mind, an investigation to study the effects of temperature and humidity on textile fibers was initiated. To make this investigation as thorough as possible, all fibers, both natural and synthetic, which were available were included, with the exception of those fibers not thought feasible for parachute use, such as wool.

The yarns studies were broken down into two categories; filament and staple. The filament yarns were:

a. Nylon
b. Orlon
c. Dacron
d. Fortisan (Saponified Acetate)
e. Tenasco (High Tenacity Viscose Rayon)
f. Vinyon NORU, NOHU
g. Japanese Gum Silk
h. Fiberglas

The staple yarns used were:

a. Acrilan
b. Dynel
c. Cotton, griege
d. Cotton, mercerized

The main objective of this investigation was to evaluate the fibers at temperatures from -70°F to the extremely high temperatures, at which the fibers fail to function efficiently. (This temperature ranges from 300°F to 375°F as shown in the report.) To make the evaluation complete in itself, such properties of the yarns as average breaking tenacity, average breaking elongation, average yield tenacity, average initial modulus, average loop tenacity and efficiencies, energy absorbed to several stress levels, and heat shrinkage were obtained at various temperatures and after exposure for specific lengths of time at each temperature. Detailed data are given in both tabular and graphic form, as well as a discussion covering the mechanical behavior of fibers.

It is quite apparent from the tables and charts, that at and after exposure to temperatures of 350°F, Dacron yarn is superior in most of the properties studied. Most noticeable is the energy absorbed at various stress levels, loop tenacity and efficiency, and especially Dacron's resistance to hot air degradation.

Fabric Research Laboratories, Inc. TR 53-21, Part 2. A STUDY OF THE EFFECT OF TEMPERATURE ON TEXTILE MATERIALS. Myron J. Coplan and Eric Singer. AF 33(038)-22932. July 1953. PB 130399. Order from LC, Mi. $3.60, Ph $9.30.

A group of fabricated textile materials including seam samples were tested at four specific temperature conditions. The materials involved included cotton in addition to the nylon materials. The samples included thread and suspension line as well as cloth. The seam samples were constructed as called out in parachute fabrication drawings for two needle seams, and skirt butterfly attachments.

The strength of several nylon materials was found to vary with temperature. It was found that the sensitivity to temperature is due in part to geometric influences on the inherent yarn strength. During the preheating of the nylon materials, sufficient dehydration took place causing a loss in efficiency at 210°F.

As expected, the cotton materials lost considerable strength at the low and high temperatures.

A number of specific conditions must exist for sticking of nylon cloth to nylon cloth to be evidenced. Indications are that prolonged exposure to heat (350° to 370°F) may reduce the cohesion of the two surfaces. Therefore sticking will not be caused due to temperature over a certain period of time. Conditions such as sharp impact, surface over surface shear at high pressures may result in some sticking or cohesion at lower temperatures.

Cheney Brothers. TR 53-351. DEVELOPMENT OF 0.9-OZ. NYLON PARACHUTE CLOTH. H. J. Bickford. AF 33(600)-23196. September 1953. ASTIA Document No. AD 26850. PB 133702. Order from LC, Mi $2.70, Ph $4.80.

A 4,000-yard lot of Nylon Parachute Cloth weighing 0.9 ounce per square yard or less was woven and finished with Silicone Oil. The properties of this fabric were tested and found to meet the target properties laid out in the contract.

Accelerated aging tests indicated that the Silicone Oil provides a measure of protection against fabric deterioration and definitely improves the tear resistance at all times.

This fabric can fulfill certain experimental purposes where an extremely light thin parachute fabric is needed for models. In addition the material could be used in critical applications requiring small yardage. However, the difficulties of manufacture are so extensive that use of this fabric should be confined to special applications which justify its use.

TR 53-444.
EVALUATION OF EXPERIMENTAL WOOL AND SYNTHETIC BLENDS IN AIR FORCE VELOUR OVERCOAT MATERIAL. Ormond J. Roberts, 1st Lt, USAF and C. A. Willis. November 1953. PB 111884. Order from OTS $1.25.

A group of 30 wool/synthetic, blended velour overcoat fabrics were evaluated to determine the effects on fabric properties when the fiber and composition was varied.

The synthetic fibers involved included Orlon, Dacron, Acrilan, Viscose and Dynel. Each synthetic fiber was blended with wool in percentages of 10, 20, 30, 40, 50 and 60 to make a series of 6 samples. One sample was fabricated to 100 percent wool to be used as a control.

The use of Dacron in the fabric increased the breaking strength. Flex abrastion resistance was increased when the synthetic was used in percentages of 20 or above. Flat abrasion resistance was not significantly affected. Shrinkage in sponging and crease resistance was not appreciably affected.

The use of Acrilan in the fabric increased the breaking strength when used in percentages of 50 and above. Reduced breaking strength was noted when 10 and 20 percent Acrilan was used. Resistance to abrasion, both flat and flex, was considerably lower in all percentages, and remained approximately equal throughout the entire series of samples. Crease resistance and shrinkage in sponging were not significantly affected.

The use of viscose in the fabric decreased the breaking strength and flex and flat abrasion resistance. Crease resistance was approximately equal to the all wool control. Shrinkage in sponging was slightly higher in the warp direction with a gain noted in the filling direction.

The use of Dynel in the fabric increased the breaking strength when used in percentages above 10. Resistance to abrasion, both flat and flex, was markedly decreased. Shrinkage in sponging and crease resistance were not significantly affected.

As the percentage of synthetic was increased in the blend the difficulties encountered at the fulling process increased and it became more difficult to obtain the desired color and finish.

Due to the difficulties encountered in processing velour fabrics using wool and synthetic fibers, the Air Force will continue to use only the 100% wool fabric. In the event of an emergency and as a wool conservation measure, synthetic fibers are recommended only in the amounts up to 20 percent.

Bjorksten Research Laboratories, Inc.
TR 53-298.
INVESTIGATIONS OF MYLAR-TYPE POLYESTER FILMS FOR PARACHUTE CANOPIES. Johann Bjorksten, Risto P. Lappala and Robert J. Roth. AF 33(616)-403. December 1953. ASTIA Document No. AD 27594. PB 139011. Order from LC, Mi $2.70, Ph $4.80.

Double layer Mylar films reinforced with Dacron fiber have exhibited promising properties for use in parachute canopies. These properties include:

Tensile Strength	82 - 90 pounds
Burst Strength	195 - 220 pounds
Weight Per Square Yard	2.44 - 4.42 ounces
Thickness (maximum)	0.007 - 0.008 inches

No embrittlement at -60°C and retention of useful properties at 150°C.

These films were prepared by cementing a reinforcing grid of 2, 3 or 4 ply Dacron thread between two 50 or 100 gauge Mylar films.

Miniature parachutes made of Reinforced Mylar Films had exceptional resistance to tearing, to failure by flexing or creasing, and to abrasion. Accelerated rapid-load tests of these parachutes did not produce any failure in the seams or material. Reinforced Mylar Film thus compared favorably with Nylon except for its weight and porosity which could probably be improved by further investigation.

University of Cincinnati. TR 53-414.
THERMAL AND AREA STABILITY OF LEATHER. Herbert C. Hamel, Robert E. Merritt and William T. Roddy. AF 33(616)-136. January 1954.

A comparision of the properties of horsehide versus cowhide leathers was made to determine the feasibility of using cowhide leathers as well as horsehide leathers for glove purposes. The data collected would indicate that cowhide leather would serve the purpose. There was the possibility that the initial run obtained in horsehide glove leather might not be present in the cowhide leather.

Tannage and the process ramifications necessary to each tannage preparation were studied as to their influence on thermal and area stability of leather. For the purpose of study a series of commercial tannages were investigated. The data obtained shows that tannage is a very important factor in preparing fire resistant leather. The data also indicates that the objective of developing a glove leather with higher heat and area stability has been accomplished.

The application of fire retardants to leather was also investigated. The fire retardants selected on the basis of the improvement in fire resistance they imparted to cloth fabrics did not impart good fire resistance to leather. Some of the disadvantages encountered when these materials were applied to leather are given in the report.

TR 54-12.
THE EFFECT OF A SNYTHETIC LUBRICANT AND TWO DI-BASIC ACID ESTERS ON CERTAIN USAF FABRICS. Clarence D. Smith, 1st Lt, USAF. March 1954. ASTIA Document No. AD 38147. PB 134712. Order from LC, Mi $2.40, Ph $3.30.

This investigation was initiated to determine if fabrics employed by the USAF were subject to degradation or deterioration when exposed to the lubricanting oil covered by Specification MIL-L-7808 and the acid esters, di-2-ethyl hexyl adipate and di-2-ethyl hexyl sebacate. Need for an investigation was relized upon receipt of information that fabrics had been damaged when exposed to synthetic lubricants. In order to accomplish the desired program a series of fabrics composed of fibers that are commonly used in USAF fabrics was exposed to the above synthetic lubricants at room temperature and at 160°F. After exposure, laundering and dry cleaning tests were conducted to determine their effect in conjunction with the lubricants. By diaphragm burst tests it was found that no damage was apparent in the fabrics used when exposed to the above synthetic lubricants.

The George W. Borg Corporation. TR 53-452. DEVELOPMENT OF DYE FORMULATIONS AND DYEING PROCEDURES FOR PRODUCING SAGE GREEN PILE FABRIC. A. Bartovics. AF 33(600)-23440. May 1954. ASTIA Document No. AD 34419. PB 130375. Order from LC, Mi $2.40, Ph $3.30.

Dye formulations and dyeing procedures are presented for producing the required shade of Sage Green Number 530 with the maximum obtainable colorfastness properties for Type I (Acrilan) and Type II (Vicara-Dynel) pile fabric conforming to Specification MIL-C-6590. Physical and colorfastness properties of the respective fabrics thus produced are tabulated, and are shown to conform to the requirements of the above specification.

A tentative dye formulation and dyeing procedure for producing Sage Green Type III (Orlon) fabric and the physical properties of a preliminary sample of this fabric are also reported. This covers the work done on Type III fabric up to the date of its suspension from the contract by the USAF.

Thomas Taylor & Sons, Inc. WADC TR 54-39. AN EMPIRICAL REPORT ON THE EFFECT OF POLY-VINYL BUTYRAL ON CARGO-PARACHUTE SHROUD LINES. Robert L. Phipps. AF 33(600)-23556. June 1954. ASTIA Document No. AD 47355.

WADC TR 54-117. A STUDY OF THE EFFECT OF TEMPERATURE ON PARACHUTE TEXTILE MATERIALS. James W. Muse, Jr. July 1954. ASTIA Document No. AD 55667. PB 136765. Order from LC, Mi $3.30, Ph $7.80.

The primary purpose of this investigation was to obtain data on the effect of oxygen, nitrogen, and compressed air on nylon and Dacron materials at various temperatures. A secondary purpose was to determine the effect of hot air, applied continuously and in an intermittent manner to parachute textile materials. The test method is described in the text of this report.

A group of standard nylon parachute textile materials comprised of webbings, cord and fabrics, was tested after exposure to various temperature conditions for continuous and intermittent intervals of time. Also tested were Dacron hot stretched threads and an experimental Dacron fabric.

The breaking strength of several nylon textile materials was found to vary with temperature and duration of exposure time. The nylon materials lost considerably more strength after exposure in hot air at 300°F than at 250°F for the same period.

The breaking strength of nylon webbings, treated with resin in accordance with Specification MIL-W-4088B, was affected more severely than the untreated webbings after exposure to the stated conditions.

The breaking strength of the Dacron hot stretched thread showed no appreciable loss in these tests.

The materials tested after exposure in hot air at 250°F for identical continuous and intermittent time intervals show no appreciable difference in breaking strength.

The materials tested after intermittent exposure in hot air at 300°F show more loss in breaking strength than when tested after continuous exposure.

WADC TR 53-333. A STUDY OF SYNTHETIC FUR RUFFS. R. M. Ellis, Jr. July 1954. ASTIA Document No. AD 50286. PB 130470. Order from LC, Mi $3.60, Ph $9.30.

Laboratory and field tests on synthetic wolverine furs were conducted at Wright Air Development Center, Wright Patterson Air Force Base, Ohio, and in Alaska, respectively. The laboratory tests were conducted on sixty constructions representing variations in methods of fabrication and fiber composition. The furs for field tests represented eight different variations of the most promising types used in the laboratory evaluation. Natural wolf fur was used for comparison in both evaluations.

In both laboratory and field tests the better synthetic furs were superior to the wolf fur in ice shedding ability. In addition, the synthetic fur is domestically and readily available and should cause no storage problems.

In wind protection and comfort to the wearer the natural fur appeared to be superior.

The tests have provided data which should be useful in designing synthetic fur which will be satisfactory in every respect. The importance of comfort is emphasized.

WADC TR 54-49. SYMPOSIUM ON PARACHUTE TEXTILES. Joyce C. McGrath. July 1954. PB 111563. Order from OTS $5.50.

Technical papers concerning recent developments and future requirements of parachute textile problems were presented at a WADC Symposium on 21 and 22 September 1953. In particular, recent developments in the studies of friction damage, effects of high temperature, air flow through cloth, and design data for use in developing parachute textile materials which will operate at high speeds were discussed.

University of Cincinnati. WADC TR 54-63. DEVELOPMENT OF LEATHER FOR MECHANIC'S WINTER GLOVE. Lewellyn G. Picklesimer, William T. Roddy. September 1954.

A comparison of the properties desired in glove leather tannages for a comfortable mechanic's winter glove was made. The data collected indicate that irrespective of tannage the leather has a high rate of oil absorption.

The application of various impregnants to glove leather gave an improvement in oil resistance. Some of the impregnants were very oil resistant but did not penetrate into the leather. Using such impregnants as finish films on the leather would give oil resistance but when the finishes were abraded through, the leather would offer very little oil resistance. The data obtained indicate that it is possible to prepare a glove leather for winter mechanic's wear which will have good resistance to oil absorption without materially reducing the desirable properties inherent in glove leather.

The Connecticut Hard Rubber Company. WADC TR 54-93. COATED FABRIC FOR USE IN PROTECTIVE CLOTHING. H. N. Homeyer, Jr., J. Becker. AF 33(616)-155. October 1954. ASTIA Document No. AD 55694. PB 134847. Order from LC, Mi $5.70, Ph $16.80.

Experimental work leading to the development of a chemically resistant coated fabric, suitable for use in protective clothing for personnel handling various fuels and oxidizers used in guided missiles, is described in this report. The fabric consists of a pigmented blend of polyethylene and Vistanex B-80 calendered on Vinyon Fabric #501, primed with a solution (18% solids) of Vistanex B-80 dispersed in toluol. It possesses good resistance to the action of nitric acid and excellent flexibility throughout a temperature range of -67°F to 157°F when tested in accordance with the procedure outlined in Specification MIL-F-4143 (USAF).

Protective clothing in the form of suits, hoods and gloves, fabricated from the coated material, as well as a number of yards of the fabric itself, has been shipped

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to the Wright Air Development Center. Since the coated fabric which has been developed is superior to suit materials in present use which exhibit only slight resistance to nitric acid, it is concluded that personnel operating in the field of guided missiles can now be provided with clothing which will offer adequate protection against rocket fuels. It is recommended that the coated fabric be used in the fabrication of all clothing designed for the protection of personnel engaged in handling oxidizers and most rocket fuels.

The coated fabric was developed under Contract No. AF 33(616)-155 at The Connecticut Hard Rubber Company, New Haven, Connecticut, during the period from May 22, 1952 to December 31, 1953.

WADC TR 54-52.
EVALUATION OF EXPERIMENTAL WOOL SYNTHETIC BLENDS IN AIR FORCE 18 OZ. BLUE SERGE. C. A Willis, C. W. Long. November 1954. PB 111676. Order from OTS $0.75.

A group of twenty-one serge fabrics was evaluated in the laboratory to determine the effects on fabric properties when the fiber and percentage of fiber was varied. The fabrics included the 100% wool standard serge and twenty wool/synthetic blends. The fibers employed in the course of manufacturer were wool, Dacron, Orlon, Acrilan, viscose rayon and Dynel. Each synthetic fiber was blended with wool in percentages of 10, 25, 40 and 60.

Comparative tests of the following properties versus wool percentage of the blends were conducted: warp breaking strength, filling breaking strength, warp percent shrinkage, filling percent shrinkage, warp abrasion, filling abrasion, flat abrasion, percent wrinkle recovery, flame time, char rate and wicking time.

Based on laboratory test results of the five synthetic fibers used and the four percentages blended with wool, Dacron, when blended in the fabric appears superior to the other four synthetics in increasing resistance to flat and flex abrasion, and increased breaking strength when used in higher percentages.

In all cases the rate of burning and wicking was increased, and the percent wrinkle recovery slightly decreased, by the addition of synthetic fiber.

Conclusions drawn from the laboratory tests are of interest; however, laboratory testing techniques are not to be considered an absolute measure when evaluating differences between wool and wool/synthetic blends.

Georgia Institute of Technology. WADC TR 52-283. Part 3.
AIR PERMEABILITY OF PARACHUTE CLOTHS - PART 3. EFFECT OF LOADING ON ELASTIC PROPERTIES OF PARACHUTE CLOTHS WITHOUT AIR FLOW. H. W. S. Lavier. AF 33(038)-15624. January 1955.

The effects of biaxial tension loads on fabric samples of nylon, Orlon, and Dacron have been studied. The biaxial-fabric-tension-testing machine, used in these tests, was designed and constructed at Georgia Tech.

It was the purpose of these studies to determine the elastic characteristics of selected nylon, Orlon, and Dacron parachute-type cloth under conditions of no air flow. Also, the effect of high temperatures (+140°F) and low temperatures (-14°F) on elastic properties was determined, using plain-weave samples of nylon, Orlon, and Dacron cloth. The data obtained from these tests were used in conjunction with the low- and high-pressure air-permeability studies reported in AF Technical Reports 52-283, Parts 1, 2, and 4.

Variations in temperature and humidity, in the ranges studied, had a marked effect on the elastic properties of the fabrics. However, as humidity was lowered, the elasticity and the tenacity of the fabrics decreased.

Dacron was found to be somewhat more elastic than Orlon, particularly at the lower temperatures.

The number of picks per inch and the weave pattern (plain, twill, and satin) do not markedly affect the elastic properties.

Georgia Institute of Technology. WADC TR 54-199.
A STUDY OF THE LAWS OF THE FLOW OF FLUIDS THROUGH FABRICS. Cecil D. Brown. AF 33(038)-15624. January 1955. ASTIA Document No. AD 63448. PB 134273. Order from LC, Mi $4.80, Ph $13.80.

In this study a method is developed of presenting the flow data in a general dimensionless from over a limited range of flow. Under the assumption that the pressure-squared gradient in the flow through a fabric is the arithmetic sum of the viscous ($2\alpha RT\frac{\mu}{gL}G$) and the inertial contributions ($2\beta\frac{RT}{g_c}G^2$), it is possible to infer the existence of a characteristic length $\frac{\beta}{\alpha}$. The relation between the flow-through-drag coefficient and the Reynolds number based on this characteristic length, $C_f = \frac{2}{N_{Re}} + 2$, is common to all fabrics. The parameters α and β and the characteristic length can be obtained from only two flow measurements.

The parameters α and β may be estimated from physical measurements of the cloth, thus permitting a rough prediction of the permeability of the fabric.

Georgia Institute of Technology. WADC TR 52-283. Part 4.
AIR PERMEABILITY OF PARACHUTE CLOTHS. H. W. S. Lavier. AF 33(038)-15624. February 1955.

The high-pressure air permeability of selected nylon, Orlon, and Dacron parachute-type fabrics was determined using a 16-square-inch sample. The Georgia Tech high-pressure permeometer used in this program permitted testing the fabric samples at pressure differentials across the cloth equivalent to 1500 inches of water. The selected cloths are described in Table I and include experimental cloths woven in the Laboratories of the Georgia Institute of Technology.

Air-permeability data for the selected fabrics are presented here in graphical and tabular form as volumetric flow (cubic feet per minute) and effective porosity versus the static pressure differential across the cloth.

The selected fabrics were chosen to demonstrate the effect on high-pressure air permeability resulting from variation of the number of ends and picks per inch, weave patterns, and material. Also, the effect on high-pressure permeability, due to variation of temperature and absolute humidity, was investigated.

Cheney Bros. WADC TR 54-468.
STUDY OF THE CONTROL OF PERMEABILITY AT HIGH AND LOW DIFFERENTIAL PRESSURES. Hamilton J. Bickford, Donald K. Kuehl, Thomas L. Rusk, Jr. AF 33(600)-26109. March 1955. PB 111855. Order from OTS $1.50.

Twenty-four differently constructed samples of nylon cloth in the desired weight range were woven, finished and tested.

A special mathematical study of the relationship between air permeability at 1/2 inch of water pressure differential and at higher pressure differentials was made. This discloses that a linear relationship in these values exists when plotted on full logarithmic graph paper.

The ability of the cloth manufacturer to vary the high pressure differential permeability, while retaining fixed low pressure permeability ranges is indicated to be a practical one within limits.

A total of 1000 yards of additional cloth duplicating two of the twenty-four constructions as selected by the Air Force was supplied for use in further evaluation of the material by the Parachute Laboratory.

Georgia Institute of Technology. WADC TR 52-283. Part 5.
AIR PERMEABILITY OF PARACHUTE CLOTHS. H. W. S. Lavier, W. C. Boteler. AF 33(038)-15624. April 1955. PB 149990. Order from LC, Mi $4.50, Ph $12.30.

The air permeability evaluations of various nylon cloths produced by different manufacturers to the same MIL specifications are presented here. The effect on air permeability due to various weaving defects was investigated and it was concluded that such normal defects had little or no effect on the cloth air permeability evaluations.

Variation of the magnitude of the calendering load was studied regarding its effect on air permeability of a cloth. It was found that loads up to fifty tons per inch caused considerable change in the cloth permeability. However, loads above fifty tons per inch caused very little additional change in air permeability over that caused by a load of 50 tons per inch.

Discharge coefficients for various kinds of parachute cloths are also included in this report. These values may prove useful in designing new parachute textiles.

Phoenix Trimming Co. WADC TR 55-3.
DEVELOPMENT OF LOW ELONGATION, HIGH STRENGTH WEBBING FOR USE IN SAFETY HARNESS APPLICATION. Russell J. Neff. AF 33(616)-2361. May 1955. ASTIA Document No. AD 80126. PB 130390. Order from LC, Mi $3.00, Ph $6.30.

Four types of Dacron webbing, developed by the Phoenix Trimming Company, were subjected to a series of tests to determine whether they were acceptable for use in aircraft safety harnesses. Samples of these webbings were made from three different types of Dacron, with the final delivered webbing being manufactured from 1100-250-0-560 Dacron yarn.

WADC TR 54-374.
PILE FABRICS FOR INSULATION. C. W. Long. June 1955. PB 111985. Order from OTS $1.25.

This is a report on pile fabrics made from synthetic fibers, cotton, wool, and numerous blends thereof. There were three techniques employed in the construction of the pile fabrics developed under this project: (1) woven cut pile fabrics by Goodall Sanford, Inc., (2) inserted pile, knitted fabrics by George W. Borg Corporation, and (3) napped and/or brushed pile, knitted fabrics by Princeton Knitting Mills, Inc. All the samples developed were compared to the standard wool pile fabric made according to requirements of AF Specification MIL-C-5563. Each sample was tested for warmth and compression characteristics to determine the effect, if any, of varying thicknesses, blends, and constructions. It was observed that the type fiber has little effect on the warmth of a pile fabric; however, Orlon, Dacron, and Dynel consistently appear slightly better. Results show that possibly a double thickness of a relatively thin pile fabric should deserve consideration. Also included in this report are the results of a study on the mathematical relationship between the warmth of a fabric and the physical properties of the fabric.

The Synthetical Laboratories. WADC TR 54-466.
PRODUCTION OF LEATHERS RESISTANT TO POWERFUL OXIDANTS AND REDUCTANTS. N. D. Cheronis, M. A. Wente. AF 33(600)-23872. June 1955. PB 111882. Order from OTS $1.75.

Lowell Technological Institute Research Foundation. WADC TR 54-570.
THE EFFECT OF FABRIC STRUCTURE ON THE FRICTIONAL FUSION OF PARACHUTE MATERIALS. Vasilis Lavrakas. AF 18(600)-136. August 1955. PB 121142. Order from OTS $0.50.

The effect of fabric structure, yarn twist, calendering, and fabric weight on the resistance to fusion of parachute cloth has been studied. A belt friction appartus, used in lubrication investigations, has also been utilized in this study.

The parameters of fabric structure, yarn twist, calendering, and fabric weight have been found to be significant in the resistance of parachute cloth to friction.

The results of this study are as follows:

1. The best fabric structure was cloth made according to MIL-C-7020, Type I.
2. Calendered cloth was superior to non-calendered.
3. Low yarn twist and fabric weight appeared to impart higher resistance to fusion than higher twist and weight.

Lowell Technological Institute Research Foundation. WADC TR 54-513.
EVALUATION OF ANTISTATIC AGENTS ON NYLON PARACHUTE CLOTH. James W. Sweeney. AF 33(616)-458. September 1955. PB 121141. Order from OTS $2.00.

A literature search covering instrumentation, antistatic agents, and theories of static electrification was conducted to provide a foundation for the evaluation of antistatic treated nylon parachute cloth. An appraisal of the techniques employed by other investigators, in the evaluation of the static behavior of materials, justified the utilization of fabric surface resistance as the measurable parameter. Special instrumentation was developed to provide the desired test conditions of -30°F and 10% RH and permit the required range of resistance measurements. While no permanent antistatic agent studied was found to be effective at the low temperature test conditions, specific non-conditions which were comparable to the resistance of untreated nylon fabric at standard test conditions of 70°F and 65% RH.

Lowell Technological Institute Research Foundation. WADC TR 54-323. Part 2.
THE EFFECT OF SURFACE FINISHES ON FRICTION AND FUSION OF PARACHUTE CLOTH AND LINE. Vasilis Lavrakas, Adolph Katz. AF 18(600)-136. October 1955. PB 121254. Order from OTS $1.50.

A belt friction apparatus was used to study the fusion of scoured parachute cloth and lubricated cord at high sliding speeds. Data obtained at speeds up to 97 ft/sec when extrapolated to higher speeds (100- 140 ft/sec) indicate that no further significant data would be obtained regardless of the type of agent applied.

The effect of speed on fusion and friction has also been investigated. Primarily, three homologous series of high molecular weight organic compounds were used: high molecular weight fatty acids, alcohols, and monoesters. In their ability to prevent fusion, fatty acids were

poorer than fatty alcohols, while fatty alcohols were poorer than fatty monoesters.

As the speed of rubbing increases, the resistance to fusion of lubricated nylon parachute materials decreases rapidly, and at high speeds (75 ft/sec) this resistance falls to alarmingly low levels.

The role of molecular weight in the phenomenon of fusion and lubrication was investigated and found to be an important factor. Other factors are undoubtedly present but were not investigated.

Anionic, cationic, and nonionic lubricants were also studied. As an insufficient number were investigated, no definite conclusions have been formulated.

Lowell Technological Institute Research Foundation. WADC TR 54-572.
AERODYNAMIC HEATING OF PARACHUTE RIBBONS. Louis C. Block. AF 18(600)-136. November 1955. PB 121150. Order from OTS $1.50.

This research program involved the evaluation of wind tunnel tests on parachute ribbons. The work was conducted at the Lowell Technological Institute Research Foundation from March, 1952, to March, 1954.

A theoretical evaluation of the expected temperature rise of these ribbons in high velocity flight was carried out. Wind tunnel tests were subsequently performed on the basic ribbon and ribbons of modified design.

The results showed that the experimental values of the heat transfer coefficient agreed very well with the theoretical values. Changes in ribbon design and ribbon material did not effect an improvement to the heat transfer characteristics of the basic sample.

Lowell Technological Institute Research Foundation. WADC TR 54-573.
DESIGN AND DEVELOPMENT OF A HEAVY WEIGHT HIGH IMPACT SHOCK MACHINE. Edward N. Sabbagh, Arthur S. Quint. AF 18(600)-127. November 1955. PB 121198. Order from OTS $1.00.

The construction and operation of a pneumatically driven impact tester designed to evaluate the dynamic performance characteristics of cushioning materials is described. The 577-pound flat impacting element is capable of being projected downward with a velocity of from 20 ft/second to over 50 ft/second. An analog computer is used in a new application to record and analyze the impact.

Lowell Technological Institute Research Foundation. WADC TR 54-571.
EVALUATION OF FABRIC FINISHES FOR HIGH TEMPERATURE OPERATION OF PARACHUTE RIBBONS. Louis C. Block. AF 18(600)-136. November 1955. PB 121197. Order from OTS $1.50.

This research program involved the evaluation of the effect of surface finishes on the high temperature properties of nylon parachute ribbons. The work was conducted at the Lowell Technological Institute Research Foundation from March 1952 to December 1954.

The fabrics were exposed to temperatures above the melting point of nylon (480°F) produced by three modes of heat propagation: conduction, convection, and radiation. Instrumentation and testing programs were developed to appraise the results.

The results showed that no single finish satisfactorily retarded the fusion of the nylon ribbon when it was tested by all three methods. The conclusions drawn were that if a technique to change the heat transfer characteristics of nylon at these high temperatures is to be realized, methods other than applying surface finishes should be investigated.

WADC TR 54-574.
A STUDY OF THE EFFECTS OF FABRIC GEOMETRY VARIABLES ON AIR PERMEABILITY. William O. Perry. November 1955. PB 121211. Order from OTS $1.75.

A series of specially designed nylon parachute fabrics was selected to represent extremes in cloth construction and to demonstrate fundamentals of fabric geometry.

By means of a somewhat unusual rest arrangement it was possible to indicate the relationship of fabric geometry to air permeability at several pressure differentials. Through the process of establishing the ratio of total fabric area to interstice area, data were obtained on yarn widths as they lie in the cloth. These data were obtained on a variety of twist constructions and will provide a knowledge and background of design data for present application and future studies in this area.

Lowell Technological Institute Research Foundation. WADC TR 54-323, Part 1.
THE EFFECT OF SURFACE FINISHES ON FRICTION AND FUSION OF PARACHUTE CLOTH AND LINE. Vasilis Lavrakas, Adolph Katz. AF 18(600)-136. December 1955. PB 121186. Order from OTS, $3.75.

An attempt to find lubricating agents which would minimize or prevent the fusion of nylon parachute materials caused by frictional heat was the main objective of this program. A friction apparatus, based on the belt friction principle, was developed to evaluate lubricating materials. Scoured parachute materials possessed high frictional forces and fused at low speeds and loads, while lubricated materials possessed lower frictional forces and fused at higher speeds and loads.

Many lubricants were examined and ten were classed as highly effective in preventing fusion. In addition, nylon parachute line coated with these best lubricants was further subjected to breaking strength, fungus resistance, and aging tests. No one lubricant was determined to be superior in all respects. It is recommended that a selection be made among several of those lubricants possessing the most desirable attribute, resistance to fusion, and subjecting them to drop tests at the indicated percentage pickup level.

Connecticut Hard Rubber Company. WADC TR 55-324.
DEVELOPMENT OF CHEMICALLY RESISTANT, HIGH-TEMPERATURE PROTECTIVE FABRIC. A. S. Kidwell. AF 33(616)-2544. January 1956. PB 121212. Order from OTS, $2.25.

A coated fabric comprising a thin coating of aluminum-pigmented polychlorotrifluoroethylene on glass fabric backed with a low-density silicone rubber sponge, and having an overall thickness of 0.070 inch, was developed for use in protective clothing for personnel in danger of being exposed briefly to a chemical fire at temperatures up to 1000°F. The fabric shows excellent resistance to fuming nitric acid and other oxidizers and fuels, is flexible and useful over a temperature range of -80°F to 390°F.

This fabric construction showed a temperature rise on the inside surface of less than 100°F after direct exposure to a flame caused by mixing white fuming nitric acid and monoethylaniline. Peak-flame temperatures averaging about 1600°F three seconds after mixing the reactants were measured with thermo-couples. The fabric

was non-porous to 1000°F steam, and was not penetrated by reaction products of the ethyl aniline-fuming nitric acid combustion.

Polyethylene and polyethylene-Vistanex blends were cured with peroxide and were resistant to temperatures of 250°F. They also showed improved resistance to penetration by white fuming nitric acid.

Lowell Technological Institute Research Foundation. WADC TR 55-83.
THE DEVELOPMENT OF WOOL-SYNTHETIC BLENDED FABRICS FOR SUMMER FLIGHT GARMENTS. George O. Langlais, Samuel L. Fuller. AF 33(600)-25892. January 1956. PB 121153. Order from OTS, $2.00.

A study of wool-synthetic blend fabrics was made to develop one suitable for summer flight clothing.

A survey of worsted manufacturers was conducted to obtain commercially available blend fabrics that approximated the basic requirements. The results of tests performed on the fabrics obtained were considered in subsequent design and manufacture.

Identically constructed fabrics composed of blends of nylon, Dacron, Orlon, and Dynel (each blended at 15%, 30% and 60%) with wool and an all-wool control were produced; these were tested and compared with respect to fabric properties.

Particular emphasis on the flame-resistance characteristics, specifically centered on the degree of melting and "dripping" after ignition - indicated of major consequence in the establishment of this program - led to the selection of the final fabric, a 70% wool-30% Dynel fabric.

The unfavorable results obtained in this program on any of the synthetic fiber-wool blends and constructions, is not to be construed as an indication of the performance of other blends or constructions or where other parameters of tests and performance are stipulated.

Lowell Technological Institute Research Foundation. WADC TR 55-343.
PERFORMANCE CHARACTERISTICS OF PAPER HONEYCOMB CUSHIONING MATERIALS IMPACTED UNDER A HEAVY WEIGHT HIGH IMPACT SHOCK MACHINE. Edward N. Sabbagh. AF 18(600)-127. January 1956. PB 121183. Order from LC, Mi $3.90, Ph $10.80.

Dynamic tests with respect to the energy absorption characteristics of certain paperboard honeycomb materials impacted under a heavy weight high impact shock machine show that these materials are more efficient energy absorbers at the lower limit of the imposed test conditions than other materials previously tested. They are much less effective in the higher portions of the test range.

A correlation between energy absorption and density is exhibited.

Cheney Brothers. WADC TR 55-432.
DEVELOPMENT OF DACRON PARACHUTE MATERIALS. Hamilton J. Bickford, Donald K. Kuehl, Thomas L. Rusk, Jr. AF 33(616)-2562. February 1956. PB 121187. Order from OTS, $1.50.

Dacron fabrics can provide excellent dimensional stability up to 350°F and possibly 400°F.

Dacron parachute fabrics do not lose appreciable strength on exposures at 350°F up to 16 hours.

Losses in strength will occur on long exposures at 400°F.

Proper design will allow the attainment of almost any air permeability range (with reasonable tolerances) for Dacron fabrics ranging from 2 ozs/sq yd up to at least 14 ozs/sq yd. The air permeability range of fabrics weighing over 14 ozs/sq yd is limited by the amount of sleaziness acceptable in the fabric. Permeabilities over 50 cu ft/min/sq ft at 1/2 inch pressure differential can be achieved, but only by using an open weave that distorts too readily to be practical.

Seam efficiencies of 90% cannot consistently be obtained on Dacron fabrics with the present knowledge of seam performance.

Dacron can be processed to provide a balanced fabric (coinciding load-elongation curves within 15%).

WADC TR 56-15.
THE EFFECTS OF GAMMA RADIATION ON TEXTILE MATERIALS. Joyce McGrath, R. H. Johnson. February 1956. PB 121206. Order from OTS, $1.00.

The primary purpose of this investigation was to obtain data on the effect that exposure to high energy gamma radiation, for specific periods of time, would have on various parachute textile materials, also to determine if the use of parachute marking ink would increase or speed up the degradation of the materials.

The materials were irradiated both in the Cobalt-60 source located in Materials Laboratory, Wright Air Development Center, WPAFB, Ohio, and the Materials Test Reactor, National Reactor Testing Station, Idaho Falls, Idaho. All materials were evaluated both before and after exposure.

The white nylon fabrics showed the greatest loss in strength after exposure for sixteen hours at a total dosage of 7.4×10^6r, (over 25% loss), while the Dacron* fabrics retained a high percentage of strength for the same exposure (less than 7% loss). The loss in strength of both nylon and Dacron fabrics marked with parachute marking ink, was no greater than for the same fabrics that were not marked with the ink.

All webbings and cords had a high percentage of loss after exposure for 18.3 hours with a total dosage of 1.7×10^8r. In this group the nylon webbing, Spec. Mil-W-4088, Type XIII, resin treated gave the best results, with a loss of 26-28%. All types of cord showed a loss in strength of 45 to 53%.

Nylon fabric from parachutes which were exposed to varying degrees of thermal and nuclear radiation in tests at the Nevada Proving Grounds showed very slight degradation except in areas where identification nomenclature had been stamped. (Spec. Mil-I-6903 Ink, Marking, Blue). In several instances the identification area was burned or melted out in the outline of the identifying cipher or letter. In others the area was so completely damaged that identification was impossible.

*E. I. du Pont de Nemours' fiber.

Georgia Institute of Technology. WADC TR 55-264.
HANDBOOK OF PARACHUTE TEXTILE MATERIALS AND PROPERTIES. James W. McCarty. AF 33(616)-2473. February 1956. PB 121100. Order from OTS, $4.50.

This report contains the summarized and consolidated information extracted from several WADC Technical Reports covering several phases of parachute-textile research. The information is arranged to make the results of these reports more readily available and more useful to parachute designers and others interested in the parachute-textile phase of Air Force work.

The report is divided into sections covering the pertinent facets of parachute-textile information. Fairly complete information is supplied on elongation, elastic recovery, energy absorption, porosity and air permeability, strength properties, and temperature properties of numerous different textile yarns, cords, and fabrics. Some information is also supplied on aging properties, basic design data, and impact loading.

Lowell Technological Institute Research Foundation. WADC TR 54-611.

IMPROVEMENT OF COLORFASTNESS PROPERTIES ON UNITED STATES AIR FORCE FABRICS. Robert J. Peirent, George O. Langlais, Roland E. Derby, Jr. AF 18(600)-182. February 1956. PB 121051. Order from OTS, $1.75.

Dye formulae to provide improved colorfastness on the following United States Air Force fabrics were developed. When tested in a preliminary manner on laboratory materials, the formulae displayed the following colorfastness to light in comparison to that of the standards:

Cloth	Shade Number	Colorfastness of Developed Formulae Compared to Standards
Wool, Serge	Blue 84	Markedly superior
Wool, Gabardine	Blue 156	Markedly superior
Wool, Gabardine	Grey 167	Considerably better
Wool, Gabardine	Tan 193	Equal or slightly better
Nylon, Satin	Sage Green 511	Appreciably better
Nylon, Rayon	Blue 157	Equal or slightly better

The formulae developed for Blue 84 and Blue 156 withstood more than 80 hours Fadeometer exposure without producing a visual break in color.

The formula developed for Grey 167 displayed only a moderate change in color after 140 hours of Fadeometer exposure --- an exposure that destroys the color of the standard almost completely.

Tests of the colorfastness of the 100-yard length of each of the first four fabrics listed which were produced using the developed formulae in the dyeing operations substantiated the laboratory-evaluated colorfastness results with one exception:

The color on the Tan 193 length was slightly less fast than the standard.

The last two fabrics listed, i.e., those involving shades Sage Green 511 and Blue 157, were carried through to the development of dye formulae -- no fabric was manufactured.

The development of the dye formulae, the problems encountered in establishing them properly in fabric manufacture, and the test measurements of the properties of the fabrics produced are presented.

Lowell Technological Institute Research Foundation. WADC TR 55-229.

PERFORMANCE CHARACTERISTICS OF CUSHIONING MATERIALS IMPACTED UNDER A HEAVY WEIGHT HIGH IMPACT SHOCK MACHINE. E. N. Sabbagh. AF 18(600)-127. February 1956. PB 121145. Order from LC, Mi $11.10, Ph $39.60.

The energy absorption characteristics of cushioning materials impacted under a heavy weight high impact shock machine and analyzed by an analog computer system are reported. The dynamic performance curves in the form of graphs of energy versus maximum stress and maximum stress versus maximum strain are given for a wide variety of materials.

Fabric Research Laboratories, Inc. WADC TR 55-104.

STUDY OF THE EFFECT OF TWIST IN YARNS ON PARACHUTE FABRICS. Chauncey C. Chu, Milton M. Platt, Charles A. Leonard. AF 33(616)-387. February 1956. PB 121193. Order from OTS, $3.50

This is the final report on the "Study of the Effect of Twist in Yarns on Parachute Fabrics." Analytical developments on the mechanics of air flow through textile structures were made by adopting classical flow equations with due consideration to the visco-elastic behavior of textile materials. Experimental results on a large number of fabric samples (MIL-C-7020, Types I and II) with yarn twists varying from 0.5 to 35 turns per inch are given to demonstrate the various changes in the performance characteristics affected by changes in yarn geometry.

From the work accomplished, it is concluded that:

1. The flow of air through the open areas of a fabric obeys the general rules of fluid mechanics, namely: flow at any given pressure differential varies with the amount of open area; and the rate of flow at varying pressure differentials follows the square root of the pressure differential with suitable modifying constants to allow for those changes in the open area which occur when the fabric is subjected to biaxial extensions exerted by the air pressure.
2. The free area available for air flow varies as a function of fabric and yarn geometry. The ellipticity of the yarn cross section is functional with the yarn twist; flatter the yarn, the less is the open space between adjacent yarns. Hence, for a given texture (threads per inch) the free area varies inversely with the yarn width.
3. The open areas change when the fabric under test is subjected to increased pressure differentials. The yarn systems in the fabric structure, when so stressed, result in biaxial extensions which widen the spaces between yarns. The rate at which the open areas vary with pressure differential may be determined by studying the fabrics' biaxial stress-strain behavior. At the present writing only limited studies have been made.
4. The magnitude of open areas in any given fabric may be calculated from the yarn widths determined microscopically and the fabric texture. However, a more precise method has been developed by measuring light penetrability through the use of a Beckman Spectrophotometer.
5. In general, the following trends have been shown to be evident: with the increase of yarn twist

Fabric thickness	increased
Denier of yarn removed from fabric	increased
Horizontal yarn diameter	decreased
Vertical yarn diameter	increased
Free area: area between yarns	increased
Light penetrability	increased
Air permeability	increased
Freedom of yarn slippage	increased
Yarn tensile strength	increased
Yarn elongation	increased
Energy to rupture yarn	increased
Strip tensile strength	no visible change
Fabric elongation	no visible change
Secondary creep	increased
Tear strength	decreased
Tear energy	decreased

Georgia Institute of Technology. WADC TR 55-485.

AN APPARATUS FOR DETERMINING BIAXIAL STRENGTH PROPERTIES OF CLOTH AND SUPPORTING TEST DATA. Winston C. Boteller. AF 33(616)-2857. May 1956. PB 121475. Order from OTS $1.25.

The Georgia Tech biaxial fabric tension testing machine was modified to permit the simultaneous recording of warp and filling load-elongation curves. It was the purpose of these studies to determine the elastic properties of selected nylon, Orlon, and Dacron parachute-type cloth under various conditions of biaxial loading.

Tests conducted at various speeds to determine the effect of testing speed on elastic properties indicated that the testing speed has no apparent effect on the elastic properties. A comparison of elongation measurements by

extensometer and jaw separation indicates that jaw separation measurements are satisfactory for light loads, but the difficulty of securely clamping the sample introduces serious discrepancies at high loads. The measurement of elongation at various locations in the sample shows a slight increase in elongation as the extensometer is moves towards the clamping jaws.

Phoenix Trimming Company. WADC TR 55-494.
THE DEVELOPMENT OF CURRENT NYLON WEBBINGS UTILIZING 840 DENIER YARNS IN LIEU OF NOW SPECIFIED 210 DENIER YARNS. Russell J. Neff. AF 33 (600)-29034. May 1956. PB 121463. Order from OTS $1.75.

A series of nylon webbings was developed by the Phoenix Trimming Co. using 840 denier yarns in lieu of the presently used 210 denier yarns. A second series of webbings was manufactured using 210 denier yarns as required by Specification MIL-W-4088B. Comparative tests were conducted between these two constructions to determine whether the use of 840 denier yarns was feasible. In addition, four webbings were manufactured in accordance with Specification MIL-W-5625 except that a solid weave was used in lieu of tubular. These webbings were also manufactured using both types of yarn and a series of comparative tests were conducted. Five experimental high tensile strength webbings were manufactured using 840 denier yarns only. These experimental webbings were subjected to similar tests. All of the webbings were piece dyed except the webbings manufactured in accordance with Specification MIL-W-5625 and the 1" 14000 pound, 1 3/4" 25000 pound, and 3" 35000 pound experimental webbings.

This investigation has shown that 840 denies yarn appears as good and sometimes better than 210 denier yarn. Also, results have indicated that piece dyeing of the webbing is not only feasible, but desirable from the standpoints of giving better original tensile strength and abrasion resistance.

Cheney Brothers. WADC TR 55-465.
THE DEVELOPMENT OF HIGH STRENGTH NYLON PARACHUTE FABRICS. Hamilton J. Bickford, Thomas L. Rusk, Jr., Donald K. Kuehl. AF 33(600)-29135. May 1956. PB 121400. Order from OTS $1.00.

Seven nylon fabrics were developed that should give stability to dry air for use at temperatures up to 250°F for at least 16 hours with little or no significant difference in effect between continuous or intermittent exposure. Severe degradation takes place when exposed to temperatures of 350°F.

Permeability and strength requirements were met and certain information on the relationship between permeabilities at different pressure levels developed.

Seam efficiency test methods and breaking strength test methods at strengths over 500 lb/in could be improved.

Nylon fabrics can be processed to provide a balanced fabric (coinciding warp and filling load-elongation curves with 15%) for the lower breaking strength fabrics.

WADC TR 55-379.
THE EFFECT OF FIVE SYNTHETIC LUBRICANTS ON USAF FABRICS. Richard A. Sublette, 1/Lt. May 1956. PB 121395. Order from OTS $0.50

This investigation was conducted to determine if textile fibers and fabrics employed by the USAF are subject to deterioration or degradation when exposed to synthetic lubricants. In order to accomplish the desired program, two series of fabrics, composed of fibers that are commonly used in USAF fabrics, were prepared by immersing one series in a similar group of lubricants at 160°F. Both series were immersed in the lubricants for 72 hours. After exposure, laundering and dry cleaning tests were conducted on the fabrics. Diaphragm burst strength tests indicated the synthetic lubricants did not cause a loss in strength of the fabrics.

Fabric Research Laboratories, Inc. WADC TR 56-151.
RESEARCH AND DEVELOPMENT OF ABRASION RESISTANT TREATMENTS FOR NYLON WEBBINGS. George Thomson, Joseph S. Panto, Myron J. Coplan, Ernest R. Kaswell. AF 33(616)-2703. June 1956. PB 121494. Order from OTS $2.00.

The purpose of the work herein reported was the development of finishes which could be applied to nylon webbings with resulting increase in abrasion resistance. The finish was to remain flexible at -65°F and be stable to artificial sunlight for 100 hours and to a temperature of 170°F for 16 hours.

The method employed for measuring flexibility was the same as that developed under a similar investigation on Dacron Webbing as described in WADC TR 55-313.

Preference was given to commercially available water dispersions of a number of different types of resins such as acrylic, acrylonitrile, natural rubber and silicones because of their freedom from hazards of toxicity and flammability, and their ease of handling.

Webbings treated with one particular silicone and catalyst emulsion were superior to all other treated samples from the point of view of abrasion resistance, low temperature flexibility and resistant to heat aging. The application of this silicone finish did not materially affect the resistance of the nylon webbing to 190 hours exposure to artificial sunlight. This finish was consequently applied to 194 yards of Type VIII O.D. and 478 yards of Type X O.D. nylon webbing.

Fabric Research Laboratories, Inc. WADC TR 55-313.
RESEARCH AND DEVELOPMENT OF ABRASION RESISTANT TREATMENTS FOR DACRON WEBBINGS. George Thomson, Joseph S. Panto, Ernest R. Kaswell. AF 33 (616)-2563. July 1956. ASTIA Document No. AD 97103. PB 121496. Order from OTS $1.75.

The purpose of the work herein reported was the development of finishes which could be appled to Dacron webbing with resulting increase in abrasion resistance. The finish was to remain flexible at -65°F and be stable to artificial sunlight for 100 hours and to a temperature of 350°F for 16 hours.

A satisfactory method for determining flexibility of webbings at standard conditions at -65°F has been developed.

Preference was given to commercially available water dispersions of a number of different types of resins such as acrylic, acrylonitrile, natural rubber and silicones because of their freedom from hazards of toxicity and flammability and their ease of handling.

Webbings treated with one particular silicone and catalyst emulsion were superior to all other treated samples from the point of view of abrasion resistance, low temperature flexibility and resistance to heat aging. However, the resistance to artificial sunlight for 100 hours was lowered so that the webbing retained only 60 to 70% of the strength of a similarly exposed untreated sample, whereas a 90% retention was specified. In spite of this, the other aforementioned properties were so superior that it was decided to commercially apply this silicone treatment to 250 yards of Dacron webbing.

Fabric Research Laboratories, Inc. WADC TR 55-135.

DEVELOPMENT OF DACRON PARACHUTE MATERIALS. Ernest R. Kaswell, Myron J. Coplan. AF 33(600)-24087. September 1956. ASTIA Document No. AD 97241. PB 121793.

Certain Air Force deceleration parachutes are exposed to temperatures in the 350-400°F range. Nylon is severely degraded when exposed to these temperatures for relatively short time periods. Dacron exhibits significantly better heat degradation resistance. Selected threads, braids, cloths, webbings, ribbons and tapes composed of Dacron rather than nylon were designed, developed and delivered to Wright Air Development Center. All confirm the improved heat degradation resistance.

Nylon is widely used in parachutes because of its high tenacity (strength/weight ratio) and energy absorption. Dacron's tenacity and energy absorption are slightly lower. However, after heat exposure for 24 hours at 350°F these values remain far higher than for nylon. While Dacron does not degrade at high temperatures, it does shrink appreciably. This required that all Dacron items be stabilized via heat relaxation shrinkage at 350-375°F. The additional shrinkage produced elongations of about twice the order of those currently found in nylon. Furthermore, this shrinkage increased the weight/length or weight/area of the Dacron items, and thus, the strength/weight ratio was further diminished as compared with nylon.

Nylon is outstanding in its ability to deform under load and recover upon load removal without evidencing a large amount of non-recoverable elongation or permanent set. Dacron, in its originally produced state has almost as good recovery properties. However, the shrinkage incorporated into Dacron via heat relaxation manifests itself as non-recoverable elongation upon load application. Thus, at about 90% of rupture load, permanent set amounts to about two-thirds of the total elongation present in the shrunk Dacron items. At lower stress levels the amount of permenent set diminishes and the recoverable elongation increases.

It has been determined that losses in strength, elongation and energy of the subject Dacron materials after heat aging for 24 hours at 350°F amount to about 10-20%.

A significant increase in stiffness of the Dacron items occurs upon heat aging, and this was definitely ascribed to the finish applied by the Dacron filament producer. Such finishes can be removed by simple detergent scouring.

In conjunction with the development of heat stable Dacron yarns, preliminary studies indicate that a proper sequence of stretching and relaxing at below-rupture loads and at high temperatures, will produce Dacron yarn of high tenacity, normal elongation and zero heat shrinkage.

Fabric Research Laboratories, Inc. WADC TR 55-297.

DEVELOPMENT OF HIGH TENACITY-HEAT STABLE DACRON YARNS. Robert J. Coskren, Thomas T. Constantine. AF 33(600)-24087. September 1956. ASTIA Document No. AD 97242. PB 121566.

Dacron yarn, because of its superior resistance to thermal degradation when exposed to temperatures of 350-400°F for prolonged periods, has been suggested as a replacement for nylon in deceleration parachutes. Dacron's strength retention after high temperature exposure is good, but a longitudinal shrinkage of the order of 20% takes place which presents problems of parachute component dimensional stability. Secondly, this 20% shrinkage is reflected in lower strength and energy to weight ratios, thus requiring proportionately heavier parachutes. Furthermore, the added elongation resulting from thermal shrinkage is composed primarily of secondary creep or permanent set. Upon deployment of the parachute the possibility exists that fabric components might deform at the time of stress application, but not recover upon stress removal unless and until the parachute or its components are again elevated to the 350°F level.

At the inception of this phase of the study there was available from the duPont Company high tenacity (6.1 grams/denier), nominal rupture elongation (9.2%), high thermal shrinkage (20%) Dacron yarn. By free relaxation at an elevated temperature this yarn could be converted to medium tenacity (4.8 grams/denier), high elongation (36%), low shrinkage (< 2%) yarn.

Preliminary experimentation showed that cyclical yarn stressing and relaxing processes at elevated temperatures would produce a yarn of the desired high tenacity, nominal rupture elongation and low shrinkage, provided that the yarn was allowed to relax completely after the last stressing cycle.

In an attempt to develop Dacron yarn of optimum properties, those factors which were found to have an influence on ultimate properties, were thoroughly investigated. These included stretching temperatures, times, and amounts and sequences of stretching-relaxing systems.

The optimum process so far developed consists of three basic steps, namely:

1. 20% hot stretch at yarn temperatures of 340-390°F.
2. Fixed length at 430-450°F.
3. Free shrinkage at 350°F.

Yarn produced by such a process has a tenacity of 6.7-7.0 grams/denier, an elongation of 14-16% and a shrinkage of less than 2% at 350°F.

This process is an improvement over the multiple cycle methods originally studied. Furthermore, it is undoubtedly cheaper in terms of both equipment cost and production.

Concomitant with the research, a pilot processing machine was developed and some consideration was given to the commercial practicability of the process insofar as production speed was concerned.

It is recommended that this study be continued in order to further determine those factors which influence the development of an optimum yarn. Also, the ultimate design and construction of a commercial prototype machine is needed in order to produce yarn in sufficient quantities so that end items may be prepared and thoroughly evaluated.

School of Textiles, North Carolina State College. WADC TR 55-340.

A STUDY OF THE EFFECTS OF CHEMICALS ON THE PROPERTIES OF PARACHUTE FABRICS. J. Glenn Templeton. AF 33(616)-2530. September 1956. ASTIA Document No. AD 97243. PB 121674.

During routine inspection of parachutes, several badly degraded nylon parachutes were discovered. Preliminary evaluation by chemical analysis indicated the presence of mineral acids. To provide background data, nylon and Dacron fabrics were exposed to sulfuric, hydrochloric, nitric, phosphoric, sulfurous, hydrosulfuric, and nitrous acids. The exposures were conducted under various concentrations for periods of time up to six months. The evaluation was conducted by establishing the breaking strength of the exposed fabrics and comparing them to the original breaking strength.

Additional phases of the work consisted of studying the effect of light in combination with the acids, studying the effect of sulfur dioxide, hydrogen sulfide, and oxides of nitrogen, and examinations of procedures for detecting, by visual means, degradation by acids.

Dacron was found to be quite resistant to attack by mineral acids in comparison with nylon. Acid-treated nylon was more sensitive to light than acid-treated Dacron.

Lowell Technological Institute Research Foundation. WADC TR 54-612.

DEVELOPMENT OF DYEING FORMULATIONS FOR WOOL/SYNTHETIC BLENDS FOR USAF SHADE BLUE 84. Robert J. Peirent, Adolph Katz. AF 33(600)-16396. October 1956. ASTIA Document No. AD 110419. PB 121756.

Dyeing formulae were developed for viscose, nylon, Dacron, Dynel, Acrilan and Orlon fibers to obtain suitable Blue Shade #84 wool/synthetic fabrics for U. S. Air Force uniforms. At the time that the development phase was terminated, no formulae were found which would give the desired colorfastness properties for Dynel and Acrilan. Consequently, these fibers were eliminated from the production phase of the project. (Subsequent developments have demonstrated that new techniques will afford adequate fastness on these fibers. However, the project had progressed to a point which precluded reconsideration of these fibers.)

A 100% wool control fabric and a series of wool/synthetic fabrics containing 10, 20, and 30% synthetic fiber were manufactured for subsequent evaluation of their physical and colorfastness properties. These fabrics were made to conform to the specifications set forth in MIL-C-849, Cloth, Wool, Serge, Blue Shade 84.

A comprehensive study of the properties of the wool and the wool/synthetic fabrics reveals that they meet the physical requirements desired and have adequate colorfastness as well as high resistance to fading.

The scope of this study was too broad to permit examination of all of the dyestuffs produced domestically or at all of the possible dyestuff combinations which conceivably would meet target properties; however, as many dyes and formulae were included as possible. Due to the rigid requirements of this investigation, many dyes and formulae were evaluated in conditions for which they were not intended. Hence, it must not be assumed that the results tabulated herein are equally valid for other test conditions or applications, nor is it to be construed that a dye or formula is not entirely satisfactory for the manufacturers intended use or advertised claims. Further, it is not to be construed that formulae other than those covered in this report cannot perform equally satisfactorily. The disclosure of dye formulae, dyeing procedures, and methods of colorimetry described herein does not constitute license for practice. The selection of a particular dye formula for producing fabric required in this project does not imply approval of the U. S. Air Force for the specific dye or formula for producing Blue Shade 84.

WADC TR 56-257.

DEVELOPMENT OF STATIC LINE WEBBING FOR THE T-10 PARACHUTE SYSTEM. Peter Y. Stanton, 1/Lt. November 1956. ASTIA Document No. AD 110570. PB 121848.

The purpose of this program was the development of static line webbing for the T-10 parachute system. The objective of this development is to replace the presently used inferior static line webbing with webbing having more satisfactory qualities. A silk webbing and linen webbing were developed along with six nylon webbings each of which had a different elongation. Subsequent to their development the webbings were forwarded to the Parachute Branch, Equipment Laboratory, for further evaluation.

The use of silk in static line webbing does not appear feasible because of the unavailability of raw silk. The use of linen in static line webbing was unsatisfactory because of the low elongation, low energy absorption qualities of linen. The developed nylon webbings appear satisfactory for use as static line webbing.

School of Textiles, North Carolina State College. WADC TR 55-340. Sup 1.

A STUDY OF THE EFFECTS OF CHEMICALS ON THE PROPERTIES OF PARACHUTE FABRICS. David M. Cates. AF 33(616)-2530. November 1956. ASTIA Document No. AD 110524. PB 121679s. Order from OTS $0.75.

The results given in the Technical Report WADC TR 55-340 were obtained by determining the percent loss in strength of nylon and Dacron parachute fabrics when the fabric was treated with chemicals under various conditions. Because fabrics are not absolutely uniform and experimental procedures are not perfectly reproducible, the results of breaking strength tests varied. Since each sample consisted of several breaking tests (usually 10), it was possible to estimate the within sample reliability by statistical means. This was done by calculating the 95% confidence limits.

School of Textiles, North Carolina State College. WADC TR 56-288.

A STUDY OF THE EFFECTS OF CHEMICALS ON THE STRENGTHS OF NYLON AND DACRON PARACHUTE FABRICS. David M. Cates. AF 33(616)-2891. November 1956. ASTIA Document No. AD 110558. PB 131030. Order from OTS $6.00.

The effect of certain organic solvents and solutions of inorganic substances on the strength of nylon and Dacron parachute fabrics was investigated. The fabrics were exposed to each of the reagents and then aged under different conditions for a period of six months. Breaking strength tests were made on the fabrics at monthly intervals. For each testing condition, the concentration of reagent, aging temperature, and aging condition were varied. Aging was carried out under three kinds of conditions: namely, with the fabrics immersed in solution; with the fabrics first immersed in the solution, then extracted and aged under low humidity conditions; with the fabrics first immersed in solution, then extracted and aged under high humidity conditions.

It was found that in general, an increase in concentration, temperature, or time gave higher strength losses of both nylon and Dacron fabrics. In most instances the same trend in loss of strength was observed whether the fabrics were aged in solution, at high relative humidity, or at low relative humidity. Finally, the data showed that the reagents could generally be ranked in three groups according to the effect produced in the fabric: (1) reagents causing small strength losses even at high concentrations and high temperature; (2) reagents causing large strength losses at high concentrations and high temperatures; and (3) reagents causing large strength losses even at low concentrations and low temperature.

Wyandotte Chemicals Corporation. WADC TR 56-541.

EFFECTS OF FUNGISTATS ON PARACHUTE SUSPENSION LINE LUBRICANT PERFORMANCE. Theodore L. Erikson. AF 33(616)-3311. April 1957. ASTIA Document No. AD 118229. PB 131060.

Nylon parachute canopy cloth is subject to occasional failure due to "line burns" produced by sliding of the suspension lines over the canopy during opening. As a means of reducing these failures, ten candidate lubricants

were selected by Lowell Technological Research Foundation under Contract No. AF 18(600)-136, (WADC TR 54-323). These lubricants were, however, found subject to mildew attack, and the present work was undertaken with the object of determining their lubricating properties on addition of a maximum of 2% of each of three specified mildew inhibitors.

A maximum lubricant loading of approximately 10% by weight of the suspension line was desirable. Values of 9.5±0.5% were obtained with all inhibitor combinations of nine of the ten lubricants by deposition from homogeneous trichlorethylene solutions. One lubricant consisted of emulsified lubricant in water.

Mildew inhibition tests were performed according to a specific procedure developed at WADC and resulted in the elimination of all but two candidate combinations. These candidate combinations showed no decrease in lubricity from the uninhibited lubricants when subjected to "friction fusion" tests on apparatus developed under Contract AF 18(600)-136, and little or no decrease in lubricity after oven aging.

Breaking strength tests performed by the Materials Laboratory, WADC, showed that one of the two combinations was detrimental to the breaking strength of the suspension line when subject to oven aging. The surviving combination consisted of Spermaceti wax plus 2% dialkldimethylammonium bromide (Isothan DL-1).

WADC TR 56-256.
EVALUATION OF DUPONT AND CHEMSTRAND NYLON YARN. Peter Y. Stanton, 1/Lt. April 1957. ASTIA Document No. AD 118227.

The investigation undertaken herein was two phase, namely:

1. To compare nylon yarns of various deniers manufactured by the E. I. duPont de Nemours and Company with nylon yarns of the same denier manufactured by the Chemstrand Corporation.
2. The service testing of personnel parachutes of which the canopy material was fabricated from Chemstrand nylon and the remainder of the parachute fabricated from duPont nylon as a comparison to the same type parachutes fabricated wholly from duPont nylon.

A comparison of the nylon yarns manufactured by duPont with the nylon yarns manufactured by Chemstrand shows no meaningful difference between the two with regard to breaking strength, tenacity, elongation, energy absorption and melting point. Also, a comparison of the high and low temperature characteristics of the yarns manufactured by both companies shows no real difference between the two with regard to the properties investigated.

Service tests have shown that the parachutes fabricated from Chemstrand yarn in the canopy are equal in performance to those manufactured wholly from duPont yarn.

Fabric Research Laboratories, Inc. WADC TR 56-576.
DEVELOPMENT OF DESIGN DATA ON THE MECHANICS OF AIR FLOW THROUGH PARACHUTE FABRICS. William G. Klein, Charles A. Lermond, Milton M. Platt. AF 33(616)-2977. September 1957. ASTIA Document No. AD 131055. PB 131431. Order from OTS $2.75.

The purposes of this report are two: (1) A determination of the factors involved in parachute fabric permeability and the quantitative prediction of their influence with a view to rational engineering design of such materials; and, (2) An assay of a representative selection of comercially produced parachute fabrics (Type I and Type II) to determine the degree to which they meet current permeability specifications and, where such requirements are not met, the reasons therefore.

The theoretical investigation of the mechanics of airflow is prosecuted as follows:

A. A classical fluid flow relation is restated in terms of fabric parameters.
B. A number of experimental and theoretical relationships are formulated.
C. The various relationships are combined to give the necessary information for checking the validity of the theoretical approach and assumption made.

The results of the various analyses support to a large extent the approach and mechanism of solution, but the work to date has been analytic only. The problem of snythesis can be rationally attacked only after the compilation of more information on the biaxial deformation of fabric structures.

The commercially produced fabrics showed a wide variation in permeability. Only 60% of the Type I and 47% of the Type II fabrics met the permeability specification of MIL-C-7020 A under which they were manufactured. In all cases the reasons for the deviations were explainable on the basis of yarn diameters and picks and ends.

The Connecticut Hard Rubber Company. WADC TR 57-416.
THE DEVELOPMENT OF A COATING FORMULATION AND METHOD OF APPLICATION FOR USE IN NYLON DOUBLE FABRIC. Ernest H. Pagliaro. AF 33(616)-3901. November 1957. ASTIA Document No. AD 142094. PB 131651. Order from OTS $1.00.

A two ply modified plain weave nylon fabric has been successfully coated on both sides using conventional knife over roll rubber spreading equipment. Blocking between the plies was prevented by closely controlling the solids concentration and the amount of compound deposited per pass. Butyl rubber was selected over the other elastomers evaluated because it was the most flexible when tested at -65°F according to requirements of the contract. Specimens coated with Butyl rubber have also met the other necessary requirements including adhesion, weight, and air retention.

Cornell University. WADC TR 57-157.
AERODYNAMIC HEATING OF PARACHUTES. A. L. Ruoff, S. W. Liu, F. Frank. AF 33(616)-3572. December 1957. ASTIA Document No. AD 142261. PB 131597. Order from OTS $1.75.

Heating rates for a parachute employed at varying Mach numbers (2 - 5) and altitudes (sea level to 100,000 feet) are obtained. Various methods of protecting the nylon parachute are discussed. Although requiring further engineering development the following methods are theoretically feasible: Coating with a silicone foam which reduces the heat transfer rate to the nylon is possible; use of a sublimating coating, e.g., hexachlorethane is feasible; and the use of water absorbed in a polyurethane foam coating on the nylon is possible.

Plots are given for the heat fluxes at various points. Since the actual total heat flux in a given case depends on the deceleration rate of the specific parachute only specific cases could be considered. However, the method is described, whereby, using the data of this report the amount of evaporating material required to keep the nylon at a safe temperature can be very readily calculated when a specific time-velocity profile is given.

Massachusetts Institute of Technology. WADC TR 57-443.
DESIGN DATA ON BIAXIAL FORCES DEVELOPED IN

PARACHUTE FABRICS. Jan G. Krizik, Ebrahim Victory, Joseph F. Cheatham, Stanley Backer. AF 33 (616)-3253. December 1957. ASTIA Document No. AD 142208. PB 131658. Order from OTS $2.25.

A high pressure permeometer has been constructed for use with parachute cloths over a differential pressure range of 0.5 to 1000 inches of water. The unit consists of a compressor, test duct with special biaxial stress jaws, and a steam ejector in series. Operation with variable air densities is possible to simulate high altitude parachute operation. A range of standard and experimental parachute materials has been tested on the permeometer and extensive data are available relating air flow, biaxial stress and strain and area increase to pressure differentials across the fabric. Data showing the effect of sample prestressing, cyclic testing and average air density are included.

Phoenix Trimming Company. WADC TR 57-538.
DEVELOPMENT AND EVALUATION OF WEBBING MADE FROM NYLON "6". Russell J. Neff. AF 33(600)-33484. March 1958. ASTIA Document No. AD 151090. PB 131832. Order from OTS $1.50.

The purpose of the work herein reported was the development and evaluation of a series of nylon webbings utilizing Type "6" nylon.

A group of webbings were manufactured. Ten were made using 210 denier yarn and four were made using modified 840 denier yarn. The webbings were in natural and OD color, untreated and treated, made in accordance with Specification MIL-W-4088C and Specification MIL-W-5625. It should be noted that a solid weave was used in lieu of a tubular under Specification MIL-W-5625 and that the number of ends and yarn ply of the 1/2", 9/16" and 5/8" webbings were in accordance with the constructions used in WADC Technical Report 55-494. A series of tests were conducted to determine if the use of Type "6" nylon was feasible in Air Force webbings.

This investigation showed that the webbings manufactured from regular 210 denier Type "6" nylon have slightly lower breaking strength and were susceptible to heat degradation at lower temperature than webbings presently being used by the Air Force which utilize the Type "66" nylon. The webbings manufactured using the Type "6" modified 840 denier yarn appeared to be equal in strength and superior in their resistance to heat degradation up to a temperature of 300°F.

Celanese Corporation of America. WADC TR 58-30.
DEVELOPMENT OF AN OUTER SHELL AND A FUNCTIONAL CLOTHING FABRIC. Joseph L. Barach, Arthur S. Tingas. AF 33(600)-32556. April 1958. ASTIA Document No. AD 151133. PB 151138. Order from OTS $2.00.

The purpose of this report is to summarize the experimental work performed and the technical data obtained in developing an Outer Shell and a Functional Clothing fabric for Wright Air Development Center. In the course of this program, a large number of experimental fabrics were prepared, dyed, finished, and evaluated. Details of fabric preparation, dyeing and finishing procedures followed, and results obtained are given in this report.

The most difficult problems encountered during this work were those of dyeing the various fiber blends and combinations to match the requested color shade and of finishing the Outer Shell fabrics for combined water repellency and flame retardancy.

Two fabrics were selected as the constructions most closely meeting the requirements. A Dacron/rayon blend for the Functional Clothing fabric and a Fortisan/cotton combination for the Outer Shell fabric. In addition, other constructions of interest were developed such as nylon/Verel, nylon/cotton, nylon/rayon, Fortisan/Dacron, Fortisan/Verel combinations and a nylon/rayon intimate fiber blend.

Fabric Research Laboratories, Inc. WADC TR 57-765.
DEVELOPMENT OF HIGH TENACITY-HEAT STABLE DACRON PARACHUTE ITEMS. Chauncey C. Chu, Ernest R. Kaswell, Donald J. Doull. AF 33(616)-3593. May 1958. ASTIA Document No. AD 155511.

A Pilot machine has been designed, constructed, and used for the production of "Hot Stretched-Heat Relaxed" (HS-HR) Dacron yarns and threads. Yarns so produced were converted, where necessary, into selected end items, namely webbings, braid, and tape.

The HS-HR process consists of hot stretching the yarns over heated surfaces at 375° to 400°F, and then relaxing the yarns at a surface temperature in approximately the same range. Considerable difficulty was encountered in producing in proper quality, the relatively large quantities of yarns needed for the selected end items, and much experience was gained in this ultimate accomplishment.

A previous investigation, WADC TR 55-135, reports on the properties of counterpart items prepared from "Heat Relaxed" (HR) Dacron yarns. The HR process consists of merely freely relaxing the yarn at 375° to 400°F.

Comparison and evaluation of the HS-HR and the HR processes with particular reference to their use for Dacron parachute component are the subject of this current work. Unprocessed producer's type 5100 Dacron (U) has high tenacity, low elongation and high heat shrinkage. Heat Relaxes (HR) Dacron has low tenacity, high elongation, and essentially no shrinkage. Hot Stretched-Heat Relaxed (HS-HR) Dacron has high tenacity, intermediate elongation, and essentially no shrinkage.

HS-HR Dacron meets essentially all of the end item specifications originally delineated for nylon. In addition it has better heat degradation resistance than nylon, better heat stability than producer's Dacron, and higher strength per unit weight than Heat Relaxed (HR) Dacron.

In a Dacron arrestation parachute the relationships among deformation upon deployment, subsequent recovery, subsequent heat stability, and utility for repeated deployment, are not completely known. The HS-HR process appears to have some advantages over the HR process, but whether it is worth the additional effort and cost, can only be decided after additional study.

HS-HR end items have been delivered to WADC for comparative field evaluation with previously delivered HR end items.

Fabric Research Laboratories, Inc. WADC TR 58-65.
RESEARCH PROGRAM FOR THE DEVELOPMENT OF A DESIGN PROCEDURE TO ENGINEER PARACHUTE FABRICS. William G. Klein, Charles A. Lermond, Milton M. Platt. AF 33(616)-3845. May 1958. ASTIA Document No. AD 155517. PB 151209. Order from OTS $1.75.

This report presents an analysis which it is felt comes closer to defining the mechanisms of air flow through parachute materials than previous attempts have done. The significant difference from prior work is that the fabric is treated as a three rather than two-dimensional structure, thus permitting a more accurate characterization of the actual flow region.

TR 4604.
MONOMERIC CEMENT FOR BONDING ACRYLATE PLASTICS E. J. Wyrostek. January 1941.

TR 5108.
MEASUREMENT OF OPTICAL HAZE IN TRANSPARENT MATERIALS. Leah K. Hendriksen. April 1944.

Battelle Memorial Institute. TR 52-38.
ELEVATED- AND ROOM- TEMPERATURE PROPERTIES OF TRANSPARENT ACRYLIC SHEET MATERIALS. John VanEcho, Gale R. Remely, and Ward F. Simmons. AF 33(038)-10818. February 1952. PB 118802. Order from LC, Mi $3.00, Ph $6.30.

Two regular grades of transparent acrylic sheet, Plexiglas Ia and Lucite HC-201, and two heat-resistant grades, Lucite HC-202 and Plexiglas II, were tested in tensile creep and creep rupture, crazing, short-time tensile, and deterioration at room temperature, 160°, and 200°F.

The creep and creep-rupture tests indicated that the heat-resistant variety was considerably stronger than the regular grade, even at room temperature. At 160° and 200°F the superiority of the heat-resistant grade was much greater. At 200°F the regular acrylate sheet did not have any practical load-carrying ability.

The heat-resistant material was also much superior to the regular acrylate sheet in resistance to crazing at elevated temperatures. A linear relationship exists between temperature and stress for incipient crazing in the heat-resistant acrylate over the temperature range of this investigation. The stress to produce crazing decreased about 15 psi for each degree increase in temperature.

Forest Products Laboratory, Forest Service, U. S. Department of Agriculture. TR 52-185.
DEFLECTION AND DISTRIBUTION OF STRESSES IN THE FACINGS OF A CENTRALLY LOADED TRANSPARENT SANDWICH BEAM. Wilhelm S. Ericksen. AF 18(600)-70. August 1952.

Rohm and Haas Company. TR 6629, Section 2.
RESISTANCE TO SHATTERING BY GUNFIRE OF TRANSPARENT LAMINATED MATERIALS FOR AIRCRAFT GLAZING. E. M. Petruska. AF 33(038)-23269. September 1952.

Two hundred thirty-nine (239) acrylic-butyral laminates have been tested under uniform conditions. These included seven configurations of both Plexiglas II and Lucite HC-202, supplied by two laminators, Pittsburgh Plate Glass Co. and Glass Products Division of American Window Glass Co.

Battelle Memorial Institute. TR 52-292.
ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF SELECTRON 44 AND 5105XP TRANSPARENT PLASTIC SHEET MATERIALS. John VanEcho, Gale R. Remely, and Ward F. Simmons. AF 33(038)-10818. November 1952.

Two transparent plastic sheet materials, Selectron 44, a polyester, and 5105XP, an acrylate, were tested in tensile creep and creep rupture, crazing, short-time tensile, and deterioration at 80°, 160°, and 200°F.

The creep and creep-rupture tests indicated that the 5105XP material had considerably more strength than Selectron 44 at all three test temperatures. The short-time tensile tests gave a similar indication at 80°, 160°, and 200°F, but at 250°F Selectron 44 appeared to have a slight superiority in strength. At 300°F, 5105XP had no practical load-carrying ability at all, while Selectron 44 showed a tensile strength of only 204 psi.

Selectron 44 displayed no crazing whatsoever at any temperature or strain rate. The crazing strength of 5105XP was, in general, equal to or greater than the rupture strength of Selectron 44 at all three test temperatures.

Bjorksten Research Laboratories, Inc. TR 52-304.
DEVELOPMENT OF HEAT-RESISTANT INTERLAYER MATERIALS FOR LAMINATED GLASS. Johan Bjorksten, Luther L. Yaeger, and Robert J. Roth. AF 33(038)-16240. December 1952. ASTIA Document No. AD 20093.

Of the various materials investigated, Mylar (duPont polyethylene glycol terephthalate) was the most effective heat resistant interlayer for aircraft laminated glass. It withstands 400°F. for as long as two hours without apparent deleterious effects. Mylar-glass laminates maintain their shatter resistance throughout the temperature range of -65° to 400°F. and under differential pressures and temperature gradients simulating flight conditions.

Other polymers, which were not as satisfactory as Mylar because of insufficient thermal stability and/or poor optical properties, included:

Cross-linked polyvinylbutryal
Alcohol soluble polyamid (Nylon, type 8)
Silicone elastomer
Chlorofluoro polymer (Kel F)

Epoxy type adhesives produced the best bonding between Mylar and glass.

In view of the developmental nature of this work, these conclusions must be regarded as tentative and subject to further investigation. Further work on Mylar interlayers for glass laminates is being performed under Contract No. AF 33(600)-22723.

Armour Research Foundation. TR 53-19.
DEVELOPMENT OF A PRACTICAL LABORATORY PROCEDURE TO BE USED IN EVALUATING THE FORMING QUALITIES OF PLASTIC SHEET MATERIALS. Paul H. Kaar. AF 33(038)-27648. September 1953. PB 120801. Order from LC, Mi $4.50, Ph $13.80.

Under the terms of the contract with Wright Air Development Center, the purpose of this research project was (1) to study the various factors which are of importance in the fabrication of flat plastic sheet material into useful shapes for a transparent aircraft enclosure, and (2) to develop a practical laboratory procedure for evaluating the forming qualities of these plastic materials. In order to achieve this objective, it was necessary to review the industrial processes and applications of the material and to determine what measurable characteristics are important in forming operations.

Generally, the investigation of a formability criterion proceeded along two separate lines. One approach was an attempt to use various standardized engineering test procedures to indicate formability. The other approach was to duplicate various manufacturers' forming operations and use data derived from these tests to evaluate forming characteristics. Apparatus development was a significant part of each approach.

This final report includes discussions and data pertinent to the selection of the formability criterion recommended and a discussion of the important factors in formability evaluation.

The formability rating system recommended consists of forming, by positive air pressure, an unconfined bubble of the plastic heated to optimum forming temperature. The rating assigned is based on the pressure required to form the bubble and the extent to which it can be formed before fracture. Plastics incapable of being stretched are rated by a bend test. Conclusions reached are as follows:

1. A suitable formability criterion for transparent plastic sheet must embody evaluation of (1) ease of forming and (2) maximum extent of forming possible.

2. Standardized engineering tests performed at optimum forming temperature and providing such data as maximum elongation, creep, and ball penetration values fail to evaluate formability satisfactorily for the following reasons:

a. Data from such tests do not correlate satisfactorily with ad hoc tests duplicating manufacturing operations.

b. Engineering tests do not provide sufficient spread in results to distinguish between plastics of similar forming properties.

3. These tests have shown that several systems can be used to evaluate the two primary formability characteristics of plastic sheet. Any of the ad hoc tests, particularly the hemispherical draw and unconfined bubble tests, can be used to evaluate the modulus of workability. The deformative maximum is more difficult to gage; only the unconfined bubble test using a lubricated specimen was judged to measure impartially this property of the plastic.

Swedlow Plastics Company. TR 53-259. F-94 ONE HALF SCALE METHYLALPHACHLORACRYLATE CANOPY. Frank Evans, M. Elber Latham, and John G. Stansbury. AF 33(616)-202. September 1953. ASTIA Document No. AD 24142. PB 122474. Order from LC, Mi $2.40, Ph $3.30.

Quarter-inch thick Methylalphachloracrylate monolithic cast sheet was satisfactorily fabricated by a "free blowing" technique into a half scale F-94 canopy. The optical quality was excellent and in every way appeared to be easily the equivalent to that of a "free blown" acrylic part (MIL-P-6886 or MIL-P-5425). However, some surface degradation was evidenced by a slight darkening. An adequate cement, as well as cementing technique, was established to assemble the edge attaching materials to the formed Methylalphachloracrylate canopy.

Forest Products Lab. TR 52-185, Suppl. 1. DEFLECTION AND DISTRIBUTION OF STRESSES IN THE FACINGS OF A CENTRALLY LOADED TRANSPARENT SANDWICH BEAM. Wilhelm S. Ericksen. AF 18(600)-70. December 1953. ASTIA Document No. AD 30551. PB 130373.

Formulas for the deflection and for the stress within the facings of a transparent sandwich beam under central load were presented in the basic issue of this report. However, photos and figures pertinent to the calculations were omitted from that report. These photos and figures are included herein as a supplement to the original issue.

Rohm and Haas Company. TR 6684. FORMING TEST FOR TRANSPARENT PLASTIC SHEET MATERIALS. E. N. Robertson and C. H. Weber. AF 33(038)-22945. December 1953.

Development of a test procedure and apparatus for use in qualification testing of transparent plastic sheet material under military specifications is described. The procedure was used to differentiate the forming characteristics of five transparent plastic sheet materials that are supplied under five different Government Specifications for use in aircraft enclosures.

The plastic materials were found to be sufficiently different in characteristics at high temperatures to have distinctly different moduli of elasticity and to require different conditions of temperatures and pressures for forming. The forming parameters (thickness, pressure, time and temperature) which will permit qualification testing to differentiate the materials are given.

Bjorksten Research Laboratories, Inc. TR 53-299. PRELIMINARY EXPLORATION OF SILICON-FLUORIDE PLASTICS FOR HIGH-TEMPERATURE-RESISTANT INTERLAYERS. Johan Bjorksten, Luther L. Yaeger, Robert P. Cox, and Robert J. Roth. AF 33(600)-23256. December 1953. PB 125204. Order from LC, Mi $2.70, Ph $4.80.

The objective of this study was a preliminary exploration of the possibilities of silicon-fluoride plastics for high-temperature-resistant interlayers. It was understood that the investigation was not to give a final and conclusive result, but rather to cover a broad scope to develop promising leads and approaches.

The following were explored experimentally and yielded transparent products with good temperature stability and high resistance to di-ester type lubricants and aromatic solvents:

1. Cross-linking a fluoroalkyl acrylate with a polysiloxane.

2. Co-polymerizing a fluorinated olefin and an unsaturated silane.

A third approach, preparing a fluoro-silicon analogue of a polyester resin, yielded an intermediate of promising appearance.

Samples of these materials have been submitted to WADC for evaluation.

Bjorksten Research Labs., Inc. WADC TR 54-57. DEVELOPMENT OF HEAT-RESISTANT INTERLAYER MATERIALS FOR LAMINATED PLASTIC AND LAMINATED GLASS. Robert P. Cox, Luther L. Yaeger, Ralph W. Buetow, Robert J. Roth. AF 33(600)-22723. July 1954. ASTIA Document No. AD 49905. PB 121665. Order from OTS, $1.25.

The following materials were considered for use as heat-resistant interlayer materials for laminated glass and laminated plastic:

Polyamides (Nylons)
Polyesters
Cellulose esters
Fluorinated resins of the Exon type
Plasticized Epon resins
Mylar
Silicone-acrylate copolymers

The optimum interlayer material was a copolymer of 99 parts of ethyl acrylate and one part of vinyl polysiloxane (Linde X-31 resin). It was prepared as a casting syrup by the photopolymerization of ethyl acrylate to a low molecular weight prepolymer and subsequent mixing with vinyl polysiloxane. Laminates were prepared by curing the interlayer casting resin between two outlayer sheets. Such laminates were prepared with glass, polyester, Plexiglas 55, Polymer K (Rohm and Haas Co.) and Gafite (General Aniline and Film Corp., polymethyl-alpha-chloro-acrylate), as surface sheet materials. These laminates exhibited slight discoloration after six hours at 350°F. and possessed satisfactory projectile and falling ball impact strengths at 0°F.

Dow Corning Corporation. WADC TR 54-207 Part 1.
DEVELOPMENT OF SILICONE OR FLUOROCARBON INTERLAYER MATERIALS FOR LAMINATED GLASS AND LAMINATED PLASTIC. Keith E. Polmanteer, John W. Cretzmeyer, John W. Erwin. AF 33(600)-23081.

The development of a transparent interlayer material for laminated glass possessing thermal stability within the range of 400° to 500°F was the object of this research by the Dow Corning Corporation during the period of 15 April 1953 to 15 April 1954. Nine polysiloxane materials were introduced and evaluated for suitability as interlayers. A Type J interlayer was found which possessed tensile strength of 400 to 800 psi, 80 to 85% transmission, 4 to 10% haze and thermal stability of several hours at 450°F.

This interlayer lends itself well to commercial fabrication techniques. The optical properties may require some improvement before this material will be completely satisfactory for aircraft glazing.

Battelle Memorial Institute. WADC TR 54-429.
ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF POLYMER K TRANSPARENT PLASTIC SHEET MATERIAL. John VanEcho, Gale R. Remely, Ward F. Simmons. AF 33(038)-10818. October 1954.

Creep and creep-rupture, short-time tensile, and deterioration data were obtained on Polymer K, a transparent, thermosetting acrylic sheet material. Tests were made at 80, 160, 200, and 250°F. A comparison of properties is made with several other transparent plastics.

The room-temperature creep and creep-rupture strength of this material is quite similar to that of 5105XP, and Plexiglas II. The creep and creep-rupture strengths at 160 and 200°F are quite superior to any other transparent tested under this program. The short-time tensile strength of Polymer K is inferior to 5105XP and Selectron 44 at room temperature but considerably greater at the higher temperature.

The deterioration losses of Polymer K are, generally, lower than those for 5105XP and Selectron 44. The test material showed a maximum of 0.7 per cent weight loss after the 1000-hour exposure at 200°F.

Goodyear Aircraft Corporation. WADC TR 54-128 Part 1.
EVALUATION OF MATERIALS FOR GUNSIGHTING DOMES. John L. Folk, Kenneth R. Iler. AF 33(616)-106. November 1954.

This report covers an evaluation of three laminated transparent acrylic materials for use in the fabrication of aircraft gunsighting domes. Regular grade (BMS 8-4), heat resistant (MIL-P7524), and modified (5105XP) acrylic laminates, all with standard polyvinyl butyral interlayer, were tested. Two laminators supplied the 5105XP test laminates.

The various testing procedures compared the laminates with respect to (1) ultimate tensile strength at -65°F, room temperature, and 160°F, (2) time required for crazing and failure under long time tensile loads of 750, 1000, 2000, and 4000 psi, and (3) resistance to natural Florida and accelerated weathering. The tests also identified suitable cements for bonding extruded rubber edge attachments of each test material and evaluated the heat stability of the interlayer material.

Of the materials evaluated, laminated 5105XP appears to be the most desirable dome material. Its ultimate tensile strength (5700 psi at 160°F, 10,600 psi at room temperature, and 19,500 psi at -65°F) and its resistance to weathering elements were found to be superior. The long time tensile tests, incomplete at this writing, have indicated to date that the 5105XP laminate will support normal service loads for a longer time than the other two test materials. (Completed data for the long time tensile tests will be submitted later in Part II of this report.)

All of the cementing systems tested would comply with the requirements of normal service specifications, even after weathering. However, a prebodied acrylic monomer type cement, such as GAC Code C-201A, was indicated to be the most satisfactory.

The heat stability (tendency to bubble at normal acrylic forming temperatures) of polyvinyl butyral was decreased with increased moisture content. The heating time and moisture content required to produce bubbling appeared to be independent of the type of laminate or the source, but the location and size of the bubbles differed in the materials obtained from the two laminators. Except for this peculiarity, the 5105XP materials showed little difference in the tested physical properties.

Armour Research Foundation. WADC TR 55-24.
DETERMINATION OF THERMAL SHOCK CHARACTERISTICS OF GLASS. David Horwitz. AF 33(616)-2240. June 1955.

This is a study of the resistance to thermal shock of glass having different coefficients of linear thermal expansion and different tempers and subjected to shock applied to a single surface. The program was initiated in order to enable the selection of a suitable glass for windshields in supersonic aircraft.

Apparatus was designed to produce a timed sudden shock to a glass specimen at elevated temperatures, simulating skin temperatures which may develop in supersonic aircraft due to aerodynamic heating.

Instrumentation was developed to record "surface" temperature of the specimen independent of the conditions of the attacking fluid.

Time-temperature change curves were drawn, and the shock rates established for the various glasses were used as a means of comparison.

Dow Corning Corporation. WADC TR 55-189.
SILICONE INTERLAYER MATERIAL PROGRAM. Keith E. Polmanteer, Francis J. Campbell, Jack Fenner. AF 33(600)-27185. June 1955.

The further development of a silicone thermally stable interlayer, originally introduced under AF Contract 33(600)-23081, was the object of this research by the Dow Corning Corporation during an eight and one-half month period ending in January, 1953. The optical properties, tensile strength and thermal stability of this silicone interlayer material were substantially improved in this work. This improved interlayer was identified as Type JA. It possessed 1.8-3.0% haze, 86.5%-87.5% transmittance, 750-1200 psi tensile strength, and thermal stability at 400°F and above for over 100 hours.

Rohm and Haas Company. WADC TR 54-619 Part 1.
THE INVESTIGATION OF MULTIAXIALLY STRETCHED ACRYLIC PLASTIC. Julius J. Gouza, Dee A. Hurst. AF 33(616)-489. July 1955. ASTIA Document No. AD 93136. PB 131933. Order from OTS, $2.75.

The effects of stretching acrylic sheet materials were determined so as to indicate the potential value of such procedures for producing transparent aircraft enclosures with properties equal to or better than laminated transparent plastic enclosures.

Specifications MIL-P-8184, 6886A, and 5425A types of acrylic plastic sheet materials have been stretched by two different methods, biaxial and multiaxial, and agred under four different conditions. A biaxial stretching machine was designed and built. Initial material thickness was chosen so as to give a final thickness of approximately 0.25 inch after 0, 25, 50, 75, and 100 percent stretch at temperatures between 248° and 284°F as required for a particular material.

There was no significant difference between the effects of biaxial and multiaxial stretching. Specification MIL-P-8184 type acrylic plastic stretched 75 percent equally in two directions possessed the best properties before and after aging. All stretched materials were better than unstretched in resistance to crazing, impact strength, and notch sensitivity but inferior in resistance to abrasion.

General Aniline and Film Corporation.
WADC TR 54-465.
STABILIZATION OF POLYMETHYL ALPHA CHLOROACRYLATE PLASTIC SHEET. Harry D. Anspon.
AF 33(600)-23883. August 1955.

When this contract was initiated, it already had been shown that polymethyl α-chloroacrylate plastic sheet had outstanding physical properties - high heat distortion temperature, high tensile and flexural strengths, excellent craze resistance, low notch sensitivity, unique self-extinguishing property in burning tests, and complete formability - which made it particularly suited for use as an aircraft glazing material especially if its heat and light stability could be improved.

In order to obtain this plastic as a completely acceptable aircraft glazing material, Wright Air Development Center awarded this contract for the improvement of the heat and light stability of polymethyl α-chloroacrylate plastic sheets.

As a result of the work completed under this contract, polymethyl α-chloroacrylate plastic sheets possessing outstanding heat and light stability were prepared and were submitted to Wright Air Development Center.

This report summarizes the research and development work involved in obtaining these heat and light stabilized polymethyl α-chloroacrylate plastic sheets.

The detailed outstanding properties of polymethyl α-chloroacrylate (PMACA) established by testing programs on previous samples of polymethyl α-chloroacrylate plastic sheets are summarized below.

1. PMACA possesses a heat distortion temperature of 130-140°C (266-284°F).
2. PMACA possesses a tensile strength of 17,000 psi at room temperature and even at 110°C (230°F) it possesses a tensile strength of 8,000 psi.
3. PMACA possesses a flexural strength of 23,000 psi at room temperature.
4. Even though PMACA is quite hard (Barcol Hardness of 65), it still exhibits excellent strength properties at room temperature after notching; for its notched tensile strength is 12,000 psi and its notched flexural strength is 13,000 psi.
5. PMACA exhibits good resistance to solvent and stress crazing. PMACA samples withstood 11,000 psi flexural stress for 23 hours before crazing and 5,000 hours before failing in a long time flexural test. They also withstood 9,000 psi stress under toluene, 8,000 psi stress under isopropyl alcohol, and 4,500 psi stress under methyl ethyl ketone, for over 300 seconds without crazing.
6. PMACA is completely formable. At 177°C (350°F) a 6 in. diameter hemishperical dome was blown from 1/4 in. thick sheet with 18 psi air pressure.
7. PMACA is rapidly self-extinguishing. When tested for flammability by Method 2021 of Federal Specification L-P-406a, the flame extinguished immediately after removal of the external burner flame.

Stabilization of PMACA plastic sheets against heat and light is believed to overcome the principal deterrent to the use of PMACA, with its superior properties as an aircraft glazing material.

Goodyear Aircraft Corporation. WADC TR 54-128 Part 2.
EVALUATION OF MATERIALS FOR GUNSIGHTING DOMES. Pt. 2. Long Time Tensile Strength Evaluation. Charles R. Frownfelter, Duane C Shearer. AF 33(616)-106. October 1955.

This report covers a series of long-time tensile tests on laminated transparent plastic materials suitable for forming gunsighting domes. These tests were part of an evaluation which also included (Reference Part 1 of this report) adhesive tests, heat stability tests, and ultimate tensile tests at various temperatures. The materials investigated were regular grade laminated acrylic (BMS8-4), heat resistant grade laminated acrylic (MIL-P-7524), and modified acrylic face sheets (5105XP) laminated with a standard polyvinyl butyral interlayer. The 5105XP laminate was supplied by two laminators.

The long-time tensile tests consisted of subjecting specimens of the above laminated materials to constant stresses of (nominally) 4000, 2000, 1000, and 750 psi for periods up to 16 months under two conditions: (1) outdoor exposure in South Florida, and (2) indoor room temperature exposure at the Goodyear Aircraft plan in Akron.

From the standpoint of time to fail at a given stress, laminated 5105XP was the best of the materials by a wide margin, having withstood 1000 psi for at least 11,000 hours in Florida and about 2000 hours at room conditions. Natural weathering in Florida also promoted delamination between the face sheets and the butyral interlayer, particularly in the laminated 5105XP material supplied by one of the two laminators. and in the BMS 8-4 material.

Battelle Memorial Institute. WADC TR 55-412
ELEVATED AND ROOM TEMPERATURE PROPERTIES OF SIERRACIN 611, PMACA, AND PLEXIGLAS 55 TRANSPARENT PLASTIC SHEET MATERIALS. John A. Van Echo, Ward F. Simmons, Gale R. Remely. AF 33(039)-10818. December 1955.

Creep and creep-rupture, short-time tensile, and deterioration properties of two transparent plastic sheet materials, Sierracin 611, a polyester, and PMACA, an acrylate, were determined over a temperature range of 80 to 300°F. Some creep and creep-rupture and tensile data were also obtained on Plexiglas 55, an acrylic transparent, for comparison with the strength properties of an experimental acrylic, 5105XP.

The PMACA sheet material displays, in general, at least 50 per cent greater creep, creep-rupture, and short-time tensile strength and elastic-modulus properties than do Sierracin 611 and Plexiglas 55. This PMACA material compares almost equally as favorable with other transparent plastics previously tested under this evaluation program, such as acrylates Polymer K, 5105XP, Lucite HC-202, and Plexiglas II and the polyester resin Selectron 44.

The deterioration losses determined for PMACA and Sierracin 611 remained relatively low for temperatures as high as 250°F. Of the three transparents tested, crazing was shown to occur in the PMACA and Plexiglas 55 materials.

Monomer-Polymer. WADC TR 55-296. RESEARCH, DEVELOPMENT, AND FABRICATION OF SILOXANE-ACRYLATE INTERLAYER MATERIAL. B. David Halpern, Leroy Tibery, Wolf Karo, William Prosk, John Isler, Jr. AF 33(616)-2528. December 1955.

The object of this research, development, and fabrication program was for the production of interlayer sheet material. This interlayer material consisting of a copolymer of 99 parts of ethyl acrylate and one part of vinyl polysiloxane was developed under Air Force Contract 33(600)-22723 and is described in WADC TR 54-57.

This developmental work was undertaken between 15 June 1954 and 15 June 1955.

Attempts to mill and calender the interlayer material were unsuccessful. This necessitated the production of interlayer sheets by casting between glass plates. Thermal polymerization was substituted for ultraviolet polymerization for quality production purposes. Sheets produced by this method, however, were optically imperfect.

An adhesive was required for laminating the interlayer sheet to glass or plastic. Residual traces of the parting agent, however, prevented good lamination.

Seven 0.200" by 37" by 42" interlayer sheets varying in quality were submitted.

Recommendations are given for methods of improving the optical quality of future interlayer sheets.

Markite Company. WADC TR 55-272. DEVELOPMENT OF ELECTRICALLY CONDUCTIVE TRANSPARENT COATINGS FOR ACRYLIC PLASTIC. Myron A. Coler, Arnold S. Louis, John C. Kalbach. AF 33(616)-2027. January 1956. PB 121208. Order from OTS, $2.50.

The Marklad process, a procedure involving repeated contact between a plastic surface being treated and small particles previously coated with a transferable conductor, has been adapted to the application of transparent static dissipating coatings to acrylic plastic, including curved surfaces such as occur in aircraft canopies.

It has been established that coated meter windows having resistivities readily attained by the Marklad process will adequately protect sensitive meter movements against external static charges of the accumulation of charges on the windows themselves.

Flat sheets have been coated so as to have resistance in the range of 1 to 10 megohms per square, light transmission of 75 to 85%, and haze of 1.0 to 2.5%. Uniformity has been improved so that 90% of the area of a flat sheet is encompassed within one order of magnitude. Methods are indicated for having all areas within these limits. Procedures have been extended in the direction of handling curved surfaces. Undercoats and overcoats which promote the uniformity of Marklad coatings and protect them against wear and washing have been developed. Undercoated and overcoated antistatic coatings have withstood the tests of Fed. Spec. L-P-406b, Method 1093, Spec. Mil-C-5547 and Spec. Mil-P-80A without deterioration of anti-static properties.

Balco Research Laboratories. WADC TR 53-378 Pt 3. THE DEVELOPMENT OF ELECTRICAL CONDUCTING TRANSPARENT COATINGS FOR ACRYLIC PLASTIC SHEET. George A. Dalin, Joseph Rennert. AF 33(616)-111. April 1956.

The deposition of transparent electrically conductive films on acrylic sheet for the purpose of eliminating fog and ice formation by dissipation of electrical power is discussed. Techniques are described for forming such films by sputtering. The optical, mechanical and electrical properties are described. Suggestions for the design of transparent sections are presented.

Cornell Aeronautical Laboratory, Inc. WADC TR 55-499. AN INVESTIGATION OF THE RAIN EROSION OF TRANSPARENT AIRCRAFT GLAZING MATERIALS AT SUBSONIC SPEED. Roy R. Lapp, Raymond H. Stutzman, Norman E. Wahl. AF 33(600)-6469. April 1956.

The rain erosion resistance of glass and transparent plastic materials currently used for glazing was determined at 500 mph and 1 in/hr rainfall on the Cornell Laboratory test apparatus.

Three glasses with coefficients of linear thermal expansion ranging from 30 to 90 x 10^{-7} in/in/°C were evaluated. Each glass was tested in the annealed, semi-tempered and fully tempered state.

The fully tempered hard lime glass possessed the greatest rain erosion resistance.

The transparent plastic materials tested were Plexiglas II UVA, Plexiglas 55, Polymer K, Sierracin and polymethyl-alpha-chloroacrylate.

In these tests all the transparent plastic materials eroded in one to three minutes. Similar specimens in glass withstood erosion 10 to 15 times as long as the transparent plastic materials, i.e. they showed erosion in from ten to thirty minutes. In general, the erosion resistance of the same type of glass increased as the temper of the glass was increased.

It might be stated that aircraft glazing materials have shown little tendency to pit due to rain erosion in service. This is due to the fact that angle of impact of rain drops upon the glazing material in flight altitude is low. In the tests described herein, the impact angle is essentially 90° which results in the highest rate of erosion.

Dow Corning Corporation. WADC TR 56-159. SILICONE INTERLAYER MATERIAL PROGRAM. Keith E. Polmanteer, Francis J. Campbell, Thomas L. Laur. AF 33(600)-27185. July 1956. ASTIA Document No. AD 97139.

The further development of a thermally stable silicone interlayer material for laminated glass, originally introduced under AF Contract 33(600)-23081 and continued under AF Contract 33(600)-27185, was the object of this research by the Dow Corning Corporation during a one year period ending in January, 1956. Improvements were made in fabricating and laminating the interlayer materials. Two new interlayer materials, Type K and Type L, were described. Both of these materials gave much better adhesion to glass and to aluminum than did the previous silicone interlayer materials studied. The materials both look promising for use in aircraft high temperature applications. Glass laminates made with Type K interlayer material will withstand temperatures up to 300°F for an indefinite period of time. Although Type L interlayer material requires further minor improvements, it appears to be thermally stable at temperatures of 400° to 450°F.

Rohm and Haas Company. WADC TR 56-133. RESISTANCE TO SHATTERING BY GUNFIRE OF TRANSPARENT LAMINATED MATERIALS FOR AIRCRAFT GLAZING. Edward M. Petruska. AF 33(616)-2479. August 1956. ASTIA Document No. AD 97191.

Gunfire tests were conducted on laminated Plexiglas 55, Sierracin 611 and Polymer K with a 300°F outside surface temperature, a 70°F inside air temperature, and a 4 psi differential on each specimen. The specimens were flat and clamped around the periphery. The tests were run

with a 50 caliber bullet fired at each test sample.

When comparing laminates of equal configuration, Plexiglas 55 had better crack resistance than Polymer K and both were better than Sierracin 611. Orientation of laminates of non-uniform configuration had little effect on crack resistance. The 0.375"-0.375" Polymer K laminate was slightly better than other Polymer K configurations tested. The 0.400"x0.200"-0.400" Sierracin 611 laminate was much better than any other Sierracin configuration tested. Laminates containing 25 parts DBS plasticizer per 100 parts PVB were slightly better than those containing 37.5 parts plasticizer.

Some 75% stretched Plexiglas 55 of .250" thickness was tested under the same conditions as the laminates. These resulted in a bullet hole with no cracking. The stretched material was therefore superior in shatter resistance to all the laminates tested.

WADC TR 56-491.
DETERMINATION OF THE ACCEPTABILITY OF A COMMERCIALLY STRETCH-FORMED ACRYLIC AIRCRAFT CANOPY. David M. Coddington, 1/Lt. December 1956. ASTIA Document No. AD 110619.

The first comercially stretch-formed acrylic aircraft canopy was tested for adequate crack propagation resistance and edge attachment strength, stretch distribution and birefringence. Results indicate crack propagation resistance values of 25-36 in.lbs/in^2 and edge attachment strength well above the 600 lbs/in. design requirement.

Based on the results of this evaluation, the canopy is considered satisfactory.

General Aniline and Film Corporation.
WADC TR 56-243.
MODIFIED POLYMETHYL ALPHA-CHLOROACRYLATE PLASTIC SHEET. Harry D. Anspon. AF 33(600)-23883. February 1957. ASTIA Document No. AD 118035.

This program was initiated to obtain a transparent polymer with elevated temperature properties superior to polymethyl-α-chloroacrylate. A modified polymethyl-α-chloroacrylate was developed with markedly improved resistance to bubbling (chemical heat stability), but with no or only a slight increase in physical strength as measured by the heat distortion test. The modified polymer exhibited a marked increase in the already excellent resistance to crazing of polymethyl-α-chloroacrylate, but the modified polymer possessed a decreased light stability. Although the modified polymer is less formable than polymethyl-α-chloroacrylate, the formability is still quite good.

In addition to the improved chemical heat stability and improved resistance to crazing, the modified methyl-α-chloroacrylate polymer was found to exhibit the property of being obtainable with better polished surfaces after annealing than polymethyl-α-chloroacrylate cast under the same conditions.

WADC TR 56-557 Part I.
EFFECTS OF GAMMA RADIATION ON AIRCRAFT TRANSPARENT MATERIALS. Donald L. Schmidt, 1/Lt., Robert H. Johnson, 1/Lt. March 1957. ASTIA Document No. AD 118111.

The effects of gamma radiation on various optical, mechanical and thermal properties of transparent plastic materials were investigated. Gafite, Plexiglas 55 and Sierracin 611 materials were irradiated in a gamma facility to total absorbed doses of 10^6 to 10^{10} ergs per gram and then evaluated in accordance with standard testing procedures.

The physical properties of the transparent plastics were not significantly affected by a radiation exposure of 10^7 ergs per gram, or less. At higher absorbed doses, most of the physical properties were degraded. In addition, several desirable changes in specific physical properties were also obtained.

The optical properties of the transparent plastic materials were more susceptible to radiation-induced changes than were the mechanical or thermal properties.

Dow Corning Corp. WADC TR 56-352.
DEVELOPMENT OF A RIGID TRANSPARENT PLASTIC MATERIAL SUITABLE FOR AIRCRAFT GLAZING AT ELEVATED TEMPERATURE. George Helvey, H. A. Clark. AF 33(616)-3065. April 1957. ASTIA Document No. AD 118248.

This program was initiated in an attempt to obtain a rigid, transparent plastic having properties at elevated temperatures superior to those of presently available transparent plastic materials.

Evaluation of several silicone formulations was conducted. The most promising material was a combination of high and low viscosity components, designated as Resin Type BB.

This Resin Type can be polymerized in cast sheet form and is thermally stable after prolonged exposure at 200°C. Optical properties of the cast sheet are excellent and not visibly affected by heat aging. The physical properties are, in general, well below those of known materials (Heat Distortion is 40°C to 66°C).

National Bureau of Standards.
WADC TR 56-645 Part I.
PROPERTIES OF GLASSES AT ELEVATED TEMPERATURES. Part I: Preliminary Program. Matthew J. Kerper, Campbell Lathey, Henry E. Robinson. AF 33 (616)-56-13. May 1957. ASTIA Document No. AD 118323.

A program has been initiated to investigate the physical properties of glasses that are suitable for aircraft glazing. The program will consist of: (1) developing suitable test methods for determining the physical properties at room and elevated temperatures, and (2) determining the values of the physical properties of individual glasses for use as design data.

During the past year all necessary test methods have been developed or adopted. A satisfactory modulus-of-rupture test was developed using two-point loading and specimens that had one surface abraded by sandblasting. The sandblasting reduced the spread of the results and made the test more sensitive.

Some thermal properties previously determined have been adopted and some mechanical properties for a commercial soda lime glass have been determined at room and elevated temperatures.

General Aniline and Film Corporation
WADC TR 57-24.
DEVELOPMENT OF A RIGID FLUORINATED TRANSPARENT PLASTIC MATERIAL SUITABLE FOR AIRCRAFT GLAZING AT ELEVATED TEMPERATURES. Harry D. Anspon, Joseph J. Baron, Jr. AF 33(616)-3240. July 1957. ASTIA Document No. AD 130914.

The field of fluoroacrylate polymers was studied as a starting point on investigation of new polymeric materials for use as high temperature resistant, transparent, glazing materials.

A literature survey of this field showed that, although several fluoroacrylate polymers had been prepared previously, almost no information was available on the polymers and their properties.

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The fluorine analogs (methyl α-(trifluoromethyl) acrylate $CH_2{=}C(CF_3)COOCH_3$; and methyl α-fluoroacrylate, $CH_2{=}CFCOOCH_3$) of the two, well-known, readily-polymerizable monomers, methyl methacrylate, $CH_2{=}C(CH_3)COOCH_3$ and methyl α-chloroacrylate, $CH_2{=}CClCOOCH_3$, were prepared by methods described in the literature.

Methyl α-(trifluoromethyl)acrylate did not polymerize under conventional methods nor under special conditions recommended in patents by a previous investigator. It was polymerized to a transparent solid by extended gamma radiation. The characteristics of the polymer obtained by gamma radiation remain to be determined.

Methyl α-fluoroacrylate polymerized readily under light irradiation to a tough, transparent, solid polymer with a heat distortion temperature of 110°C (230°F) with excellent solvent craze resistance, and with a heat stability of about 2 hours at 204°C (400°F) in an air oven. Its heat distortion temperature was lower than 135°C(275°F), the heat distortion temperature of the corresponding chloro compound, methyl α-chloroacrylate; but its toughness, solvent resistance, and heat stability were superior to those of methyl α-chloroacrylate polymer ("Gafite"). Additional quantities of methyl α-fluoroacrylate are in preparation to provide samples for more extensive testing of this polymer.

Rohm & Haas Co. WADC TR 54-619 Part 2.
THE INVESTIGATION OF MULTIAXIALLY STRETCHED ACRYLIC PLASTIC - Part 2: Biaxially Stretched Plastic. Alfred Batzdorff, Julius J. Gouza, Dee A. Hurst. AF 33(616)-489. July 1957. ASTIA Document No. AD 142084.

The purpose of this investigation was to further define the effects of stretching procedures and of simulated warm forming treatments on the material properties of acrylic plastic. It also expanded the scope of previous work by including studies on stretched poly (methyl alpha chloroacrylate). A new phase of these investigations was the gunfire tests on stretched materials.

Conditions were determined for stretching poly (methyl alpha chloroacrylate), or Gafite sheet, and physical properties of the stretched sheet were obtained. Stress-solvent crazing resistance increased with the increase in percent stretch, and the Gafite was significantly more stress-solvent craze resistant than the MIL-P-5425 and MIL-P-8184 materials. All three materials behaved similarly in showing much less stress-solvent crazing resistance after six months outdoor aging.

Both Gafite and MIL-P-8184 materials with moderate to high degress of stretch were highly resistant to stress-only crazing after six months outdoor aging, and were better than stretched MIL-P-5425 materials in this respect.

The effect of varying the conditions of stretching, especially the rate of stretching, on the properties of three types of acrylic plastic with several different percents of stretch was evaluated by means of dW/dA tests, 50°C crazing tests, and gunfire resistance. The results indicated definite effects of stretching conditions on the gunfire resistance and crazing properties, but no significant effects on the dW/dA results on the stretched materials.

The effect of a simulated warm forming cycle on the properties of stretched materials was of no great magnitude. There was some reduction in stress-solvent crazing resistance, but the properties were still much improved as compared to unstretched material.

Conditions of temperature, speed of testing, and moisture content of the specimen affected the dW/dA test results quite significantly. Limited investigation was made of these phenomena. The effect of specimen size on the dW/dA results was investigated.

Some of the sources of variation of results on dW/dA tests were analyzed. There was considerable variation with percent stretch and the 60% stretch region on MIL-P-8184 material represented a wide range of dW/dA values which were heterogeneous in their source. It would be wise to avoid this degree of stretch in order to reduce variability in crack propagation resistance.

Dow Corning Corporation
WADC TR 57-213.
SILICONE INTERLAYER MATERIAL PROGRAM.
Keith E. Polmanteer, Francis J. Campbell, Thomas L. Laur. AF 33(600)-27183. July 1957. ASTIA Document No. AD 130887.

Improvement of thermally stable silicone interlayer materials, Types K, L and M, was the object of this research. A method was developed to completely confine interlayer samples during heat aging. Falling ball impact and peel strength test methods for elevated temperature testing were developed. Work on Type L interlayer was ended due to its poor shelf life. Improvement in Type M interlayer material was achieved. Introduction of capillaries in Type M interlayer made it possible to prepare 12 x 12 inch laminates which withstood 400°F for 100 hours without forming defects.

Tensile strength, peel strength, bullet impact and falling ball impact properties of Type K and Type M interlayer were measured over a broad temperature range. Falling ball impact results showed PVB laminates to be better than Type K laminates over the temperature range of 30°to 97°F. Type K laminates had slightly higher impact strengths than Type M laminates over the temperature range of -65° to+ 400°F. Impact resistance was shown to be directly related to adhesion.

Dow Corning Corporation
WADC TR 56-352 Part II
DEVELOPMENT OF A RIGID TRANSPARENT PLASTIC MATERIAL SUITABLE FOR AIRCRAFT GLAZING AT ELEVATED TEMPERATURE. George Helvey, Harold A. Clark. AF 33(616)-3065. October 1957. ASTIA Document No. AD 142047.

Additional research work was conducted to obtain a rigid, transparent plastic having properties at elevated temperatures superior to those of the presently available transparent materials.

Evaluation of straight silicone resins, designated as Resin Type BB, was continued but no significant improvement in properties was realized. Although this resin type has excellent optical properties (luminous transmittance of over 90 percent and haze less than 1 percent) which are not visibly affected by heat aging (thermally stable after prolonged exposure at 200°C) the physical properties are, in general, well below those of other commercially available materials. The Heat Distortion Temperature of Resin Type BB is 40°C to 66°C. This material will not be satisfactory for glazing applications requiring rigidity at elevated temperatures but may be adapted for other uses.

Flake glass reinforcement of the resin was attempted but was not successful because of poor orientation.

Large sheet castings were successfully fabricated by processing the silicone resins using special techniques.

Battelle Memorial Institute. WADC TR 57-447.
ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF TRANSPARENT PRESTRETCHED PLEXIGLAS 55.
G. M. Gynn, J. A. Vanecho, W. F. Simmons.
AF 33(616)-3215. October 1957. ASTIA Document No. AD 142045.

Creep and creep-rupture, short-time tensile, and deterioration data were obtained for prestretched Plexiglas 55, a transparent acrylic, sheet material. Data were obtained at 80, 160, 200, and 250°F. A comparison of properties is made with unstretched 5105XP, an experimental Plexiglas material.

The stress-rupture strength properties of the prestretched material were found to be superior to the unstretched 5105XP at 80, 160, and 200°F. The prestretched Plexiglas deformed more than the unstretched material did, initially resulting in inferior creep-strength properties even though minimum creep rates were essentially the same. The short-time tensile strength of the prestretched Plexiglas is superior to the unstretched material at temperatures to about 170°F. At temperatures above 170°F, the strength of the stretched material fell below that of the unstretched 5105XP Plexiglas.

WADC TR 57-421.
TRANSPARENT MATERIALS FOR AIRCRAFT ENCLOSURES - WADC-UNIVERSITY OF DAYTON JOINT CONFERENCE. Robert E. Wittman. October 1957. ASTIA Document No. AD 142021.

The purpose of this report is to make available all technical papers presented at the recent Seventh Conference on "Transparent Materials for Aircraft Enclosures." This conference was held for the exchange of knowledge on new developments and design concepts concerned with crew enclosures and to make known the state of the art with respect to transparent plastics, interlayer materials, and glass.

The papers contained herein have been reproduced directly from the original manuscripts.

WADC TR 57-640.
EVALUATION OF CAST UNFILLED TRIALLYL CYANURATE-POLYESTER LAMINATING RESINS. D. F. Starks. December 1957. ASTIA Document No. AD 142182.

An exploratory evaluation of the properties of three cast unfilled triallyl cyanurate-polyester laminating resins was conducted to determine if this chemical type of resin would provide a useful material for further development as a heat resistant transparent glazing material for use in aircraft. The castings were made and all tests were conducted by the Plastic Products Section of the Organic Materials Branch, Materials Laboratory, Directorate of Laboratories, Wright Air Development Center. The tests were selected as those that would best present a basic representation of the merits of the resins. These included specific gravity, water immersion, heat distortion, tensile strength, izod impact and room and elevated temperature flexural tests. Optical quality castings with good light transmission were not obtained; however, it was considered that if the other properties were promising, the optical characteristics could be included in later development studies.

The triallyl cyanurate-polyester laminating resins are not 2-stage materials in the form evaluated but are cast and cured to the final form. They were compared to Selectron 400 which is a 2-stage material with satisfactory optical properties. Since most of the properties of the triallyl cyanurate-polyester laminating resins investigated were inferior to Selectron400, development work as described in WADC TR 57-580 will be continued on Selectron 400.

WADC TR 57-580
EVALUATION OF SELECTRON 400 TRANSPARENT PLASTIC MATERIAL. Donald L. Schmidt. December 1957. ASTIA Document No. AD 142180.

Evaluation test data are presented on the optical, mechanical and thermal properties of a new experimental transparent plastic material, designated Selectron 400.

Selectron 400 has outstanding physical properties with respect to strength at high temperatures, heat distortion temperature and resistance to crazing. The material in its present stage of development has several undesirable characteristics such as brittleness, notch sensitivity and low impact strength.

Selectron 400 is suitable for use as monolithic canopy material in nonpressurized aircraft, and as surface sheets of laminated glazing assemblies on pressurized aircraft. However, Selectron 400 laminates with a silicone heat resistant interlayer have not yet been successfully fabricated and evaluated.

General Aniline and Film Corporation
WADC TR 57-24 Part II.
DEVELOPMENT OF A RIGID FLUORINATED TRANSPARENT PLASTIC MATERIAL SUITABLE FOR AIRCRAFT GLAZING AT ELEVATED TEMPERATURES. Harry D. Anspon, Joseph J. Baron, Jr. AF 33(616)-3240. February 1958. ASTIA Document No. 150966.

Fluoroacrylate esters were investigated to determine whether their polymers possessed properties suiting them to use as high temperature resistant, transparent, glazing materials.

Two sheets (12" x 12" x 1/8") of methyl α-fluoroacrylate were cast. The properties of these sheets were determined by tests run at Wright Air Development Center and by General Aniline and Film Corp. This polymer posses good forming properties, excellent craze resistance, good stability toward light and heat [Polymethyl α-fluoroacrylate is at least 100 times more stable than polymethyl α-chloroacrylate at 191°C (375°F)] and is tougher than the chloro-analog. However, its heat distortion temperature (108°C) is lower than that of polymethyl α-chloroacrylate (135°C).

Polymers and copolymers of methyl α-fluoroacrylate, methyl α-(trifluoromethyl) acrylate, methyl α-chloroacrylate, methyl methacrylate, and glycidyl methacrylate were prepared and examined. Only cross-linked glycidyl methacrylate possessed a higher heat distortion temperature than methyl α-chloroacrylate polymer. The glycidyl group seems to confer some heat stability to the acrylate over that possessed by a methyl ester.

Exploratory synthesis of methyl α-chloro-β,β-difluoroacrylate, $CF_2{:}CClCOOCH_3$, and methyl α,β,β-trifluoroacrylate, $CF_2{=}CFCOOCH_3$ were carried out. It is probable that the former compound has been isolated, but the last steps of the synthesis of the latter compound remain to be completed.

WADC TR 56-557 Part II.
EFFECTS OF GAMMA RADIATION ON AIRCFART TRANSPARENT MATERIALS. Donald L. Schmidt. May 1958. ASTIA Document No. AD 155580.

This report presents additional data on gamma radiation-induced changes in commonly-used and experimental transparent plastic materials.

Eight materials of an acrylic or polyester type were irradiated to maximum absorbed energy levels of 10^6 to 10^{10} ergs per gram in a cobalt-60 isotope facility. The irradiated materials were then evaluated in accordance with standard testing procedures to determine (a) which physical properties were altered by gamma irradiation, (b) the nature of these changes, and (c) the magnitude of

these changes.

Results of this investigation show that most of the plastic materials properties were affected by a high intensity gamma radiation environment. The principal effect was found to be that of degradation, but initial property improvements were also noted. This latter effect, however, was almost always followed by degradation upon continued irradiation.

Comparative radiation stability data are presented on the various irradiated transparent plastic materials. Threshold and 25 percent damage dosages for the individual materials properties are reported. The optical properties of the materials were more susceptible to gamma radiation than were the mechanical, thermal or permanence-type properties. Threshold dosages were about 1 to 5 x 10^7 ergs per gram for optical properties that change with irradiation, and about 10^8 to 10^9 ergs per gram for other radio-sensitive properties.

Dow Corning Corp. WADC TR 57-213 Part II. SILICONE INTERLAYER PROGRAM. Keith E. Polmanteer, Francis J. Campbell, Thomas L. Laur. AF 33(600)-34984. August 1958. ASTIA Document No. AD 155803.

The main objective during the contract was to improve Type M interlayer or to develop a subsequent material to eliminate the need for capillaries in Type M laminates. A number of modifications of Type M interlayer were tested but none of these had all the requirements to make them better than Type M. One modification, which had greater thermal stability than Type M, was not self-adhering to glass or aluminum. A number of adhesive materials were tried in conjunction with this modification, but none were satisfactory. However, properties of several of these materials were encouraging. Development work will continue.

A study was made of the temperature gradients existing in Type K laminates when subjected to simulated flight conditions. This study pointed out the effect of glass thickness on the relation between the surface temperatures of the laminate and the peak interlayer temperature

PART III

PHYSICAL METALLURGY AND SOLID STATE SCIENCES

Physical Metallurgy

Rutgers Univ. Dept. of Physics, New Brunswick, N. J. In cooperation with Harvard Univ. Lyman Lab. of Physics, Cambridge, Mass. Annual Research Report, No. 8, AFOSR-TN-54-27.

EFFECTS OF DIFFUSION ON FREE PRECESSION IN NUCLEAR RESONANCE EXPERIMENTS. H. Y. Carr and E. M. Purcell. Sponsored jointly by Office of Naval Research, Atomic Energy Commission, The Radio Corp. of America, Rutgers Univ. Research Council, and Air Force Office of Scientific Research. AF 18(600)975. Sept. 1, 1954. ASTIA Document No. AD 48577. Also published in Phys. Rev., v. 94: 630-638, May 1, 1954.

Nuclear resonance techniques involving free precession are examined, and, in particular, a convenient variation of Hahn's spin-echo method is described. This variation employs a combination of pulses of different intensity or duration ("90-degree" and "180-degree" pulses). Measurements of the transverse relaxation time T_2 in fluids are often severely compromised by molecular diffusion. Hahn's analysis of the effect of diffusion is reformulated and extended, and a new scheme for measuring T_2 is described which, as predicted by the extended theory, largely circumvents the diffusion effect. On the other hand, the free precession technique, applied in a different way, permits a direct measurement of the molecular self-diffusion constant in suitable fluids. A measurement of the self-diffusion constant of water at 25°C is described which yields $D=2.5(\pm 0.3) \times 10^{-5}$ cm^2/sec, in good agreement with previous determinations. An analysis of the effect of convention on free precession is also given. A null method for measuring the longitudinal relaxation time T_1, based on the unequal-pulse technique, is described.

Chicago Univ. Inst. for the Study of Metals, Ill. Published in Journ. Chem. Phys., v. 23: 405, Feb. 1955.

SELF-DIFFUSION IN SODIUM NEAR THE MELTING POINT. R. E. Meyer and N. H. Nachtrieb. AF 18(600) 1489. 1955.

Self-diffusion coefficients for solid Na, previously reported for the 0°-94.5°C temperature range (Jour. Chem. Phys., v. 20: 1185, 1952) are determined for temperatures to within 0.10°C of the melting point of polycrystalline and single crystal states of the metal. Spectrographic examination of the once-distilled metal revealed 0.05 percent K, 0.001 percent Si, and less than 0.001 percent of Fe, Mg, and Cu. Its melting point was determined to be 97.91 ± 0.06°C by means of a cooling curve taken in vacuum with a chromelalumel thermocouple calibrated at the steam point and several other standard thermometric points. Cylinders of radioactive and ordinary Na of very coarse grain size (~2 mm diam) were pressure-welded together at liquid nitrogen temperatures and diffusion was allowed to proceed in an oil thermostat which was controlled to ± 0.015°C. The composite cylinder was then sectioned in a microtome and the diffusion coefficient calculated. The results of four runs are tabulated, together with values of D calculated for 94.4° and the melting point from the empirical equation resulting from the earlier study. The newer values are slightly higher, but the agreement is to within the limits of error. Diffusion coefficients for the liquid were measured from 98.0° to 226° by the capillary reservoir technique. The results of these runs may be represented by the equation: $D=1.10 \times 10^{-3} \exp(-2450/RT)$. The values found just above the melting point, also tabulated, deviate only slightly from the value of 4.06×10^{-7} cm^{2}sec^{-1} calculated for 98.0° by means of this equation. It appears that the transition from the rate of solid state self-diffusion to the rate of liquid state self-diffusion is as discontinuous in Na as the melting process, itself.

Chicago Univ. Inst. for the Study of Metals, Ill. AFOSR-TN-56-174.

THE EFFECT OF HIGH PRESSURES ON THE RATES OF ATOM MOVEMENTS IN CRYSTALS. N. H. Nachtrieb. AF 18(600)1489. April 30, 1956. ASTIA Document No. AD 86594. Presented at Third International Meeting on the Reactivity of Solids, Madrid, Spain, Apr. 3-7, 1956.

The significance of diffusion measurements as a function of pressure is considered for the provision of information about the volume of the activated state. The basic assumption is that diffusing atoms pass through a transition state whose existence is sufficiently well defined to have meaningful thermodynamic functions ΔF, ΔH, ΔS, ΔV, etc. Two crystalline solids (Na and P) were studied. Curves are presented which show (1) the effect of pressure on 90°C diffusion profiles for Na for pressures up to 12,000 kg/sq cm^{-2}; (2) the variation of ln D (diffusion coefficient) with pressure for Na at 90°C and for P at 30° and 41.30°C; and (3) $\overline{V}_{act}$ (activated volume) as a function of pressure. Results indicate that the volume of the defect (presumably a moving lattice vacancy) is not greater than half the atomic or molecular volume which implies an inward relaxation of atoms toward a lattice vacancy. Experiments showed no temperature dependence in the enthalpy of activation for self diffusion and a significant variation of ΔH with pressure. The simularity of the ratio of the enthalpies of activation and fusion to the ratio of the volumes of activation and fusion suggests that the fusion mechanism is the same as the self-diffusion mechanism. The proposition is being examined that an atom neighboring a vacancy in a solid must surmount the same activation barrier as in the liquid state.

Chicago Univ. Committee on Math. Biology, Chicago, Ill. AFOSR-TN-56-411. Also published in Bull. Math. Biophysics, v. 18:271-315, 1956.

CONTRIBUTIONS TO THE THEORY OF ACTIVE TRANSPORT. C. S. Patlak. AF 18(600)1454. May 15, 1956. ASTIA Document No. AD 96220.

In many biological situations, the net transport of material from one region to another cannot be explained by a passive movement following thermodynamic laws. The term "active transport" has been applied to such cases, and is discussed in this study. It is pointed out that a system consisting of 2 solutions separated by a membrane may be in one of four possible states: (1) transient; (2) steady; (3) equilibrium; or (4) pseudo-equilibrium. The latter state denotes that in the solutions the net flow of all components is zero, but at least one of the components is not in thermodynamic equilibrium. Transient and steady-state systems may or may not have active transport; thus only systems in either equilibrium or pseudoequilibrium are considered here, since the former indicates that there is no active transport, whereas, in the latter case there always is active transport. This simplifies the problem of finding whether a system does or does not have an active transport mechanism, since it is frequently fairly easy to determine experimentally whether a system is in equilibrium or pseudoequilibrium. The assumption that electric neutrality exists within very thin membranes is shown not to be valid; however, electric neutrality does exist in the solutions in a system in a pseudoequilibrium state with fixed charges and impermeative ions. It is then shown how the presence and sign of an electric potential may be found by use of electroneutrality. The mechanism of active transport may result from a general force acting on all particles of a particular component or to an individual force acting on the individual particles of a particular component. A general solvent flow or a diffusion drag force illustrates the first mechanism, while the second is accomplished by either a carrier or a Maxwell Demon. The Maxwell Demon is a hypothetical mechanism which allows each particle of A to cross a potential barrier by itself more readily in one direction than in the other. It differs from a carrier in that no part of it in contact with A moves while A is crossing the barrier. The Demon operates either by lowering the potential barrier height for A, by increasing A's speed of crossing the barrier, or by modifying A so that it can cross the potential barrier more readily. The general type of active transport has been extensively treated in the literature, while the individual type has not been treated in a generalized form. Therefore, the individual type of active transport is discussed at length, and a simple illustrative model is intensively analyzed. Following this, there is a discussion of the Maxwell Demon, and some models of it are presented

WADC TN 58-369.

OBSERVATIONS ON THE EFFECT OF SURFACE AND STRUCTURE ON THE TENSILE STRENGTH OF IRON WHISKERS. Helmut Weik. May 1959. ASTIA Document No. 212151. PB 151904. Order from OTS $0.75.

Investigations were made of iron whiskers grown by reduction of iron chloride. Characteristic whisker shapes were found to be dependent on the growth conditions. Observations of the surface of the whiskers after the test revealed normal slipping on slip planes connected with low tensile strength in the case where the whiskers were grown from wet chloride. In the case where they were grown by reduction of dried chloride in hydrogen and argon, however, much higher values for the tensile strength were obtained. The strength increases with decreasing whisker diameter. On the surface of some whiskers from this series shearing of surface layers could be observed giving the impression that the whisker surface has a layer or shell structure. By means of microstructure investigations, the layers observed in the surface were also discovered in the whisker core. The distance between the layers was found to be constant and about 1200 to 1500 atoms thick. It may be concluded that the layers are a structural element of the whiskers grown under the conditions above mentioned.

Nickel coating of the whiskers increased the tensile strength. In the case of a 100 atoms thick nickel coating the amount of increase was about 100% compared to the strength value of the uncoated whisker. That means that surface effects are important in considering the reasons for the extremely high tensile strength of very thin whiskers.

WADC TR 58-615, Part II.

A COMPENDIUM OF CONSTITUTIONAL TERNARY DIAGRAMS OF THE METALLIC SYSTEM. Dr. W. Guertler, Berlin, Germany. AF 61(052)-74. June 1954. ASTIA Document No. AD 215427.

The purpose of this project is to collect and present a complete collection of ternary phase diagrams of the various metallic systems.

The ternary diagrams presented are built from available binary diagrams. In each instance the most reliable binary diagram has been selected.

The basis for the designation of the pages is the order of the atomic numbers. It is thus possible to attribute to each ternary alloy three numbers below 100 corresponding to the three combining elements and to assemble them in order of magnitude from the smallest to the highest figure. Consequently each system can be located easily.

Metallwerk Plansee. WADC TR 55-243, Part II.

DEVELOPMENT OF ALLOYS HAVING GOOD HIGH TEMPERATURE PROPERTIES THROUGH POWDER METALLURGY TECHNIQUES. Dr. R. Kieffer, Dr. F. Benesovsky. AF 61(514)-1144. September 1959.

Wet milled powder mixtures of Fe-Al-Mo 80-14-5-TiC/Cr_3C_2 alloys with 0-30% of a 90-10 TiC/Cr_3C_2 mixed crystals were sintered in a vacuum and their properties were evaluated.

With increasing additions of carbide mixed crystals the hot strength increases; however, the alloys become brittle.

The reinforcing of these alloys by means of Ni-Cr, megapyr or molybdenum wire and nets pressed into the powder compacts increases the impact strength provided no reaction occurs between the alloy and the reinforcing structure.

Materials Research Corp. WADC TR 59-441, Part I.

THE INVESTIGATION OF THE MECHANISM OF SUBSTRUCTURAL FORMATION IN REFRACTORY METALS AND THE RELATION TO THE OBSERVED MECHANICAL PROPERTIES. A. Iannucci, G. Murray, J. Intrater, S. Weinig. AF 33(616)-5908. January 1960. PB 161489. Order from OTS $1.00.

The formation of substructure in molybdenum as a function of prestrain and polygonization anneal temperature has been demonstrated by metallo-graphic and x-ray techniques. The x-ray diffraction line width is markedly increased by the presence of substructure. In both polycrystalline and single crystal creep tests the creep resistance increased as a function of prestrain. In single crystal tests the creep resistance decreased with increased polygonization anneal time.

Engineering Supervision Co. WADC TR 59-730.

LITERATURE SURVEY ON RESEARCH AND DEVELOPMENT ON HIGH PRESSURE TECHNOLOGY. Lawrence Berg, Harry Herman. AF 33(616)-6729. March 1960. PB 161887. Order from OTS $1.50.

In view of the varied work being accomplished by various people and organizations throughout the world in the field of high pressure technology, it became expedient to compile in one bibliography the reports, articles, reviews, etc. pertaining to this subject which have been published to date. This report is such a bibliography, listing alphabetically by author the results of a survey of available literature, both foreign and domestic, classified and unclassified, in the field of high pressure technology. The compiled information includes, for each article, the title, source, page number, number of pages, chemical abstract number (CA), physical abstract number (Ph.A.), and a general description of the subject matter. Co-authors of any given publication have been cross-referenced.

It is intended to reissue this report from time to time as new authors emerge and new publications are reviewed.

New York Univ. WADC TR 59-414.

A SURVEY OF DISPERSION STRENGTHENING OF METALS AND ALLOYS. R. F. Bunshah, C. G. Goetzel. AF 33(616)-5882. March 1960.

This report is a survey of the current work in this area. A review of the theories advanced to explain dispersion strengthening is presented. The structural and other variables which govern the strength and stability of these alloys are outlined and the results discussed in terms of these variables. Comparison of theories with experimental results have been made.

The results of the survey indicate that dispersion strengthened alloys exhibit outstanding properties at elevated temperatures when the right structural conditions are present, i.e., the inter-particle spacing is in the range 0.01-0.1 micron, with high hardness of the dispersed phase, low interfacial energy between matrix and dispersed phase (in most cases). Other contributing factors are stored energy, coherency strains and matrix strengthening by solid solution. It must be emphasized that no general prescription for high strength in these alloys can be written as the predominant strengthening mechanism or variable depends on the temperature and duration of test or service. The stability of these alloys at service temperatures is enhanced by high thermodynamic free energy of formation of dispersed phase, low or negligible solubility and low diffusivity of the components of the dispersed phase in the matrix.

A large number of techniques can be used to produce dispersions in metals and alloys. The report contains a summary of these techniques.

Much further work both of a fundamental nature to delineate the role of various variables and that oriented towards alloy development remains to be done. Dispersion strengthening of refractory metals appears most promising and should be a very fruitful area of investigation.

University of Minnesota. WADC TR 59-674.

THE PLASTIC COEFFICIENTS FOR WORK-HARDENING MATERIALS. W. H. Warner, P. Mahmoodi. AF 33(616)-5449. April 1960. PB 161886. Order from OTS $0.50.

In the establishment of the form of their generalized stress strain law for work-hardening inelastic materials, Warner and Handelman did not show how the plastic coefficients could be evaluated in terms of material properties. This report first shows how this can be done, giving the general form of the coefficients for various states of stress. The validity of the assumptions behind this representation must be experimentally tested; comparison with existing results is carried out as far as possible. The conclusion suggests what types of experiments are needed to test the validity of the law, and if they are successful, what further experiments are needed to provide the information necessary to solve various combined stress problems analytically.

Manufacturing Laboratories, Inc. WADC TR 59-655.

RESEARCH AND DEVELOPMENT ON THE EFFECTS OF HIGH PRESSURE AND TEMPERATURE ON VARIOUS ELEMENTS AND VINARY ALLOYS. J. S. Harvey, L. Kaufman, S. A. Kulin, A. Leyenaar, H. Udin. AF 33(616)-5996. April 1960. PB 161920. Order from OTS $2.25.

The accomplishments of the contract period July 1, 1958 to September 30, 1959 are summarized. High-pressure high-temperature apparatus capable of subjecting test specimens to 100,000 atmospheres pressure at temperatures in excess of 1500°C was designed and constructed. Investigations carried out in this apparatus demonstrate that high hydrostatic pressure acts to lower the temperature of martensitic transformations in iron-nickel alloys and expands the temperature range of the gamma loop in the iron-chromium system. The precipitation hardening process in both aluminum-copper and copper-beryllium alloys is significantly inhibited and modified when carried out at high hydrostatic pressure. The use of pressure during heat treatment of a series of carbon steels results in transformation products radically different in structure from those obtained by the same treatment at atmospheric pressure and also significantly retards the tempering reactions. Evaluation of mixtures of carbon in a ratio of 1:1 atomic percent with platinum, palladium and nickel which were subjected to pressures in excess of 80,000 atmospheres at approximately 3000°C reveals the existence of a new phase which is extremely hard and brittle and contains essentially no metal atoms. Preliminary experiments indicate that the Ti-N phase diagram is significantly modified by pressure.

WADC TN 59-393.

RESEARCHERS ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS: SILVER AND LEAD. L. Peraldo Bicelli, B. Rivolta, Milano, Italy. AF 61(052)-144. April 1960.

Hydrogen overvoltage on silver and lead single crystal cathodes oriented following the (100), (110) and (111) planes has been measured, in sulfuric acid solutions for silver, and perchloric acid and sulfamic acid for lead. Tafel law holds true, the parameters are coincident for all of the silver electrodes, while they are different for the different lead electrodes.

Research Chemicals. WADD TR 60-74, Part I.

THE METALLURGY OF YTTRIUM AND THE RARE EARTH METALS. PART I. Phase Relationships. Bernard Love. AF 33(616)-5905. May 1960. PB 171085. Order from OTS $3.50.

Partial constitutional diagrams were established for sixteen binary systems containing rare earth metals. These studies were made as the first step in efforts to produce alloys with improved mechanical properties.

Erbium and yttrium form simple binary eutectic systems with titanium. No compounds are present. The transition temperature of titanium is not significantly

affected. Copper, tin, and cobalt form intermetallic compounds with erbium and yttrium. The melting points of the rare earths are rapidly lowered, eutectics forming with the respective compounds. Vanadium forms extensive (and chromium forms limited) liquid immiscibility regions with erbium and yttrium. Eutectics are formed at the rare earth end of these systems. Terminal solubilities are low in all of the above systems.

The system erbium-zirconium is characterized by appreciable solubility of zirconium in erbium and extensive solubility of erbium in both alpha and beta zirconium. There are no inter-metallic compounds. The alpha erbium and beta zirconium solid solutions enter into a simple eutectic reaction. A high temperature peritectic reaction involving beta erbium is suggested. The system yttrium-zirconium is similar in all major respects except that the solubility limits at both ends of the system are somewhat lower. Preliminary investigation of the gadolinium-zirconium system also indicated similarity in all major respects.

Beryllium and ytterbium form an intermetallic compound. The terminal solubility of ytterbium in beryllium is low. No eutectic is observed at the beryllium end of the system.

The results obtained suggest zirconium as a promising alloying element. Evidence was also found for the purification of vanadium and beryllium when these metals were melted together with rare earths.

Research Chemicals. WADD TR 60-74, Part II. THE METALLURGY OF YTTRIUM AND THE RARE EARTH METALS. PART II. Mechanical Properties. Bernard Love. AF 33(616)-5905. June 1960. PB 171086. Order from OTS $2.00.

Study of the mechanical properties of rare earth metals was continued. Tensile compression fatigue, and impact properties were determined. Evaluation of the metals was extended to include hot and cold working characteristics, and studies were made of alloy systems predicted to have improved mechanical properties.

Cast yttrium, dysprosium, and erbium can be cold worked to improve mechanical properties. All are easily swaged at 980°C (1800°F). The resulting structures are uniform and dense, and the properties are improved over the cast condition. Tensile properties are quite comparable when the metals are in the same condition. Ductility (as measured by reduction of area) was somewhat limited for the cast structures, but was improved by working. The cast rare earth metals are notch sensitive. The fatigue properties follow the normal pattern of increased life with decreased maximum applied load. Extrapolated S-N curves indicate fatigue endurance limits in the order of 40-50% of the corresponding ultimate tensile strengths.

Zirconium enters into solid solution in erbium and results in marked tensile strengthening. Dispersed, second phase zirconium, also contributes to hardening.

The recrystallization behavior of erbium and yttrium follows a normal sequence of recovery, recrystallization and grain growth, complete recrystallization of erbium, cold worked 60%, occurs at approximately 1100°C; yttrium at approximately 900°C.

Studies were made of several methods for improving the purity of rare earth metals. Carbon deoxidation decreased the oxygen content slightly. Attempts to purify yttrium, erbium, and dysprosium by electron beam melting were not successful. Small quantities of both dysprosium and scandium were, however, successfully vacuum distilled at elevated temperatures. Analytic results indicate that the distilled metals are of significantly higher purity.

*Southern Research Inst., Birmingham, Ala. DIFFUSIVITY OF IRON IN MOLTEN SILICATES. E. B. Dismukes. Project 9760(802), Contract AF 49(638)-523, AFOSR, DCS.

This research is concerned with studying the self-diffusion of radioactive iron in molten silicates and determining from the results whether iron in these solutions possesses anionic or covalent structure.

*Illinois Univ., Urbana. DIFFUSION AND IMPERFECTIONS IN METALS. R. W. Balluffi. Project 9760(802), Contract AF 49(638)-880; AFOSR, DSS.

The motions of atoms in a metal or alloy will be studied with the objective of obtaining an improved understanding of the kinetics of processes taking place in these materials. Among the phenomena which may receive particular attention in the course of this investigation are the Kirkendall effect in the silver-gold couple and the effects of plastic deformation on diffusion. Experiments involving the effects of plastic deformation on diffusion are of particular interest. Work of this type is of considerable importance in establishing mechanisms of diffusion and of high temperature deformation, and it appears imperative to clarify the situation further. The proposed experiments using vapor-solid alpha brass diffusion couples will eliminate the difficulties associated with the sectioning technique and would allow a large number of specimens to be run over wide ranges of temperature and strain rate.

*Metallurgy and Ceramics Research Branch, ARL, Dayton, Ohio. EFFECT OF COMPOSITION ON THE SELF-DIFFUSION CHARACTERISTICS OF NICKEL. L. R. Bidwell. Project 7021 (802), Internal.

The effects of aluminum and titanium alloying additions on the diffusion coefficients and activation energies for self-diffusion of nickel in nickel-base solid-solution alloys are to be investigated for correlation with X-ray dynamic displacement and Debye characteristic temperature measurements to determine if the latter constitute an adequate measure of relative interatomic binding forces.

*Northwestern Univ., Evanston, Ill. EFFECT OF SOLUTE IMPERFECTION INTERACTIONS UPON PLASTICITY AND FRACTURE. J. O. Brittain. Project 9761 (802), Contract AF 49(638)-780; AFOSR, DSS.

The objectives of this research are to make a systematic study of (1) grain boundary cohesion and (2) solute-imperfection interactions in metals. These are distinctly related objectives since grain boundaries are composed of imperfections and experience has shown that small additions of foreign elements profoundly affect the mechanical behavior of materials at both elevated and low temperatures. The research on grain boundary cohesion is being carried out through a study of the role of solute elements upon the grain boundary migration, relaxation

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

and fracture over a range of temperatures. The study of solute imperfection interactions is being accomplished by means of strain-aging and internal friction studies on iron and steel. A further aim of the research is to study yielding of materials that deform in a heterogeneous manner in the transition from elastic to plastic deformation and the relation of yielding to the initiation and/or propagation of a crack. A final item will be the study of strain aging at elevated temperatures of high-temperature materials.

*Rice Inst., Houston, Tex.
EFFECT OF VIBRATIONS ON LATTICE IMPERFECTIONS MOVEMENT IN METALS. F. R. Brotzen. Project 9760(802), Contract AF 49(638)-78; AFOSR, DSS.

It is the objective of the research herein to establish the mechanism of interaction between vibrations and imperfections. The study of the interaction is expected to yield valuable fundamental information concerning the role that lattice imperfections play in plastic deformation and diffusion processes. The effect of mechanical vibrations on the migration of various types of imperfections will be investigated primarily by means of changes in electrical resistivity at different temperatures. Vibrations in the sonic and ultrasonic ranges will be applied, so as to determine the influence of frequency upon the imperfections movements. A complete study will be made of the influence of vibrations upon the strength and ductility of certain metals. Special attention will be given to the effect of vibrations upon creep in metals. Diffusion in the solid state is affected by vibrations will be investigated. The effect of vibrations upon the activation energies of diffusion in substantial and interstitial alloy systems will be established. The kinetics of solid-state precipitation reactions will be studied in the presence of mechanical vibrations.

*Technion Research and Development Foundation, Israel.
EFFECTS OF ORDER-DISORDER PHENOMENA ON THE STRENGTH PROPERTIES OF HIGH TEMPERATURE ALLOYS. P. S. Rudman. Project 7021(802), Contract AF 61(052)-122; ARL, MCB.

Metallic strength generally depends on metastable equilibria that relax rapidly at elevated temperatures. Order is an equilibrium state and its strengthening properties can persist even at the higher temperatures. The strengthening mechanism of order-disorder phenomena will be investigated by simultaneous X-ray diffraction, metallographic, and mechanical property studies on Fe-Ni-Al alloys.

*Case Inst. of Tech., Cleveland, Ohio.
ELECTRONIC STRUCTURE OF METALS AND DILUTE ALLOYS. W. L. Gordon, T. G. Eck. Project 9760(802), Contract AF 49(638)-621; AFOSR, DSS.

The purpose of this investigation is to gain an improved understanding of the electronic structure of solids. This will contribute to our ability to engineer the electronic, magnetic, and absolute strength properties of metals and to test our present theories of alloys. De Haas - van Alphen measurements are being carried out in magnesium crystals and in magnesium-silver alloy crystals. Cyclotron resonance measurements are being carried out in aluminum crystals cut sufficiently thin so that the orbital motion of the electrons begins to be damped out. The degree of damping under these circumstances may be related to the shape of the Fermi surface in this material.

*Ohio State Univ., Columbus.
FACTORS AFFECTING THE KINETICS OF THE ALLOTROPIC TRANSFORMATION IN METALS. J. W. Spretnak. Project 7021(802), Contract AF 33(616)-6109; ARL, MCB.

The potentialities of the Faraday cage incorporated in the electron emission microscope will be evaluated for use in making quantitative measurements of the distribution of elastic and plastic strain within grains as a function of solute content and applied stress. Initial work will be on iron while later studies may include other pure metals and alloys. The clustering effects of like and unlike atoms in austenite prior to transformation will be studied in iron-carbon alloys as a function of temperature as affected by alloying elements.

*Oxford Univ., Great Britain.
FREEZING AND MELTING TEMPERATURES OF IRON ALLOYS/BeO/ThO_2. W. Hume-Rothery. Project 7021(802), Contract AF 61(514)-1062; ARL, MCB.

A highly accurate study is being made of the precise melting and freezing temperatures of iron-rich alloys with binary additions of zirconium, niobium, molybdenum, ruthenium, rhodium, palladium, aluminum and silicon. This information has been obtained previously but not to the accuracy required for use in a theoretical treatment of alloying phenomena.

*Illinois Univ., Urbana.
INTERNAL FRICTION TECHNIQUES IN THE STUDY OF DIFFUSION AND PHASE CHANGES IN METALS. C. Wert. Project 9760(802), Contract AF 49(638)-672; AFOSR, DSS.

This research is concerned with the development of new techniques for internal friction measurement. Current developments include very low temperature studies at frequencies of about one cycle per second, and a high frequency apparatus for use at 100KC. These techniques are being applied to studies of diffusion in solids at such low temperatures that other methods are useless. In addition, the method may be used to measure energy absorbed by dislocations as they are being driven through a crystal under the influence of stress. Current research is concerned with atom motion in iron near magnetic transitions, in Fe-V alloys, and in Mg-Cd alloys.

*Materials Central, WADD, Dayton, Ohio.
KINETICS AND MECHANISMS OF WHISKER GROWTH. H. Weik. Project 7023(802), Internal.

Our present knowledge of the growth characteristics and formation of iron whiskers indicates that a refined approach with careful chemical control is necessary in order to obtain detailed and more accurate data on the growth mechanism. Attention will be focused on the kinetics of the reaction, $FeCl_2+H_2 \rightleftarrows Fe+2HCl$, which upon consideration of free energy calculations and qualitative observations is probably operative. The reaction will be studied in the vapor phase in a Y-shaped furnace accommodating the preheating of the hydrogen gas, vaporization

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

of the ferrous chloride (anhydrous), and its entrainment in a stream of argon, and a chamber for the reaction. It is anticipated that surface effects will also be of great importance. The crystallography of the whiskers so obtained under varying conditions of temperature, hydrogen-ferrous chloride ratio, etc. will also be scrutinized. At present the assembly and construction of equipment is the major occupation.

*Metallurgy and Ceramics Research Branch, ARL, Dayton, Ohio.
LATTICE DISTORTIONS AND ATOMIC INTERACTIONS IN METALLIC SOLID SOLUTIONS. L. R. Bidwell. Project 7021(802), Internal.

The influence of solute atoms in changing the mean dynamic and static displacement of atoms from their ideal lattice sites will be investigated by the use of X-ray diffraction techniques. Precise lattice constants and the integrated intensities of selected Bragg reflections will be measured at 88°K and 298°K to determine Debye characteristic temperatures and the mean amplitude of thermally induced atomic oscillations. This dynamic displacement of the atoms from their time averaged lattice positions is considered to be a direct measure of the factors which control the elastic constants and the diffusional characteristics of the bulk materials. The static displacements of the atoms from their ideal lattice positions resulting from the solution of atoms with different electronic configurations and disparities in size can be determined from the same diffraction data. This information will present a much more detailed picture of the lattice strain energy than the usual lattice parameter measurements. The results of this investigation will be used for a theoretical treatment of the strength of metallic solid solutions.

*Carnegie Inst. of Tech., Pittsburgh, Pa.
MECHANISM OF EVAPORATION OF METAL CRYSTALS. J. P. Hirth, W. H. Robinson. Project 9761(802), Contract AF 49(638)-551; AFOSR, DSS.

The objective of this research is to determine quantitatively the evaporation rates from single crystal and polycrystal metal surfaces as a function of time, temperature, crystal orientation, and degree of crystal perfection. The results of the investigation will be compared with predictions of a theoretical treatment which is under continuing development. In addition, the theoretical kinetics of evaporation from crystals in equilibrium with atomic or molecular vapor phases will be derived. The method of investigation will include a study of thermal etching with the formation of etch pits at the sites of dislocations in the crystals. This aspect of the study will be correlated with X-ray studies to determine crystal perfection.

*Carnegie Inst. of Tech., Pittsburgh, Pa.
OPTICAL PROPERTIES OF METALS AND ALLOYS. W. R. Bitler. Project 9761(802), Contract AF 49(638)-478; AFOSR, DSS.

The optical properties of metals are a function of the electronic structure of the metal. In this research an attempt is being made to determine this electronic structure from reliable optical data: the coefficient of reflection of the metal, the absorption of light by thin films, and the rotation of the plane of polarization of magnetic metals, these being determined over a range of temperature down to liquid helium. Research such as this has been unable to attract serious interest in the past because vacuum equipment has not been available in which the metal surfaces could be kept clean. In this work copper containing small percentages of nickel or manganese is being studied. With modern vacuum equipment, it is possible to study in a controlled way the effects of small amounts of gases on surface contamination.

*Ohio State U., Columbus.
OXIDATION CHARACTERISTICS OF TUNGSTEN OVER SUITABLE TEMPERATURE AND OXYGEN PRESSURE RANGES. R. Speiser. Project 7022(802), Contract AF 33(616)-5721; ARL, MCB.

A fundamental investigation will be conducted on the oxidation behavior of tungsten in the temperature range of 500°C to 1400°C and at oxygen pressures of atmospheric and below. Emphasis will be placed on determining the rate controlling process within these environmental ranges in order to better define the operative mechanism. The structure and physical properties of the scales formed will be thoroughly investigated. The tungsten-oxygen phase diagram will be determined over the environmental ranges of interest. Present high temperature oxidation theories will be critically tested, and if necessary, a new or modified theory will be proposed. The volatility of WO_2 and WO_3 have been studied in various air-water vapor mixtures from 1000°C to 1350°C. The thermodynamic properties of WO_2 have also been determined. The rate controlling process for tungsten oxidation is observed to be diffusion of oxygen ions through the oxide scale to the oxide-metal interface.

*Virginia Univ., Charlottesville.
OXIDATION AND INHIBITION MECHANISMS. J. W. Cole. Project 9761(802), Contract AF 18(603)-103; AFOSR, DCS.

This is a study of the mechanism of inhibition of the oxidation and corrosion by heterocyclic compounds, especially the phenothiazine series between the temperatures 20° to 250°C. Attention will also be given to the mechanism of formation of metallic sulfides.

*Delaware Univ., Newark.
OXIDATION OF METALS AND ALLOYS. C. E. Birchenall. Project 9760(802), Contract AF 49(638)-872; AFOSR, DSS.

This research concerns the study of the oxidation of metals and alloys including detailed investigation of some of the component processes. Particular attention will be devoted to the study of diffusion in solid oxides and sulphides, and the dependence of ion mobilities on the crystal structure and defect concentrations. Attempts will be made to extend the understanding of the mechanisms of oxide plasticity in the growth of adherent and non-adherent scales.

*Princeton Univ., N. J.
OXIDATION OF METALS AND ALLOYS. C. E. Birchenall. Project 9761(8002), Contract AF 49(638)-533; AFOSR, DSS

This research concerns the study of the oxidation of metals and alloys including detailed investigation of some of the component processes. Particular attention will be devoted to the study of diffusion in solid oxides and sulphides and the dependence of ion mobilities on the

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

crystal structures and defect concentrations. Attempts will be made to extend the understanding of the mechanisms of oxide plasticity in the growth of adherent and non-adherent scales.

*McMaster Univ., Canada.
OXIDATION OF METALS AND ALLOYS. W. W. Smeltzer, Project 9761(802), Contract AF 49(638)-734; AFOSR, DSS.

The purpose of this research is to investigate the kinetics of metal and alloy oxidation. Quantitative oxidation rate studies will be carried out on some metal and alloys that are important as potential aircraft and missile structural materials (for example: titanium, zirconium, hafnium, and certain transition metal alloys). In addition, a theoretical analysis will be carried out for the diffusion processes of oxygen in the surface oxide and metal substrate during oxidation of the metals titanium, hafnium and zirconium.

*Carnegie Inst. of Tech., Pittsburgh, Pa.
OXIDATION, VAPOR NUCLEATION AND SURFACE DIFFUSION OF METALS AND ALLOYS. G. M. Pound. Project 9761(802), Contract AF 18(600)-1572; AFOSR, DSS.

1. Oxidation: The objective of this investigation was to examine the oxidation of metals; the influence of metal substrate properties of an alloy on the oxidation characteristics, the effect of oxygen solubility in the metal, titanium for example, upon oxidation mechanism, a re-examination of the oxidation of pure iron in carbon dioxide, and the structure of the oxide formed on iron-chromium alloys. 2. Vapor Nucleation and Evaporation: This part of the research has as an objective the determination of the behavior of condensed metal vapor on metallic substrates under ultra high vacuum (better than 10^{-9}mm. mercury). A thermal beam of silver atoms has been deposited onto a tungsten tip. The Mueller field emission technique is being utilized to make direct observations on the processes of surface diffusion, nucleation, growth and evaporation of the deposited silver. Nucleation, growth, and evaporation characteristics as well as mobility of the condensate may be determined for various degrees of super-and under-saturation at various target temperatures.

*Metallurgy and Ceramics Research Branch, ARL, Dayton, Ohio.
SHORT RANGE STRUCTURE DISTORTIONS. H. J. Garrett. Project 7021(802), Internal.

Physical structural factors affecting diffuse scattering of X-rays are temperature diffuse scattering and structural scattering by structural distortion information. This information is needed for theoretical developments in almost all phases of metallurgy. The phenomena of alloying, cold work, impurity atom effects, radiation effects, fatigue, and many others that determine the properties of metallic materials are associated with structural distortions. Initial investigations in this area of research shall be studies of mechanically induced distortions, specifically measurements of microstrains in aluminum.

*Denver Univ., Colorado
PRINCIPLES OF ALLOY FORMATION USING RARE EARTH METALS. J. F. Nachman. Project 7021(802), Contract AF 33(616)-6787; ARL, MCB.

A thorough study will be made of the principles of alloy formation using the rare earth metals and being guided by a critical use of the Hume-Rothery rules. A suitable binary system (probably neodymium-praseodymium) with the least number of variables will first be studied. The variables include electronic structure, crystal structure, atomic diameter, valency effects, electronegativity, mean Fermi energy and electron concentration. Using two or three property determinations as criteria, other rare earth binary alloys will be made in which the variables are introduced one by one and the alloys studied using the same criteria. The criteria will be solubility relationships in the alloy system, the approach to ideality in both the liquid and solid solutions, and possible resistivity.

*Stanford Research Inst., Menlo Park, Calif.
STUDIES OF ELECTRON DEVICES MATERIALS TECHNOLOGY. L. Feinstein. Project 4619(760), Contract AF 19(604)-7323; ERD, EML.

This will be a study of brazing and metal joining problems of a basic nature. The effect of diffusion zones of brazine metals on the mechanical behavior of brazed joints, the formation of diffusion zones and intermetallic layers of metals shall be studied.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
SUBMILLIMETER REGION RESEARCH. J. N. Plendl. Project 5621(802), Internal.

This investigation covers the spectral region from about 3 to 1000 microns in wavelengths, i.e., the inter-region between optics and microwave electronics. It also covers the ultraviolet with respect to the Raman effect.

GE Metallurgical Products Department, General Electric Research Laboratory. WADC TR 59-747. RESEARCH AND DEVELOPMENT ON THE EFFECTS OF HIGH PRESSURE AND TEMPERATURE ON VARIOUS ELEMENTS AND BINARY ALLOYS. E. W. Goliber, K. H. McKee, J. S. Kasper, J. E. Hilliard, J. W. Cahn, V. A. Phillips. AF 33(616)-5995. July 1960. ASTIA Document No. AD 244767. PB 171348. Order from OTS $2.00.

An investigation has been made of the effect of pressure on the properties and kinetics of transformation in various alloy systems. Many of the experiments were exploratory and yielded no evidence for irreversible changes with pressures of up to 100,000 atm at elevated temperatures. However, a shift was observed in the gamma loop of the iron-chromium system from 12.5 to about 20 per cent chronium. Evidence for a similar shift in the gamma loop of the iron-aluminum system was also found. Pressure heat treatment of a tool steel gave a refinement of the austenite grain size and an improvement in the distribution of carbides.

An apparatus for X-ray diffraction analysis at

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

pressures up to 35,000 atm has been constructed, and observations have been made on the bismuth I to II transition.

In the kinetic experiments it has been found that pressure markedly decreases the transformation rate of austenite to pearlite and the precipitation rate in the systems; aluminum-copper, copper-beryllium, lead-tin, and gold-nickel.

The Martin Company, WADD TR 60-31. A STUDY OF THE EFFECTS OF SURFACE FILMS ON THE MECHANICAL PROPERTIES OF METALS. Irvin R. Kramer, AF 33(616)-6220. July 1960. PB 171150. Order from OTS $1.00.

It is shown that by removing the surface of aluminum single crystals while they are being deformed, the extent and slope of Stage I and Stage II are affected. The stress at which Stage III starts is also lowered. A dislocation pop-out phenomenon was observed when the rate of metal removed was suddenly increased during the tensile deformation. Deformed crystals of zinc, aluminum, and copper immersed in solutions containing surface active agents reacted with these agents to form metal soaps.

Research & Development Laboratories. WADD TR 60-543. ATTEMPTED SYNTHESIS OF BERYLLIUM HYDRIDE. John C. Powers, Donald W. Vose, Edward A. Sullivan. AF 33(616)-5847. October 1960. PB 171489. Order from OTS $1.00.

The direct synthesis of beryllium hydrides from the elements was attempted in two sets of equipment designed to grind beryllium metal while subjected to heat and hydrogen pressure. Grinding action was supplied by mechanisms which provided the reaction vessels with either reciprocating vertical motion, or motion in which the reactor's horizontal axis described a circle. The ability of both mechanisms to grind metals satisfactorily was demonstrated in test runs, in which magnesium hydride was synthesized. However, beryllium hydride was not prepared by this method, even when catalytic additives were used. There were no indications even of partial hydriding.

Operation with beryllium, which is highly toxic, was carried out safely by conforming to recognized standards of industrial practice.

Pennsylvania State University. WADD TR 60-534. FIELD ION MICROSCOPY OF IRON WHISKERS. Erwin W. Muller, AF 33(616)-6397, ASTIA Document No. AD 249733. Oct. 1960. PB 171598. Order from OTS $0.75.

The design, construction and operation of a complete field ion microscope unit is described. The restrictions due to the high rate of field evaporation of iron are considered. This microscope is used for the study of the atomic structure of iron whiskers. Indications of crystal defects such as screw dislocations and interstitials are found, although the whiskers did usually not have sufficient strength to withstand the extremely high field forces.

WADD TR 60-705. THE INFLUENCE OF PRESSURE AND TEMPERATURE ON ZIRCONIUM OXIDE. F. W. Vahldiek, C. T. Lynch, L. B. Robinson. ASTIA Document No. AD 249765. Nov. 1960. PB 171948. Order from OTS $1.00.

The effects of temperature and pressure on the monoclinic-tetragonal phase transformation and on the sinterability of superpure zirconia have been studied. Investigations were undertaken at temperature ranges from 1140°C to 1800°C and at pressures from 310 to 12380 atmospheres. Experiments showed that no tetragonal phase was stabilized after firing and rapid quenching in air, water and liquid nitrogen. The region of transformation was found between 1193° to 1200°C. Pressure lowered the sintering temperature of zirconia, and specimens pressed at 9280 atmospheres and fired to 1800°C in an inert atmosphere resulted in compacts with a 98.8% theoretical density.

Grain refinement was observed with increasing temperature and at various pressures. Average particle sizes of 4 microns and less were found in compacts originally consisting of particles averaging 60 microns. A mathematical equation for the compressibility of zirconia was derived.

WADD TN 60-242. THE APPLICATION OF COMPUTER TECHNIQUES TO PREFERRED ORIENTATION STUDIES. J. R. Holland, December 1960.

A method has been devised and is currently being used to plot normalized pole figures by computer techniques. As the pole distribution traces, i.e., intensity of the diffracted X-ray beam, versus angular position of the specimen are not an acceptable input for the computer, the data required from such traces are entered onto punch cards. Corrections for defocusing effects may be readily made as the data are transferred from the Brown recorder traces to punch cards. It is possible to program other correction factors into the computer operations, providing that these correction factors or curves can be established accurately. The computer translates angular position of the specimen into rectilinear co-ordinates which are an identical representation of stereographic co-ordinates. This computer method has been used in conjunction with the Schulz reflection technique, but modification of the computer program permits its use with other quantitative X-ray techniques for determining preferred orientation.

The feasibility of using computer techniques to plot inverse pole figures has been considered.

Creep

University of Minnesota. WADC TR 57-104 ANALYSIS OF DYNAMIC CREEP CONSIDERING STRAIN RATE EFFECTS. F. H. Vitovec. AF 33(616)-2803. May 1957. ASTIA Document No. AD 130762. PB 131 256.

The dynamic creep behavior of a number of materials is discussed and classified. The creep properties under simultaneous action of static stress and alternating stress have been derived by several authors from the creep rate-stress relationship under static load. The use of this so derived stress range diagram for creep as a basis for determination of acceleration or retardation of dynamic creep is emphasized. Retardation of creep is attributed to aging effects, and to strain rate effects caused by alternating stress frequency. Acceleration of creep is attributed to metallurgical changes, and overtemperature at very high frequencies. Special consideration is given to the effect of superposition of strain rate and creep rate. The theoretical analysis of the strain rate effects in dynamic creep, presented here, conform with the experimental observation that retardation of creep increases with increasing temperature, and with increasing alternating stress frequency if metallurgical effects are negligible.

WADC TR 57-297.
DESCRIPTIONS OF SOME CURRENT METHODS FOR DETERMINING CREEP PROPERTIES UNDER COMPRESSIVE, BEARING AND SHEAR TYPE OF LOADING. E. L. Horne. June 1957. ASTIA Document No. AD 130802. PB 131 259.

This report gives descriptions of several methods currently used for evaluating creep behavior in compressive, bearing and shear types of loading. Four methods for compressive creep testing are described. Two methods for bearing creep and four methods for shear creep, respectively, are also given. Only unclassified and nonproprietary information are discussed. Special mention is made of methods developed under WADC sponsorship. Future panel action will be based on recommendations by panel members and the organizations they represent resulting from a study of methods described in this report.

Univ. of Michigan. WADC TR 57-150, Pt. II
EFFECT OF PRIOR CREEP ON MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL METALS. Part II - 17-7PH Alloy (TH 1050 Condition). Jeremy V. Gluck, Howard R. Vorhees, James W. Freeman. AF 33(616)-3368. April 1958. ASTIA Document No. AD 151115. PB 131 826.

A study was carried out of the effect of exposure to elevated-temperature creep conditions on subsequent mechanical properties of aircraft structural metals. The present report considers a precipitation hardening stainless steel, 17-7PH (TH 1050 condition). Exposures were conducted for times of 10, 50, or 100 hours either unstressed or at stresses giving up to 3-percent total deformation at temperatures between 600° and 900°F.

Following the exposures, short-time tensile, compression, or tension-impact tests were run at either room temperature or the temperature of exposure. The principal effects found were a loss in ductility in tensile tests and a substantial loss in compression yield strength after exposure to creep at 600°F. In general, remarkably little change in the other mechanical properties was found as a result of exposure to creep, with any such changes primarily confined to increases in strength.

Battelle Memorial Institute. WADC TR 57-96.
A SUMMARY OF COMPRESSIVE-CREEP CHARACTERISTIC OF METAL COLUMNS AT ELEVATED TEMPERATURES. R. L. Carlson, G. K. Manning. AF 33 (616)-3317. April 1958. ASTIA Document No. AD 151114. PB 131 825.

A summary of an extensive study of the creep buckling of metal columns is presented. The column behavior prior to collapse is described and the column action at the time of buckling is interpreted in terms of stability.

Solutions to creep buckling are discussed. It is concluded that in spite of certain limitations, Shanley's time-dependent tangent-modulus method has several practical advantages over available analytical solutions.

An application of the time-dependent tangent-modulus method to four structural metals indicates that estimates are consistently conservative for small values of column imperfection. Imperfection variations were generally observed to have a very marked effect on the column lifetime.

The possible existence of a lower column-load limit below which time-dependent collapse will not occur is discussed. From a rational consideration of known creep behavior it is concluded that there may be a temperature below which finite lower limits exist and above which the lower limit is zero.

University of Michigan.
WADC TR 57-150, Pt. III.
EFFECT OF PRIOR CREEP ON MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL METALS - Part III: C110M Titanium Alloy. Jeremy V. Gluck, Howard R. Voorhees, James W. Freeman. AF 33(616)-3368. May 1958. ASTIA Document No. AD 155578. PB 151 145. Order from OTS $2.25.

A study was carried out of the effect of exposure to elevated temperature creep conditions on the subsequent mechanical properties of C110M, an 8 percent manganese binary titanium alloy. Exposures were conducted for times of 10, 50, or 100 hours either unstressed or at stresses causing up to 3 percent total deformation at temperatures between 650° and 800°F. Specimens were taken parallel to the sheet rolling direction.

Following the exposures, short-time tensile, compression or tension-impact tests were run at either room temperature or the temperature of exposure. Prior creep-exposure was found to have little effect on the fracture strength or ductility in either tensile tests or tension-impact tests. The original material had an abmormally high tension yield strength and a low compression yield strength. Exposure to temperature alone caused a decrese in the tensile yield strength and an increase in the compression yield strength depending on the exposure time and temperature, and on the test temperature. Plastic deformation either during loading, or principally during creep, resulted in an increase in the tensile yield strength and a decrease in compression yield strength from the values established by exposure to temperature alone. This behavior can be attributed to a Bauschinger-type effect.

University of California.
WADC TR 58-63.
THE ROLE OF SUBGRAINS IN HIGH TEMPERATURE CREEP. Lawrence A. Shepard, John E. Dorn. AF 33(616)-3860. May 1958. ASTIA Document No. AD 155565. PB 151 208. Order from OTS $1.00.

The role of the subgrain structure in the high temperature creep process is evaluated in the light of recent experimental and theoretical developments in creep theory. The origin of subgrains is discussed, as well as the specific deformation modes which produce the variety of observable substructures. The effect of creep variables on the subgrain development, size and structure is described. Finally, an attempt is made to analyze the manner in which the subgrain structure affects creep rate, recovery and grain boundary shearing. It is concluded that the contribution of the substructure to high temperature creep resistance is small relative to other factors.

University of Michigan. WADC TR 58-204.
AN INVESTIGATION OF THE RELATIONSHIP BETWEEN MICROSTRUCTURE AND CREEP-RUPTURE PROPERTIES OF HEAT-RESISTANT ALLOYS. A. Phillip Coldren, James W. Freeman. AF 33(616)-3239. July 1958. ASTIA Document No. AD 155711. PB 151 421.

Progress is reported for an investigation of the influence of conditions of hot working on the properties of alloys at high temperatures. Conditions of working can be used to control microstructural variations in a manner which cannot be obtained in any other procedure and is capable of developing structures superior in properties to any other treatment. The major objective is to define the basic principles involved so that they can be applied to the general problem of developing optimum properties in any

alloy. The relatively simple structure of "A" Nickel is being used as an experimental material for study of the role of working for properties in the as-worked condition. A-286 alloy is being used as an example of a material whose properties are influenced after solution and aging treatments by the conditions of prior working.

The results reported cover the initial surveys of the relationships of working conditions to creep and rupture properties. Structural analyses to define the basic principles involved were confined to preliminary partial studies. The investigation is being continued with emphasis on the structural studies.

Univ. of Minnesota. WADC TR 58-340. FATIGUE, CREEP, AND RUPTURE PROPERTIES OF THE ALLOYS UDIMET 500, HASTELLOY R-235, AND GMR-235. F. H. Vitovec. AF 33(616)-2803. October 1958. PB 151 608. ASTIA Document No. 202502. Order from OTS $2.00.

Fatigue, rupture, and creep data at 1200 and 1650°F obtained under various combinations of mean and alternating stress are presented. Tests were performed under axial stress on unnotched specimens and specimens having a theoretical stress concentration factor of 3.4. The data are presented as S-N curves and stress range diagrams to show the effect on the fatigue and creep properties of stress concentration, temperature, ratio of alternating-to-mean stress, and stress magnitude.

Univ. of Illinois. WADC TR 59-526. PROTECTIVE COATINGS FOR REFRACTORY METALS. C. G. Bergeron, A. L. Friedberg, R. C. Anderson, V. E. Bradford, D. M. Maroney, R. W. Boyl. AF 33(616)-5734. January 1960. PB 161 739. Order from OTS $1.25.

Preliminary experiments of a probing nature demonstrated that oxygen-free atmospheres, but not high vacuum conditions, were required for the successful application of ceramic coatings on tungsten.

Tests with tungsten encapsulated in evacuated fused silica tubes demonstrated that glassy materials were indeed impermeable to oxygen at temperatures above 3000° F for long periods of time, as evidenced by the non-oxidation of the encased tungsten.

In order to avoid the excessive oxidation and extremely rapid rate of volatility of the tungsten oxides at coating processing temperatures, experiments were carried out by processing zirconia-containing vitreous-bonded coatings in an argon atmosphere.

Experiments with siliconized tungsten wire indicated that silicide coatings are most protective in regions where glass formation at the surface of the coating had been well developed. Because of the silicide "pest" and the brittleness of the silicide, such coatings may not alone be satisfactorily protective.

High temperature testing using the direct resistance heating of tungsten indicated that ceramic coatings consisting of admixtures of zirconia and glass provided protection of tungsten wire for several hours at temperatures in the range of 3000° to 3500°F.

New York University. WADC TR 59-405. DEVELOPMENT OF PROTECTIVE COATINGS FOR REFRACTORY METALS. C. G. Goetzel, P. S. Venkatesan, R. F. Bunshah. AF 33(616)-5735. February 1960. PB 161 736. Order from OTS $1.50.

A preliminary study of the feasibility of protecting tungsten against oxidation at 1650°C (3000°F) was undertaken. Rhodium was selected from the noble metal group as the most promising metallic type of coating. It was applied by electrolytic deposition, either directly onto thoriated or pure tungsten wires, or onto intermediate coats containing the elements rhenium, chromium and silicon. These sub-coats served several purposes, such as: 1) providing a layer of intermediate expansion characteristics between those of the substrate and the rhodium coat; 2) modifying the oxides formed on the surface of the substrate; or 3) promoting the formation of liquid phases at the surface that would freely flow and seal exposed areas of the substrate.

All substrate wires were thoroughly degassed by a vacuum heat treatment before and after electroplating or vapor deposition of the different coats. The wires were heated by their own resistance to temperature sufficient to effect diffusion alloying and bonding between the different coats and the substrate under protective atmosphere, to permit fusion and flowing of the molten rhodium coating over the wire surface under protective atmosphere, and to expose the coated wires to stagnant or streaming air at progressively higher temperatures up to 1650°C (3000°F).

It was found that the methods used permitted protection of tungsten against oxidation up to temperatures at least 550°C (1000°F) above those for uncoated tungsten. A multiple coating consisting of a first inner layer of chromium, a layer of silicon, a second layer of chromium, and a thin layer of rhodium after appropriate heat treatments protected thoriated tungsten at 1650°C (3000°F) for periods up to 20 minutes. Evidence points toward further improvements if complete diffusion alloying, bonding and melting of the surface coat is achieved over the entire length of the specimen.

*Battelle Memorial Inst., Columbus, Ohio. COLUMN ACTION DURING CREEP-BUCKLING. R. L. Carlson. Project 7063(806), Contract AF 33(616)-6301; ARL, MRB.

An experimental and analytical study will be made to obtain basic and new knowledge relating to the behavior of columns subjected to creep buckling, and to determine the accuracy of Girard's theory. An attempt will be made to develop new analytical methods to predict the behavior and ultimate life of columns subjected to creep buckling. Relationship will be derived between creep buckling instability, and the elementary stability theory for columns. The basic mechanisms of column creep buckling will be mathematically described.

*Southwest Research Inst., San Antonio, Tex. CREEP COLLAPSE OF LONG CYLINDRICAL SHELLS UNDER HIGH TEMPERATURE AND EXTERNAL PRESSURE, T. Wah. Project 7063(806), Contract AF 33(616)-5727; ARL, MRB.

This is an analytical and experimental program to study the creep collapse of thin-walled, long, cylindrical shells subjected to elevated temperatures and external pressure. Attempts will be made to establish the relationship between collapse time and parameters of loading, temperature, shell dimensions, material properties, and initial discrepancies in the roundness. Confirming theories of Timoshenko and others, it was found that initial eccentricities in the specimen were critical. Because of the flexibility of long, thin shells, it was necessary to design a special fixture to measure the initial eccentricities without distorting the specimen in the process. This fixture was combined with a loading fixture to prevent dimensional distortion in handling between the measuring and loading operations.

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*California U., Berkeley.
FORMULATING METHODS TO PREDICT CREEP FROM INTERMITTENT STRESS AND TEMPERATURE HISTORIES, J. E. Dorn. Project 7021(802), Contract AF 33(616)-7213; WADD; MC.

Research will be performed on high purity aluminum and alpha solid solutions to more completely formulate accurate methods of predicting creep resulting from various intermittent stress and temperature histories from constant stress isothermal creep data. Work shall be conducted in an attempt to ascertain the role of dislocation barrier density, substructure and abrupt changes in stress on the creep rate in the regions of cross-slip and dislocation climb. Mechanisms applicable to creep rate control in the cross-slip region and the mechanisms involved in recovery and vacancy diffusion shall be investigated.

*Metallurgy and Ceramics Research Branch, ARL, Dayton, Ohio.
MECHANICAL PROPERTY STUDIES OF NICKEL-BASE ALLOYS AND EVALUATION OF THE CENTRIFUGAL CREEP METHOD, L. R. Bidwell. Project 7021(802), Internal.

The effects of aluminum and titanium additions on the creep and elevated temperature tensile properties of nickel-base solid solution alloys are being determined to provide data for correlation with current lattice distortion, atomic interaction and diffusion studies. Centrifugal creep properties of the same materials are being determined by the method developed by I. I. Kornilou of the A. A. Baikov Institute, Moscow. This method holds considerable promise as a screening technique for alloy development programs. Centrifugal creep data will be compared with conventional creep data to test the method's applicability.

Crystal Growth and Purification

*Battelle Memorial Inst., Columbus, Ohio.
BASIC TRANSPORT PHENOMENA IN GERMANIUM AND INDIUM ANTIMONIDE, A. C. Beer. Project 9762(802), Contract AF 49(638)-222; AFOSR, DSS.

Present research under this contract is primarily concerned with inhomogeneity effects in indium antimonide, both from the theoretical and experimental standpoints. Factors affecting crystal growth are being studied using radioactive doping agents to see how they segregate. The electronic properties of crystals with inhomogeneous electronic properties are then being studied. It appears that galvanomagnetic properties of the crystals can be strongly influenced by inhomogeneities in the right part of the sample.

*Westinghouse Electric Corp., Pittsburgh, Pa.
CRYSTAL GROWTH MECHANISMS, R. L. Langini, A. I. Bennett, Project 9761(802), Contract AF 49(638)-599; AFOSR, DSS.

Crystal growth is a process wherein an ordered atomic lattice structure is extended by accretion of atoms from an adjacent liquid or vapor phase. In many cases the details of the accretive mechanisms and their dependence on experimental conditions are poorly understood. It would therefore seem appropriate to look for a crystal growth system which from an experimental standpoint is easy to work with and on which results of controlled reproducibility can be obtained, and to investigate it thoroughly. The research falls into three overlapping phases. First, the effects of seed geometry and structure, temperature distribution and time variation, growth rate, cooling after solidification, and other pertinent growth variables will be investigated to further expand the range of validity of the present theory of this type of growth. Second, research will be directed toward determining the distribution of impurities both within the grown crystal and within the melt. This will involve measurement of the electrical properties. And finally, theoretical considerations will be made of such things as the effects of the liquid on the rate and direction of growth of the crystal and the preferred stacking of atoms through a double bond mechanism.

*General Electric Co., Schenectady, N. Y.
CRYSTAL PHYSICS, G. W. Spears. Project 5621(802), Contract AF 19(604)-5505; ERD, EML.

During the past year, the influence of various impurity atoms on the dissociation or decrystallization process was investigated as a means of isolating the influence of the impurity atoms. An inherent assumption in this study is that the association or crystallization and the dissociation or decrystallization processes operate the same mechanisms, and that the influence of impurties on crystal growth have their parallel in crystal dissociation processes. This assumption as well as the influences of impurity ions are to be established.

*Madrid U., (Spain).
CRYSTALLOGRAPHY, J. L. Amoros. Project 9760(802), Contract AF 61(052)-193; AFOSR, DSS.

The purpose of this research is to study the thermal motion in crystals, with special reference to thermal expansion, polymorphism and melting. This will include a determination of amplitudes and directions of atomic vibrations in crystals in relation to chemical compositions and crystal structures; a study of transverse and longitudinal waves traveling through the crystal as a function of crystal symmetry and structure; determination of the influence of the vibrations on thermal expansion and polymorphism; and preliminary research on the correlation of the above results in a unified theory of crystal dynamics. Theoretical and experimental investigations of crystal structure and behavior will include: (A) A quantitative measure will be made of the thermal diffuse scattering between reciprocal lattice points and the correlation of observed values with the optical frequency band of the vibration spectrum of the crystal. (B) The temperature dependence of the atomic vibration and its influence on the optical branch of the vibration spectrum at high and low temperatures will be determined. A harmonic vibration will be correlated with thermal expansion and polymorphism. An attempt will be made to register thermal diffuse scattering, through study of changes in vibration spectrum, at the moment of transition. (C) Counter techniques will be used to study the temperature dependence of intensity and to determine the scattering power at a given temperature. (D) The relationship between physical properties of a crystal and its growth history will be studied for the case of organic crystals with melting points up to 300°C and for inorganic compounds with melting points up to 1300°C. Optical, dielectric and piezoelectric properties will be measured in the temperature range from -180°C to 300°C; and (E) Theoretical consideration will be given to relating the physical properties with crystal imperfection thermal history, known impurity and crystal growth.

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Alfred U., N. Y.
DIFFUSION PROCESSES IN IONIC SOLIDS, T. J. Gray. Project 9760(802), Contract AF 18(600)-1448; AFOSR, DSS.

This investigation is concerned with the motions of atoms in such ionic solids as potassium chloride, both highly purified crystals and crystals doped with controlled amounts of impurities. Atomic mobility is being determined from measurements of ionic conductivity. The dependence of this mobility upon impurities, point imperfections such as vacancies, and dislocations is being determined. The imperfections are being identified in part by optical properties of X-ray irradiated crystals which are characteristic of the color centers produced from them. There is evidence from this research that crystals of KCl are obtained with much higher purity by crystallization from water solution than from the molten chloride. Thus, it is found that the ionic conductivity in such crystals is much lower at low temperatures (250-350°C) than in melt grown crystals. However, once the crystals have been heated above about 350°C their conductivity remains high and this may be attributed to mobile point imperfections having been introduced into the bulk of the crystal.

*Hebrew U., (Israel).
DOUBLE X-RAY REFLECTIONS OF SINGLE CRYSTALS, B. S. Fraenkel. Project 9763(802), Contract AF 61(052)-222; AFOSR; DSS.

Using an integrating photometer, the intensity of double reflections will be measured. The variation of intensity of double x-ray reflections with wavelength relative to the intensity of allowed reflections will be investigated. Intensity of double and forbidden reflections as a function of temperature, impurities, and other parameters will be determined in diamond, silicon, and germanium. The wavelength to which double reflections are particularly sensitive will be determined and reflection intensity formulae will be developed for perfect and mosaic structures. The existence and intensity of double reflections in anthracene and other organic crystals will also be studied as a function of temperature, tension and other parameters.

*Pennsylvania U., Philadelphia, Pa.
FORMATION AND ANNEALING OF POINT DEFECTS IN GERMANIUM, J. N. Hobstetter. Project 9760(802), Contract AF 49(638)-626; AFOSR, DSS.

Specifically the research will include a study of the relative numbers of defects introduced by quenching as functions of quenching temperature so as to find directly the energies of formation of vacancies and interstitials in germanium. Subsequent annealing studies will then reveal the fate of these defects as they combine with each other, or with dislocations of known densities, or are trapped at defects in the structure. Similar studies will be made of defects introduced during plastic extension of germanium. These will reveal dislocation motion at various temperatures as well as on annealing when widely disparate numbers of vacancies and interstitials are present. Finally similar studies on electron as other particle bombarded germanium is anticipated in an effort to demonstrate annealing when equal numbers of defects are initially present.

Hebrew U. (Israel).
GAMMA RAY CORRELATION TECHNIQUES IN CRYSTAL STRUCTURE STUDIES, S. G. Cohen, W. Low. Project 9763(802), Contract AF 61(052)-340; AFOSR, DSS.

The study of crystal structure by gamma ray correlation techniques, particularly in the case of rare earth nuclei, is a very rich field of investigation which should contribute significantly to our knowledge of the internal electric and magnetic fields interacting with nuclei in solids and liquids. This kind of information is closely related to that obtained from studies of nuclear magnetic and electron paramagnetic resonance. Although the resonance methods are more precise in general, the techniques of angular correlation offer two important advantages. First the number of atoms required for a measurement is much smaller. This allows one, for example, to work with cases in which a very small number of impurity centers are present in the crystal lattice and to measure the fields present at such impurity centers. Second, the fields in a solid can be measured without the need of applying an external radio-frequency field. Such measurements may be useful in the investigation of the rearrangement of excited electron shells and hence could further our understanding of basic mechanisms controlling the properties of solids.

*Solid State Physics Research Branch, ARL, Dayton, Ohio.
GROWTH OF CADMIUM SULFIDE AND ZINC SULFIDE CRYSTALS, L. C. Greene. Project 7021(802), Internal.

Single crystals of cadmium sulfide, zinc sulfide and mixed zinc sulfide-cadmium sulfide are grown from the vapor phase. Emphasis is placed on growing two types of crystals, (1) a large bulk crystal and (2) whisker and platelet type crystals. The whiskers and platelets are the purest and most nearly perfect that can be grown. They are relatively free of structure defects and have a low concentration of foreign impurities as well as small deviations from stoichiometry. The larger bulk crystals are produced for investigation of such phenomena as photovoltaic effects. In producing the large crystals, efforts are made to purify the material by fractional sublimation. Single crystals of the zinc sulfide-cadmium sulfide compound are grown in all zinc to cadmium ratios and the energy gap has a linear relationship with respect to this ratio. Growth of crystals of the sulfides and selenides of zinc and cadmium and mixtures of these compounds are included in the long range planning for this program.

*International Telephone & Telegraph Corp., Nutley, N. J.
GROWTH OF SINGLE CRYSTALS BY THERMAL FUSION UTILIZING R. F. HEATING, P. Lightly. Project 5621(802), Contract AF 19(604)-7219; ERD, EML.

The flame fusion process for the growth of single crystals has produced many types of crystals for research evaluation and for utilization in various devices. This technique has, however, several serious drawbacks. This contract was initiated for the purpose of modifying the conventional process in order to obtain more perfect crystals. A method was developed in which the usual flame is replaced by an r.f. heat source which, consequently, permits

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

maintaining a controlled atmosphere. This technique has been developed into a successful method for the growth of single crystals. Crystals of ferrites, germanium, silicon, and other materials have been grown. The equipment has also been developed into a usable configuration.

*North Carolina U., Chapel Hill.
IMPERFECTIONS IN IONIC CRYSTALLINE SOLIDS. L. M. Slifkin. Project 9760(802), Contract AF 49(638)-865; AFOSR, DSS.

These experiments are primarily concerned with dislocations and point defects in silver chloride crystals. The obvious interest in silver halide crystals stems from their application in the photographic process. More important to the solid state physicists, however, is the fact that silver halide crystals offer an unusual combination of properties: (a) Silver chloride is very plastic, making studies of dislocations and their effect feasible; (b) It is transparent in the visible region, thus permitting the observation of strain patterns and inclusions such as silver precipitates; (c) A strong absorption in the near ultraviolet allows for limited spectroscopy of the solid; (d) This absorption produces photoelectrons, suggesting studies of photoconductivity and electron and hole traps; (e) When not illuminated, silver chloride conducts electricity by ionic motion, by means of vacancies and interstitial silver ions, the relative contribution being variable by changing the purity and/or temperature; and (f) The trapping of photoelectrons results in charge neutralization by recombination with interstitial silver ions, forming silver atoms which may be made to grow to colloidal silver specks.

*Sylvania Electric Products, Inc., Bayside, N. Y.
INFRARED SCATTERING. P. H. Keck. Project 5621 (802), Contract AF 19(604)-3482; ERD, EML.

The purpose of this contract is to investigate infra-red scattering cross sections of structural defects in single crystals of silicon. The primary interest is the detection of edge type dislocations in semiconducting materials. Unfortunately, the scattering cross section of these dislocations is not high enough at low densities. Valuable information has been derived concerning the precipitation of Cu in Si. In addition, the study of point and line imperfections is to be extended through the development of a new technique, anomalous X-ray absorption, and through the comprehensive comparison of the various techniques available thus far. Anomalous transmission is extremely sensitive to microstrains in crystals and is therefore ideally suited for the determination of microstrain due to imperfections such as vacancy clusters.

*Athens U., (Greece).
INVESTIGATION OF HEAT VIBRATIONS IN SOLIDS WITH X-RAYS. K. D. Alexopoulos. Project 9763 (802), Contract AF 61(514)-1248; AFOSR, DSS.

Basic information about the arrangement and position of atoms in a solid subjected to various temperatures provides useful data in predicting such mechanical properties as volumetric thermal expansion, tensile strength, rupture, and deformation. Since the intensity of the diffracted X-rays depends on the heat vibrations of the atoms, the characteristic temperature, theta, can be determined by measuring the diffracted X-ray intensities at various temperatures. A number of elements will be investigated to find the characteristic temperature (theta) by means of X-ray diffraction, and these will be compared with the characteristic temperature found from specific heats. X-ray measurements of theta will be made to determine its variation with heat. Differences in values of theta will be followed for various types of crystal lattices and for elements of either very high or very low atomic weight. For some of these elements, the contribution of electronic heat to total specific heat will be traced through to high temperatures.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
MAGNETIC MATERIALS CRYSTAL GROWTH. C. A. Pitha. Project 5621(802), Internal.

The paramagnetic resonance absorption of a metal ion in a crystal reflects the influence of its neighbors upon it. Since a structural imperfection represents a very significant variant in the crystal structure and the field it generates, it is important to minimise their concentration and effect. Similarly, an impurity atom perturbs the crystal field pattern and may influence significantly the magnetic characteristics of its neighbors. In order to help establish the magnitude of these effects, single crystals of nominally the same structure but containing different "guest" atoms have been and are being prepared. Controlled amounts of nickel, cobalt, iron, manganese, chromium, vanadium, titanium, platinum, and palladium have been introduced into aluminum oxide "host" lattices. Also, single crystals of garnet-structured rare earth-iron oxides have been prepared in which the rare earths have been lutetium, ytterbium, thulium, terbium, holmium, dysprosium, erbium, gadolinium, europium, samarium, and yttrium. In the immediate future, the effect of transition element "guest" atoms in spinel-structured hosts and of the rare earths on non-ferric garnet structured compounds will be considered.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
MECHANISMS OF CRYSTAL GROWTH. J. R. O'Connor. Project 5621(802), Internal.

Silicon and germanium are being used to study growth mechanisms from the melt, while silicon carbide is being used to study growth from a vapor. One technique for examining these processes is to study microsegregation impurities at a solid-liquid interface. Studies of this general type are being continued by growing bi-crystals of germanium. A definite segregation constant is to be associated with each crystallographic orientation. The Peltier cooling effect at a solid-liquid interface is being examined to determine its magnitude for various materials. This effect may provide an additional means of controlling and enhancing crystallization rates. A resume of the general state of the art for silicon carbide grown from the vapor has been reported. An improved vapor growth furnace for silicon carbide is being completed.

*Louvain U., (Belgium).
MECHANISMS OF LUMINESCENCE. A. Luyckx. Project 8503(803), Contract AF 61(052)-166; RADC; GEL.

An investigation shall be carried out to devise techniques for the preparation of thick single zinc sulfide crystals. In addition, the various phenomena associated with single ZnS crystals will be investigated. These studies will attempt to gain information regarding

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

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activation techniques, nature of electrodes and work functions, ion mobility, conductivity as a function of temperature, activation energy of electrons, fine structure of brightness waves, physico-chemical relations between ohmic and capacitive crystals, and relaxation times of electrons after excitation by variable frequency. Study will also be initiated to determine the enhancement of light emission by irradiation with X-rays. Magnetic effects on electro-luminescence and observation of the Hall effect in ZnS crystals will be studied.

*Electronic Technology Lab., WADD, Dayton, Ohio.

PREPARATION OF SOLID STATE MATERIALS, J. M. Blasingame. Project 4150(803), Internal.

The current objectives are: (1) to prepare single crystal substrates; deposit thin films of various electrically active materials by techniques devised on other tasks of this project; obtain single crystal thin films where necessary by epitaxial growth, heat treatment, and/or electron beam zone leveling; (2) to evaluate the electrical, magnetic, and other physical properties of these films and crystals; and (3) to study the interaction of composite layers of materials with each other, their surface ambients, various electromagnetic waves and other forms of inputs, the phenomena which they exhibit, and the operations which they can perform in molecular electronic circuits.

*Eagle-Picher Co., Miami, Okla.

PURIFICATION OF CADMIUM SULFIDE CRYSTALS, W. Medcalf. Project 7116(801), Contract AF 33(616)-6203; ARL, SSB.

This effort has produced cells with efficiencies for converting solar to electrical energy as high as 8.5 percent. These cells were produced from cadmium sulfide crystals grown from the melt. Purification is currently achieved by zone refining the cadmium and distilling the sulfur. The two elements are then reacted to form cadmium sulfide. The contractor has progressed to the point of having no detectable impurities in the purified cadmium and sulfur by spectrographic analysis. Problems are encountered in reacting the cadmium and sulfur without picking up additional impurities. The chief contaminant added during reaction is silicon. Currently the reaction tubes are being coated with cadmium sulfide prior to reaction, with the object of preventing silicon pickup from the reaction chamber. Improvements in the growth of crystals from the melt both from the standpoint of purity and perfection will be the primary objective.

*Brown U., Providence, R. I.

RECRYSTALLIZATION OF DEFORMED SINGLE CRYSTALS. C. Elbaum. Project 9760(802), Contract AF 49(638)-479; ERD, EML.

The work involves investigation of two fundamental features of recrystallization that have been experimentally established relatively recently. One of these is the dependence of the rate of recrystallization upon impurities and whether these impurities are precipitated or in solution. The other feature is concerned with the orientation of the recrystallized grains relative to the deformed matrix. In this field completely different results have been obtained by two independent groups of workers and it seems important to determine whether or not this can be related to differences of the state of impurities (solution or precipitated). The recovery of growth rate after both continuous isothermal heating and after repeatedly interrupted heating will be studied. Investigation of the connection between dislocation structure in the deformed specimen and the rate of growth of the new crystals is part of the program.

*Michigan State U., East Lansing.

TRANSPORT PROPERTIES OF SOLIDS, F. G. Blatt. Project 9763(802), Contract AF 49(638)-70; AFOSR, DSS.

The investigation will include: (a) Study of the influence of impurities of various kinds on transport in metals; (b) Study of the relaxation times and relaxation mechanisms in solids as observed under a variety of experimental conditions; and (c) Extension of the method of partial waves for calculating scattering cross sections and the situation appropriate for germanium and silicon; namely, ellipsoidal energy surfaces.

*Mineralagisk - Geologisk Museum, Norway.

RELATIONSHIP BETWEEN MAGNETIC AND PHYSICAL PROPERTIES OF SULFIDES AND THEIR CRYSTAL STRUCTURE AND CHEMICAL COMPOSITION. T. F. W. Barth. Project 7021(802), Contract AF 61(052)-178; ARL, MCB.

A study into the structural and thermodynamic relationship of sulfides of synthetic and natural origin shall be conducted through exploratory research with an objective of developing theories of ferromagnetism of semi-conductors. Initially the research shall consist of a study of the solid phases in the system copper-silver-sulfur and then be extended to other ternary systems. For each phase, accurate determinations of the crystal structures at various temperatures, measurements of the optical properties and determination of the electrical and magnetic properties shall be made.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.

SINGLE CRYSTAL GROWTH. C. A. Pitha. Project 5620(802), Internal.

This research concerns the conditions necessary for the growth of single crystals of aluminum oxide doped with the various transition elements, and of conditions required for the growth of single crystals of the rare earth-iron oxides, particularly those involving the rare earths at the end of the series. This work will be supplemented by studies of the conditions necessary for the growth of single crystals from aqueous and non-aqueous solutions.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.

SINGLE CRYSTAL GROWTH FROM THE VAPOR. J. J. Rohan. Project 5620(802), Internal.

This research concerns a detailed study of the factors which affect the growth rate of highly physically and chemically perfect massive single crystals from the vapor. Two such studies are (1) growth of single crystals of low melting metals (e.g., cadmium, zinc, magnesium) from polycrystalline masses and/or melts via vapor transport and (2) growth of high melting elements (e.g., silicon) by decomposition of a halide of these elements (e.g., silicon iodide). The aims are to observe

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Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

optimum experimental conditions, correlate with existing theories, modify or formulate new theory, and establish basic principles applicable to a wide variety of crystal growth problems.

*Device Development Corp., Weston, Mass.
RECOMBINATION COEFFICIENTS IN CESIUM AND OTHER ALKALI METALS. Project 7667(770), Contract AF 19(604)-602; GRD, PL.

This research is directed toward a study of existing knowledge of recombination, attachment and other atomic processes in cesium and other alkali metals. Secondly, measurements will be made of unknown atomic rate coefficients in cesium and other alkali metals.

*Battelle Memorial Inst., Columbus, Ohio.
SOLID STATE PHENOMENA IN RARE EARTH METAL ALLOYS AND COMPOUNDS. R. C. Himes. Project 4150(803), Contract AF 33(616)-7321; WADD, ETL.

Rare earth alloys, intermetallics, oxides, mixed oxides, compounds both with each other and with other metallic and non-metallic elements, and metalloorganic compounds and complexes will be prepared where possible by purification, doping, deposition as thin films, heat treatment, crystal growth, etc. These materials will be examined by optical and electron microscopy, X-ray and electron diffraction, and X-ray spectrometry to determine crystal structure, deformations, impurities, film thickness, etc. The pertinent electrical and magnetic properties of these materials will be measured and studied to determine unique and unusual properties or phenomena which these materials exhibit. The possible utilization of these materials in composite molecular electronic circuits will be considered and investigated.

*Ohio State U., Columbus.
STRUCTURE CHANGES AND RELEVANT PHYSICAL AND CHEMICAL PROPERTIES ATTENDANT TO CERTAIN CHANGES OF STATE. P. M. Harris. Project 9760(802), Contract AF 49(638)-397; AFOSR, DSS.

The purpose of this investigation is to study relevant physical and chemical properties attendant to certain changes of state, especially order-disorder transformations in solids. This approach is based on the premise that the mechanism of transformation can only be understood if the structures of the initial and final states are known. The research includes: (a) Structure investigations primarily of inorganic structures by x-ray and neutron diffraction methods. Past work on crystals having hydrated ions is to be continued. A continuation of previous work on inorganic crystals exhibiting first and second order transformations is contemplated. (b) The variation of the dielectric constant as a function of temperature and frequency and the magnetic susceptibilities as a function of temperature will be investigated where these measurements would appear to clarify the mechanism of first and second order transformations. (c) The components of the thermal vibration tensor for the atoms of certain crystals of interest will be evaluated from x-ray (or neutron) diffraction data using an IBM-704 computer. (d) In connection with the IBM-704 computations, an attempt will be made to evaluate the electronic field, E, as a function of position in the crystal unit cell from values of the charge sensitivity in the cell.

*Illinois Inst. of Tech., Chicago.
STRUCTURE STUDIES OF IMPERFECTIONS IN CRYSTALS. L. V. Azaroff. Project 9763(802), Contract AF 49(638)-425; AFOSR, DSS.

This is a study of the structure of imperfections in crystals using x-rays. This technique will reveal the electron density distribution in crystals around imperfections. The distribution will be compared with theoretical calculations carried out with considerable care. The imperfections in solids are the factors governing their mechanical properties. This x-ray study carried out with unusual precision will indicate the concentration of electrons around these imperfections. Since bonding between atoms in a solid is electronic, this study should give important information about the source of the strength of solids. Also, since this is a study in semi-conductors, the electronic character of these materials as affected by imperfections will be elucidated.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
VAPOR PHASE CHROMATOGRAPHY AS AN ULTRA-PURIFICATION TECHNIQUE. J. K. Kennedy. Project 5620(802), Internal.

The objective of this research is to investigate new physical and chemical methods for the purification of elements and compounds for direct or indirect application to semiconductor materials. These methods will be capable of removing impurities from a given material so that the resulting matrix will contain no more than a total of one part of impurity to 100 million of matrix. It will aim to study gas-liquid, gas-solid, liquid-liquid, and liquid-to-solid equilibria in an attempt to derive from the results suitable purification techniques based on the principles investigated. The data obtained will be used to design, develop, and construct an apparatus for the purification of solid state materials via volatile intermediates. The purification factors will be determined for each system studied in an attempt to attain final purities in the part per billion range.

*Philips Labs., Inc., Irvington-on-Hudson, N. Y.
X-RAY DETERMINATION OF PRECISION LATTICE PARAMETERS. W. Parrish. Project 9763(802), Contract AF 49(638)-620; AFOSR, DSS.

The need for a new approach in the x-ray determination of lattice parameters was shown vividly by the results of a recent international test of the accuracy of lattice parameter determinations. Fifteen of the most experienced x-ray analysts throughout the world working under carefully controlled conditions cooperated in measuring the same homogeneous samples of three substances, using various existing techniques and methods they had developed. The spread of results was about 0.035% and even after the values which were furthest removed from the others were taken out the spread was 0.014%. Prior published claims for precision were between 0.001% and 0.0003%. It is obvious that such claims cannot be substantiated and that the problem requires careful study from a theoretical as well as experimental point of view. The results should be of the greatest interest in the solid state sciences where precision measurements are required for studies of crystal perfection, the role of impurities, strain, and similar problems. In the field of general physics, there is now considerable doubt as to the precision of the x-ray determination of the

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

Siegbahn-Avogadro number. It is hoped that this research will clarify these problems.

*Cornell U., Ithaca, N. Y.
X-RAY STUDIES OF SOLIDS. L. G. Parratt. Project 9760(802), Contract AF 49(638)-402; AFOSR, DSS.

X-ray spectroscopy provides our most precise and reliable information about the inner states of atoms. This technique is likewise powerful in elucidating the electronic structure of solids--metals, semiconductors and insulators, the heart of many devices and systems. This research is for the purpose of (1) obtaining quantitative information on the electronic structures and density of states in solids; (2) developing a new quantitative method of studying impurity-type states in solids; (3) extending the present inadequate one-electron approximations to a theory of impurity perturbations involving a self-consistent many-electron model.

Deformation, Fracture of Metals and Crack Propagation

Columbia Univ. School of Mines, New York. Quarterly Progress Report No. 2, December 1, 1953-February 28, 1954.
X-RAY MICROBEAM STUDIES OF BRITTLE FRACTURES OF METALS. L.-C. Chang. (Bound with it is The Nature of Fracture in Metals. M. Gensamer, J. O. Brittain and others.) AF 18(600)-898; Task I. March 15, 1954. ASTIA Document No. 67374.

A back-reflection x-ray microbeam camera was used in the study of brittle fracture of zinc and iron. Strain distribution curves obtained by the experimental procedure are given for zinc specimens fractured in slow tension at 77°, 195°, and 225°K; for an electrolytic iron specimen fractured at 35°K; and for a carbon steel specimen fractured at 38°K. The average plastic strain decreases rapidly within a short distance from the fracture surface and remains more or less constant at larger distances. There is good evidence that uniformly distributed plastic strain precedes the plastic process resulting in brittle fracture. A formula is given for the work done to produce the plastic strain associated with fracture, or the plastic work factor, w. An estimation of this factor was accomplished graphically. The w values for zinc and iron are about two orders of magnitude larger than their respective surface energies, and w decreases slightly with decreasing temperature in the case of zinc.

Columbia Univ. School of Mines. New York. CU-7-55AF899-Met. AFOSR-TN-54-359. Also published in Jour. Metals, v. 8: 106-111, Feb. 1956.
CREEP-RUPTURE BY VACANCY CONDENSATION. E. S. Machlin. AF 18(600)-899. November 1954. ASTIA Document No. AD 57328.

The possibility that formation of voids under creep-rupture conditions may take place by the condensation of vacancies has been investigated theoretically. It has been concluded that nucleation of voids under creep-rupture conditions by vacancy condensation is highly improbable; however, growth of pre-existent voids by vacancy condensation is probable. A number of predictions made in this theory have been verified by the data, e.g., the elongation at which voids just become visible has been predicted and found to be in good agreement with experiment. It has been predicted and verified that the product of rupture life and steady-state creep rate for preannealed metals and single phase alloys is an invariant quantity independent of stress, temperature, and atomic number for a given type structure. The direction of the effect of cold work on this product has been predicted and found in agreement with experiment. A number of experiments to further evaluate the vacancy condensation mechanism are described.

Carnegie Inst. of Tech. Dept. of Mathematics. Pittsburgh, Pa. Technical Report No. 4. AFOSR-TN-55-166.
SAFETY FACTORS AND SUPERPOSITION IN THE ELASTIC AND PLASTIC ANALYSIS OF FRAMES. W. S. Dorn and H. J. Greenberg. AF 18(600)-1138. May 1955. ASTIA Document No. AD 64233.

In this study, upper bounds are found for the elastic safety factor for plane structures. This provides an estimate of the plastic safety factor in terms of the elastic safety factor and vice versa for a given system. In addition, upper and lower bounds to both the elastic and plastic safety factors for a superimposed load system are found in terms of bounds on the component load systems.

California Univ. Dept. of Engineering. Los Angeles. Report No. 55-31. AFOSR-TN-55-183.
X-RAY LATTICE STRAINS IN PLASTICALLY DEFORMED METALS. M. Kaufman and D. Rosenthal. AF 18(600)-1022. May 1955.

It has been observed in the diagrams of lattice strains plotted vs the applied stress that a departure from proportionality occurs with the onset of yielding. In the present study, specimens of an Al alloy (face-centered-cubic structure), a Mg alloy (hexagonal structure), and a mild steel (body-centered-cubic structure) were examined. Data indicated that following the initial departure from proportionality a tendency toward tha recovery of the original trend occurs after a few % of plastic strain. This tendency is corroborated by the levelling off of the residual lattice strain diagrams. Similar results were obtained at subzero temperatures (-202° and -320°F). The tendency toward recovery coincides with the change of the strain-hardening exponent found in the ordinary stress-strain diagrams of Al and Al alloys. It can be interpreted in the same way as the changes observed in single crystals and bi-crystals of face-centered-cubic metals; that is, the departure from proportionality is associated with the phenomenon of easy slip, while the tendency toward recovery marks the advent of the difficult slip. A simplified model of heterogeneous plastic deformation based on the above interpretation accounts satisfactorily for the magnitude of the variations which have been observed in the Al alloy.

Reed Research, Inc. Washington, D. C. Report No. RR-1054-B-TN-3. AFOSR-TN-56-292.
A SURVEY AND ANALYSIS OF VISCO-ELASTIC MATERIALS WITH PARTICULAR REFERENCE TO PLATES AND BEAMS. G. C. K. Yeh, J. Martinek and C. Torre. AF 18(600)-1382. June 27, 1956. ASTIA Document No. AD 90004.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

Fundamental formulations for Maxwell solid (visco-elastic material) and Kelvin-Voigt solid (firmo-viscous material) are summarized and analyzed. Equations of motion for plates and beams consisting of such materials are derived. Several recent publications along this line are discussed.

Princeton Univ. James Forrestal Research Center, N. J. Metallurgy Report No. 10. AFOSR-TN-56-435. Also published in Corrosion, v. 13: 783, 786, December 1957.
PLASTIC FLOW OF IRON OXIDES AND THE OXIDATION OF IRON. J. D. Mackenzie and C. E. Birchenall. AF 18(600)967. August 31, 1956. ASTIA Document No. AD 96518.

In this study, it is shown that the oxidation of Fe in O_2 is accompanied by the formation of a central box-like cavity, the dimensions of which are identical to those of the original Fe specimen. The decrease in size of this cavity with increasing temperature and its disappearance when the formation of magnetite and hematite is excluded by a controlled oxidizing potential indicates that the plastic properties of the oxides may be an important factor. This is qualitatively confirmed by the measurement of the relative plasticity of the 3 oxides at 800°-1000°C in a specially designed apparatus, which is diagrammed.

*Pennsylvania Univ., Philadelphia.
ORIGIN OF DEFORMATION BANDS, R. Maddin, D. Kuhlmann-Wilsdorf. Project 9760(802), Contract AF 49(638)-435; AFOSR, DSS.

High temperature plasticity limits the use of many metals. Such deformation takes place with the appearance of deformation bands extending across individual crystals of the metal as the parts of the crystal slip past one another. It is generally agreed that this slipping is made possible by the motion of crystal lattice defects known as dislocations. This investigation is undertaken to test the hypothesis that deformation bands are the boundaries between regions of the crystal which are bent in different directions because of the presence of a large surplus of edge dislocations of unlike kind. It is intended that the investigation will be carried out with single crystals of aluminum or other suitable material grown in slightly curved moulds. The specimens will then be stretched and the location of deformation bands will give data which will provide the test of the hypothesis.

*California Inst. of Tech., Pasadena.
STATIC STRESS DISTRIBUTION AT THE BASE OF A CRACK IN FLAT AND CURVED SHEETS, M. L. Williams. Project 7063(806), Contract AF 33(616)-5740; ARL, MRB.

Observations will be made of the plastic and elasto-plastic stress fields in the vicinity of cracks in initially flat sheets. The effect of sheet curvature on stress distribution will then be determined. Photoelastic techniques, and an extremely fast camera, will be used in portions of this research. Efforts will be made to compute the distortion energy of crack propagation. If possible, both internal and external cracks will be studied, and the curved sheet investigation will be extended to include the case of a cylindrical shape with an initial crack.

*Metallurgy and Ceramics Research Lab., ARL, Dayton, Ohio.
DEFORMATION OF CHROMIUM, H. A. Lipsitt. Project 7024(802), Internal.

A preliminary study of chromium in torsion at high strain rates has revealed an interesting anomaly. The maximum upper yield point was observed at the same temperature as the maximum ductility. This study, accomplished in cooperation with Prof. G. M. Sinclair, University of Illinois, will be continued at various strain rates to determine the activation energy of the upper yield point.

*Michigan Univ., Ann Arbor.
EFFECT OF STATE OF STRESS ON THE FAILURE OF METALS AT VARIOUS TEMPERATURES, S. K. Clark. Project 7021(802), Contract AF 33(616)-6041; WADD, MC.

This program is aimed at a more precise interpretation and prediction of the failure in metals than is now possible. Unifying theories are needed to cover the range between brittle and ductile behavior. The biaxial loading apparatus which maintains a constant ratio of the torsion and tension strain will be used for the testing over a temperature range from -250°F to 200°F. Stress combinations in the plastic range are to be emphasized. The contractor will use tubular specimens and, for comparison, thin sheet specimens of various widths material in order to correlate uniaxial and triaxial stress conditions. An evaluation of the stress redistribution in the plastic state and possible trends for reproductibility to further development of a theory will be attempted. The materials used will be a Bessemer steel AISI B1113 of a special heat treatment and a zinc alloy, Zamac 3, with a refined grain obtained by a special mold casting procedure.

*Carnegie Inst. of Tech., Pittsburgh, Pa.
EFFECT OF STIFFENERS ON FAST CRACK PROPAGATION, J. P. Romauldi. Project 9782(806), Contract AF 49(638)-237; AFOSR, DAS.

This is an analytical and experimental investigation pertaining to the mechanism of fracture in complex built up structures, with particular attention to the effect of riveted stiffeners on the propagation of the crack front. The stress field in the vicinity of a crack tip will be interpreted in terms of a generalized crack extension force, as proposed by Irwin, and general expressions for the effect of stiffeners in the crack front in terms of stiffener spacing, stiffener size, rivet spacing and geometry of stiffener will then be derived. Tests will be conducted to check the accuracy of the theoretical results and to evaluate the fracture resistance of certain aircraft-type alloys.

*Michigan Univ., Ann Arbor.
EFFECT OF SURFACE TENSION OF A LIQUID METAL ENVIRONMENT ON THE FRACTURE STRENGTH OF SOLID METALS, E. E. Hucke. Project 9761(802), Contract AF 49(638)-422; AFOSR, DSS.

This research consists of two phases. In the first, the effect of liquid-solid surface energy is measured before application of stress, on the fracture strength of solid copper. The liquid-solid surface energy will be varied by adding Bi to liquid Pb. Since Pb only partially wets Cu and since Bi can be made to completely wet Cu, it is felt that a continuous variation of wetting angle can be obtained by varying the Bi content of quid Pb-Bi alloys.

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

Copper is being used initially since the absolute grain boundary and surface energies have been previously measured. Phase two of the program will be aimed at measuring the change of wetting angle with stress, up to the fracture stress, for a given liquid-solid combination. This phase is again initially employing Pb-Cu with possible extension to other systems.

*Armour Research Foundation, Chicago, Ill.
ENERGETICS OF CRACK FORMATION AND PROPAGATION, A. G. Metcalfe. Project 7024(802), Contract AF 33(616)-6148; WADD, MC.

A study will be initiated through exploratory research to determine if the genesis of fracture is governed by a thermal activation process. There appears to be a possible connection between the thermal activation processes and the formation and propagation of a crack in brittle material under stress loads, initially critical and then sub-critical.

*Carnegie Inst. of Tech., Pittsburgh, Pa.
ENERGY OF CRACK FORMATION AND CRACK PROPAGATION, C. Wells. Project 7024(802), Contract AF 33(616)-5830; ARL, MCB.

The relative energies of crack formation and propagation under impact loading are being determined. A critical evaluation of the semi-statistical techniques for estimating these energies has been completed. The materials used were a variety of forging steels, heat-treated in various fashions. The impact transition behavior was determined between -321° to 212°F. This program is now concentrating on the anisotropy of the formation and propagation energies as a function of the material anisotropies. Pure metals will be investigated next and finally single crystals.

*New York Univ., N. Y.
KINETICS OF DEFORMATION AND FRACTURE, R. F. Bunshah. Project 7024(802), Contract AF 33(616)-6430; ARL, MCB.

Extremely high speed photomicrographic techniques (about 10^6 F. P. S.) will be used for a basic study of the several processes associated with fracture, and the effects of variables thereon. Included in this study are a measurement of the velocity of slip propagation parallel to the slip direction of single crystal and polycrystalline materials existing in the three common crystal structure types; a study of the kinetics and nature of crack propagation in large grained pure metals, including any variations in velocity as a function of grain orientation or at a boundary; an examination of the kinetics and nature of cleavage in single crystals as a function of temperature and microstructure; a determination of whether a crack progresses uniformly or in discrete jumps for ductile fracture; and a study of crack propagation in polycrystalline aggregates treated so that their undesirable properties are prominent, i.e., steel made temper-brittle.

*California Inst. of Tech., Pasadena.
KINETICS OF DEFORMATION AND FRACTURE, M. L. Williams. Project 7024(802), Contract AF 33(616)-6270; ARL, MCB.

It has been determined that the Ellis type high speed framing camera operating at 100,000 frames per second is suitable for the study of deformation and fracture in metals. A new camera, specifically for this study, is being built. The contractor will conduct exploratory investigations of crack propagation rates in ionic crystals, pure metals and single crystals and study the kinetics of slip and other deformation processes.

*Metallurgy and Ceramics Research Branch, ARL, Dayton, Ohio.
MECHANISM OF CRACK PROPAGATION IN METALS, H. A. Lipsitt. Project 7024(802), Internal.

This is a joint research effort with the Mechanics Research Branch of our Laboratory. Low cycle - high stress fatigue tests in fluctuating tension are being performed. The number of cycles required to initiate and propagate a crack to failure are determined from stop-motion photographic records. Crack growth is shown to be a discontinuous, stepwise process consisting of bursts of growth followed by no measurable growth. The topography of the fractured surfaces correlates well with the growth curve obtained by analyzing the motion pictures frame by frame. This research is being continued in an effort to learn more about the subsurface propagation kinetics.

*Polytechnic Inst. of Brooklyn, N. Y.
MECHANISMS OF FRACTURE OF RIGID POLYMERS, R. Ullman. Project 7021(802), Contract AF 33(616)-6222; WADD, MC.

The program will involve stress cracking investigations on various polymers, macroscopic and microscopic investigations to study distentions and to determine the effects of varying parameters on such properties as birefringence, crystallization and orientation. Materials studied will be characterized by molecular distribution. Attempts will be made to formulate a molecular model of the fracture process.

Diffusion in Metallic Systems

Harvard Univ. Cruft Lab. Cambridge, Mass. Technical Report No. 181.
INJECTION AND DIFFUSION OF HOLES AND ELECTRONS IN A SEMICONDUCTOR. H. Brooks. Sponsored jointly by Office of Naval Research, Signal Corps, and Air Force Office of Scientific Research under N5ori-7601. June 3, 1953. ASTIA Document No. AD 20495.

A small-signal transient solution is obtained for an injected pulse of holes which is applicable to semiconductors that are intrinsic or nearly so, as well as to ordinary n- and p-types. A criterion for the validity of the assumption of space-charge neutrality is discussed. An approximation to take into account the beginning of nonlinear effects for large injected pulses is also derived and discussed. The theory of conductivity pulses resulting from injected carriers in a filament is discussed, and it is shown that the apparent quantum efficiency for photoconductivity in an intrinsic semiconductor can become much larger than $b+1$, where b is the mobility ratio.

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*Abstract of unclassified basic research project supported by U.S. Air Force Office of Aerospace Research, Fiscal Year 1960.

Alfred Univ. New York State Coll. of Ceramics, New York. Technical Report No. 1 for period January 10, 1953 - January 11, 1954. Report AU No. 1. AFOSR-TR-54-9.

STUDY OF BASIC MECHANISM OF DIFFUSION OF METALS INTO CERAMIC MATERIALS. R. C. Turnbull. AF 18(600)591. ASTIA Document No. AD 29788.

Three techniques are discussed for evaluating the concentration changes of radioactive tracers in a diffusion experiment: (1) assays of slices removed from the specimen; (2) measurement of the change of radioactivity at the surface; and (3) photographic means. In this experimental study, samples of optically clear periclase were prepared by splitting large single crystals along crystal planes into specimens 1 cm on a side and .5 cm high. The sides of the specimens were covered with a strippable organic coating. The radioactive metal was condensed on the crystal face. The diffusion coefficients were determined for Fe, Co, and Ni in MgO at different temperatures. The activation energy was obtained by plotting the log of the diffusion coefficient against the reciprocal of the absolute temperature.

American Inst. of Aerological Research, Pasadena, Calif. Technical Report No. AR-1; Report No. 193. AFOSR-TN-54-53.

DISCUSSION AND EXTENSION OF GOLDSTEIN'S MATHEMATICAL MODEL REPRESENTING DIFFUSION WITH HYPERBOLIC PARTIAL DIFFERENTIAL EQUATION (PART I). I. Michelson. AF 18(600)476. January 11, 1954. ASTIA Document No. AD 27865.

The random walk problem examined by S. Goldstein, is given an orientation convenient for comparison with well understood problems, and also for extension to the case in which diffusion is not assumed isotopic. The relationship of the several processes so considered, with the corresponding cases of continuous movements, is explored to determine the necessary modifications required in Fick's Law. The proper formulation of a complete mathematical problem is next considered, and further consequences of the use of hyperbolic equations are pointed out. General properties of solutions are discussed and the detailed analytic procedure given for giving representations of solutions in one case of interest.

Purdue Univ. School of Chemical and Metallurgical Engineering, Lafayette, Ind. Report No. PRF-2. AFOSR-TN-56-35.

UNSTEADY-STATE DIFFUSION THROUGH THIN SHEETS. A. S. Yue and A. G. Guy. AF 18(600)1463. December 6, 1955. ASTIA Document No. AD 80546.

A theoretical formulation is proposed for treating the process of steady-state diffusion in substitutional solid solutions. Equations governing steady-state diffusion and the approach to the steady-state are derived. A proposed experimental arrangement for attaining steady-state diffusion of Zn through α brass is illustrated and analyzed.

Horizons Inc. Cleveland, Ohio. Final Report AFOSR-TR-56-54.

INVESTIGATION OF A NEW METHOD FOR THE DETERMINATION OF THE COEFFICIENTS OF SURFACE DIFFUSION OF METALS. P. F. Mataich. AF 18(600)644. September 10, 1956. ASTIA Document No. AD 110344.

The 3 basically different methods of measurement studied were: (1) an optical method which follows the height changes of a diffusing strip by using interferometric measurements; (2) an electrolytic method which utilizes a solid electrolyte to transform the mass flow of the diffusing atoms into an electric current; and (3) an autoradiographic method which utilizes photographic processes to trace the progress of the diffusing atoms. For the interferometric method, differences in surface level could be theoretically determined with accuracies of ± 10 Angstrom units under ideal conditions. This method gave consistent reproducible results when the base surface was made sufficiently flat. For the different electrolytic systems studied, the diffusion rate was so low that it was masked by background currents inherent in the system. The autoradiographic measurements varied with the film and the small deviations in the processing techniques. However, the radioactive technique has an important advantage in cases where the diffusing layer is very thin. In these cases, the exposure time could be increased to the point where the layer becomes visible whereas the interferometric method would not be sensitive enough to show the layer at all. The autoradiographic and interferometric methods can supplement each other.

Purdue Univ. School of Chemical and Metallurgical Engineering. Lafayette, Ind. PRF Report No. 4. AFOSR-TN-56-194. Also published in Jour. Metals, v. 9: 1204-1206, October 1957.

APPROXIMATE METHOD FOR CALCULATIONS USING CONCENTRATION-DEPENDENT DIFFUSION COEFFICIENTS. A. G. Guy and M. Golomb. AF 18(600)1463. ASTIA Document No. AD 87067.

The diffusion equation is modified to account for variations in the value of the diffusion coefficient with concentration by the use of a special coordinate system x_o, such that for every elementary region dx there is a corresponding region dx_o of such thickness that $dx/\sqrt{D}\ dx_o/D_o$, where D_o is some constant reference value of the diffusion coefficient. Illustrative calculations are given for the semi-infinite and finite solid conditions for the diffusion of Zn into Cu.

*California Univ., Berkeley.

EFFECT OF STRAIN RATE ON DIFFUSION. J. E. Dorn. Project 9760(802), Contract AF 49(638)-58; AFOSR, DSS.

No completely satisfactory atomistic model for creep has yet matured. Recent investigations, however, have demonstrated that the activation energy for high temperature creep (or tensile straining) is equal to that for self-diffusion. On the basis of these investigations, several dislocation climb models have been suggested for high temperature creep. In each of these models, however, the tacit assumption is made that the equilibrium number of vacancies are present during the process of creep. This assumption is necessary in order to achieve coincidence between activation energies for creep and those for self-diffusion. The above observations emphasize the importance of understanding more completely the effect of strain rate on diffusivity in order to provide the essential background for a more complete understanding of the basic mechanisms involved in high temperature creep. The major objective of this investigation is to determine the effects of strain-rate on the concentration of vacancies in metals at elevated temperatures. In particular, the effect of strain-rate on the self-diffusivity of Ni is being evaluated. These results will be correlated

*Abstract of unclassified basic research project supported by U.S. Air Force Office of Aerospace Research, Fiscal Year 1960.

with current concepts on the mechanisms of creep at elevated temperatures. Some effort will be devoted to a basic understanding of the vacancy mechanism for diffusion and the effect of vacancies on creep.

*Battelle Memorial Inst., Columbus, Ohio.
FUNDAMENTAL STUDIES OF DIFFUSION. A. E. Austin. Project 7021(802), Contract AF 33(616)-6265; ARL, MCB.

A research effort shall be started to study diffusion in metallic systems as follows: (a) investigate grain boundary and lateral lattice diffusion in one metallurgical system (nickel-copper) as a function of grain misalignment, temperature, and time; (b) initiate a study of solute diffusion in anisotropic crystals for one metallurgical system (silver-zinc); and (c) conduct a theoretical analysis at such times when sufficient experimental data have been obtained. The electron probe microanalyzer will be the principal instrument used during this investigation.

*Materials Central, WADD, Dayton, Ohio.
HYDROGEN PENETRATION MECHANISMS. R. J. Barton Project 7022(802), Internal.

This is an investigation of the basic phenomena controlling the entry of hydrogen into metal cathodes. It will investigate those factors of solution composition that effect hydrogen entry by employing a diffusion electrode technique. The hydrogen penetrating an iron diaphragm is determined by an electrochemical technique and at steady state is a direct function of the hydrogen entering the metal. From the non-steady state studies a measure of the rate of diffusion of hydrogen in iron should be obtained. Initial results indicate that the amount of hydrogen entering the diaphragm varies only slowly with the current density above a given threshold value; the charging efficiency increases with decreasing current density attaining an appreciable value near the i_o value.

*Arizona Univ., Tucson.
IMPERFECTIONS IN METALS. C. T. Tomizuka. Project 9760(802), Contract AF 49(638)-790; AFOSR, SSL.

The specific studies shall include the following: (1) Diffusion experiments in pure noble metals will be carried out using radioactive tracer techniques under hydrostatic pressure. (2) Some diffusion experiments will be carried out not as a function of pressure but simply to obtain the activation energy to supplement item 1. (3) Internal friction (anelastic) measurements will be conducted in alpha-brass type alloys; especially the possible isotope effect will be investigated. (4) Some tracer diffusion experiments in alloys will be undertaken to supplement item 3. (5) Resistivity increase of quenched-in-wires of noble metals will be measured as a function of hydrostatic pressure in which the quenching took place.

*Massachusetts Inst. of Tech., Cambridge.
ROLE OF IMPERFECTIONS AND FINE STRUCTURES. M. Cohen. Project 7021(802), Contract AF 33(616)-6873; ARL, MCB.

A study will be made of the role of imperfections and fine structure produced by combinations of plastic deformation and phase transformations (particularly diffusionless reactions) on the enhancement in properties. Techniques will be used to obtain a quantitative determination of imperfections, although a moderate amount of mechanical testing shall be conducted to establish strength levels being produced. The presence of local lattice strains, subgrain particle sizes, and number of stacking faults in the material before and after deformation will be determined by X-ray diffraction. Static and dynamic (thermal) displacements of the atoms from the lattice points will be obtained by measurements of diffraction line intensities to obtain additional information on the lattice bonding. The materials studied will include Ni, Fe-C, Fe-Ni-C, Cu-Zn and Cu-Al. Gold-nickel alloys will also be included to permit the study of the effects of precipitation reactions in a continuous series of alloys.

Elasticity

*Massachusetts Inst. of Tech., Cambridge.
AEROTHERMOELASTICITY. R. L. Bisplinghoff. Project 9782(806), Contract AF 49(638)-219; AFOSR, DAS.

The purpose is to study, both analytically and experimentally, the effects of elevated temperature and resulting thermal stresses on the deformation, stiffness and frequency of vibration of various structural elements important to aircraft and missile components, namely shell-type beams, thin panels and plate-type structures. As a necessary step in the determination of flutter boundaries for tapered cylindrical shells, the influence of taper on their natural vibration modes and frequencies are being established. Since no exact solutions of this problem have been obtained, various approximate methods of approach had to be tried so that reliable conclusions may be drawn about the behavior of these shells from the sometimes diverse results of different approaches. A basic understanding of the process for membrane and plate-like shells has been obtained, and the appropriate methods of analysis have been determined. These results are now being applied to the solution of the flutter problem by incorporating the aerodynamic effects. The flutter of an extremely low aspect ratio free-free delta wing characterized by only chordwise vibratory modes has been studied on the basis both of piston theory and slender body theory aerodynamics. Progress has been made in developing the equation of energy balance for a fluttering system in the supersonic regime; and some simple applications have been made.

*Toronto Univ., Canada.
BEHAVIOR OF AEROSTRUCTURES. E. D. Poppleton. Project 9782(806), Contract AF 49(638)-548; AFOSR, DAS.

The contractor will investigate the validity of nonlinear damage laws for predicting the failure of hypothetical flight structures. As the initial phase of the investigation, applicability of the results of small-scale dynamic tests, to be performed on material specimens, for determining the values of the components in an analytical expression for a structures damage law, in particular when alternating axial loads are applied to the specimens, will be studied.

*Minnesota Univ., Minneapolis.
BEHAVIOR OF CURVED STRUCTURAL SURFACES. L. A. Scipio. Project 7063(806), Contract AF 33(616)-5723; ARL, MRB.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

The objective of this research is to derive analytical methods to predict the structural behavior of curved surfaces subjected to an environment that simultaneously imposes high heating rates, temperatures, loadings, and accelerations. Particular study will be made of the time-dependent effects of various parameters, and to inelastic and large deflection behaviour, up to complete collapse. The theoretical studies will be supported by laboratory experimentation.

*Columbia Univ., New York
DYNAMIC BEHAVIOR OF PLATES AND SHELLS, G. Herrman. Project 9782(806), Contract AF 49(638)-430; AFOSR, DAS.

This research is directed toward obtaining a better understanding of existing theories which govern the dynamic behavior of plates and shells. It is further directed toward developing more adequate theories for similar structural elements such that a greater range of applicability can be realized. It is presently planned to conduct a separate study of axial shear buckling of a plate. In addition, it is contemplated to pursue the study of variational principles for the analysis of thermal stresses and heat conduction in structures. Subsequent studies will deal with the systematic study of frequencies of free vibrations of cylindrical shells.

*Pennsylvania State Univ., University Park.
EFFECTS OF TRIAXIAL STRESSES ON MECHANICAL PROPERTIES OF METALS UNDER HIGH PRESSURE, L. W. Hu. Project 9782(806), Contract AF 49(638)-676; AFOSR, DAS.

This research consists of an experimental and theoretical investigation of the elastic and plastic behavior of metals under triaxial stresses created by subjecting specimens to hydrostatic pressures up to 200,000 p.s.i. with superimposed loading. The immediate research objectives are to develop testing techniques for conducting triaxial stress on elastic constants, plastic behavior and fracture properties of metals. Although the triaxial state of stress is not presently well understood, it is known that prediction of the desired information predicated on linear or biaxial stress theory is not valid. The use of hydrostatic pressure with superimposed loading appears to offer considerable promise for obtaining reliable data for triaxial stress studies.

*Minnesota Univ., Minneapolis.
EXPERIMENTAL AND THEORETICAL RESEARCH ON COMPOSITE STRUCTURES, C. C. Chang. Project 9782(806), Contract AF 18(603)-112; AFOSR, DAS.

This research consists of a theoretical and experimental study of the elastic characteristics of sandwich-type structures subjected to temperature gradients. Particular consideration will be given to sandwich construction with non-isotropic cores and different facing materials. Moreover, this research includes a study of the vibrational characteristics of sandwich panels with linear viscoelastic cores. An additional aspect of this study is concerned with the plastic buckling of simply supported composite panels with orthotropic cores and different face thicknesses. The theoretical phase of this program is being augmented by an experimental phase to establish the validity of the necessary simplifying assumptions used in phrasing the problems analytically.

*Biot, M., New York.
INCREMENTAL AND NONLINEAR THEORY OF DEFORMATION OF SOLIDS, M. Biot. Project 9782(806), Contract AF 49(638)-837; AFOSR, DAS.

This research constitutes a unified treatment of the deformation of solids and is applicable to a large variety of phenomena including finite elastic deformations, flow and creep, stability, incremental deformation and wave propagation in prestressed solids. The original approach to study the nonlinear deformation of solids enables a number of rigorous theories to be developed without recourse to tensor theory or notation. Specifically, a rigorous theory of stability will be presented and its use illustrated by considering problems involving thick plates, buckling of anisotropic and laminated plates, torsional buckling, etc. A nonlinear theory of twist will also be derived for thin sections and will permit investigation of the stability of such sections under axial compression or tension. Other information which will be presented includes (1) a comparison of the elementary equations and those obtained by the more advanced (and powerful) techniques of classical tensor theory, (2) study of dynamical problems and wave propagation, (3) wave propagation in fluids under initial stress, and (4) wave velocities in prestressed media.

*New York Univ., New York.
NONLINEAR THEORIES OF DEFORMATION AND BUCKLING, J. J. Stoker. Project 9782(806), Contract AF 49(638)-161; AFOSR, DAS.

This is an analytical and numerical study of nonlinear theories of elasticity when the non-linearity arises through the relations between stress and displacement that occur in the failure of columns under compression and the buckling of spherical shells. The program deals with both general and specific problems: the derivation and analysis of mathematically tractable formulations of the basic equations on the one hand, and the study of methods of dealing with special cases on the other. Included in the latter are the deformations of thin plates or slender rods under large external force; the buckling of geometrically simple solids; the deformations and buckling of spherical and cylindrical shells; the development of practical computational procedures for the numerical solution of buckling and large-deflection problems; and the establishment of criteria for distinguishing between situations in which thick-shell or thin-shell approximations are appropriate.

*Massachusetts Inst. of Tech., Cambridge.
RANDOM VIBRATION IN AEROSTRUCTURES, S. H. Crandall. Project 9782(806), Contract AF 49(638)-564; AFOSR, DAS.

The research objectives are to study the nature of random vibrational energy and how it is affected by transmission through structures. Principal emphasis will be placed on extension of existing theory which is considered adequate for treating cases where the structural systems are completely linear and where the random vibration is stationary and ergodic. Consideration will thus be given to the problem of characterizing non-stationary random processes. The effect of nonlinear damping and nonlinear elasticity of the structure on the filtering of random vibration will also be examined.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Rensselaer Polytechnic Inst., Troy, N. Y.
THEORY OF DYNAMIC EFFECTS DUE TO VIBRATIONS AND ELASTIC WAVES IN STRUCTURES, G. Handelman. Project 9782(806), Contract AF 18(600)-1586; AFOSR, DAS.

The objectives of the investigation have been reoriented to permit studies in the applicability of the techniques of geometrical optics to elastic wave motion and diffraction problems in solids, and in the extension of methods applicable for ordinary loading to the response of composite structures subjected to random loading. An investigation of the vibration of a rotating beam with a tip mass is being continued to permit study of the second and third modes of vibration.

*Frola, E., Turin, Italy.
RESONANCE, E. FROLA. Project 7063(806), Contract AF 61(052)-231; ARL, MRB.

This effort involves an analytical study on the problem of resonance as it affects the reactions in certain types of elastic structures. The studies will include various cases of free and forced vibrations, with combinations of two stretched rods, as well as one stretched rod and one bent rod. The objective of these studies will be the formulation of mathematical solutions to each problem in a form suitable for use by engineers.

Electrical Properties of Crystals

WADC TN 58-192
MATERIALS INFORMATION CENTERS, L/Col. M. E. Fields and Mr. D. H. Cartolano. September 1958. ASTIA Document No. AD 155885.

Slightly more than one hundred information centers are reported. These centers are engaged in collecting and disseminating information on a variety of materials and hardware. In some cases, access to the information is restricted to members, supporters and government organizations, and in other cases information is disseminated to all inquiries.

WADC TR 58-655
AIR RESEARCH AND DEVELOPMENT COMMAND TECHNICAL SYMPOSIUM 9-10 JULY 1958 - Statler-Hilton Hotel, Dallas, Texas. December 1958. PB 151 627. Order from OTS $7.00.

In perhaps no other period of recorded history has the attention of the world's peoples been more closely focussed upon the endeavors of the scientific community than they are today. In exchange for this newly-acquired fame, the men of science have responded to an astounding degree. Without a doubt, the most singular event they have precipitated is the birth of the Space Age - the opening of a vast new frontier whose possibilities, like its dimensions, are limitless.

The exploration of the immensity of space will call upon vehicles and propulsion systems with characteristics previously believed unattainable. All of the classes of materials which combine to make up these vehicles and their attendant systems must function reliably and durably within an atmosphere alien to that known during their terrestrial life. Cosmic radiation, aurorae, meteorite dust, operation in a near-vacuum, the questionable effects of zero-gravity, and a host of other factors in combination represent formidable barriers to long life of space materials. It may truly be said that of all the building blocks which comprise a weapon system, the materials of construction form the keystone.

The problems which confront us in the development of superior materials may best be resolved by a free discussion of ideas and information between representatives of science, industry, and the military services. It is our hope that the technical papers and the panel discussions of this symposium will provide the opportunity for such a mutual interchange.

*Rochester Univ., N. Y.
EFFECTS OF IMPERFECTIONS ON THE OPTICAL AND ELECTRICAL PROPERTIES OF SOLIDS, D. L. Dexter. Project 9760(802), Contract AF 49(638)-432; AFOSR, DSS.

Imperfections are of great theoretical and practical significance; most of the measurable properties of solids are determined almost exclusively by the presence of imperfections. Since optical properties likewise are largely determined by imperfections, investigations of the latter may be expected to lead to better understanding of the former. Conversely, the well-developed optical techniques will continue to aid us in measuring the properties of imperfections. Consequently, a theoretical study of the effects of imperfections on the optical and electrical properties of solids is undertaken along the following lines: (1) investigation of the role of exchange in insulating crystals; (2) theory of concentration quenching of luminescence; (3) measurement of the vacuum ultraviolet absorption spectrum and photoconductivity of films in rare gases; (4) "boiling off" of incipient F-centers; (5) edge luminescence in cadmium sulfide; and (6) absence of luminescence in alkali halides after excitation in the F-band at liquid helium temperatures.

*Ohio State Univ., Columbus.
ELECTRICAL PROPERTIES OF HIGH-PURITY BORON, M. O. Thurston. Project 9760(802), Contract AF 49 (638)-424; AFOSR, DSS.

The objectives of the proposed research are: (1) to produce boron crystals having the greatest possible purity, size, and crystalline perfection; (2) to measure the physical and electrical characteristics of these crystals; and (3) to study the effects of the addition of carefully controlled impurities. Recent rapid advances in the development of semiconductor devices such as transistors, rectifiers and masers make it increasingly important to study the characteristics of a wide range of semiconducting elements and compounds. Although a tremendous amount of work has been done on the properties of silicon, germanium, various intermetallic compounds, and certain oxides, other semiconductors of potential importance have not been adequately investigated. For example, different sources give values of resistivity for boron ranging from 10^3 to 10^{18} ohm. Similarly, the intrinsic energy gap is given by different authors as 0.5, 1.0, 1.25, 1.55, and 2.0 ev. Carrier mobilities measured in samples of moderate purity have differed by a factor of ten but are believed to be low. There is not at present any agreement even on the crystalline structure of boron or its melting point. Qualitatively, the present knowledge of boron indicates that it is a semiconductor of very high resistivity at room temperature and low resistivity at temperatures of 600-800°C.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Michigan Univ. Research Inst., Ann Arbor.
ELECTROMAGNETIC RADIATIONS IN CRYSTAL DEFECT CENTERS. C. Kikuchi. Project 9763(802), Contract AF 49(638)-875;AFOSR, DSS.

The purpose of this investigation is to obtain definitive information about interactions of electron spin with nuclear spins, electron spins with phonons, and nuclear spins with phonons, and to determine their effects upon the mutual interactions of electromagnetic radiations at crystal defect centers. The unpaired electrons associated with the paramagnetic ions, are like microscopic electric and magnetic probes, and as such can be used for the submicroscopic examination of crystal defects. Crystal defects have pronounced effects upon the properties of materials, such as electrical conductivity and photoconductivity. The disturbance in the distribution of electron charge and spins in the vicinity of such defects can in turn be used to gain detailed knowledge of the mechanisms and control of conduction and other similar properties in crystals.

*Hebrew Univ., Israel.
ELECTRON SPIN RESONANCE EXPERIMENTS ON PARAMAGNETIC IONS AND FREE RADICALS, W. Low. Project 9763(802), Contract AF 61(052)-59: AFOSR, DSS.

The purpose of this series of investigations is to obtain a better knowledge and understanding of paramagnetism in single crystals. The experiments are so designed as to yield information on: (a) level scheme of the ground state and excited states of paramagnetic ions; (b) the magnetic anisotropy; (c) the relaxation mechanisms operative in these ions; and (d) the effect of irradiation in inducing paramagnetism in crystals. In order that the experiments can yield data which are amenable to theoretical treatment, the experiments are performed on selected crystals of high symmetry, i.e. cubic or axial symmetries in which the paramagnetic ions are incorporated as substituted impurities.

*Minnesota Univ., Minneapolis.
ELECTRON-SPIN RESONANCE STUDIES OF SELECTED SOLIDS, T. M. Sanders. Project 9763(802), Contract AF 49(638)-811; AFOSR, SS.

During the course of this research studies will be carried out on: (1) Electron spin resonance and spin relaxation in germanium and other semiconductor materials; (2) Electron spin resonance studies of surfaces whose electron density may be enhanced by electrostatic charging; and (3) Nuclear polarization produced in paramagnetic materials by saturation of electron spin resonance transitions. An object of these investigations will be to determine some of the details of the electronic structure of germanium which correspond to previously observed structures in silicon. These details such as donor conduction band effects have only recently come within the range of the techniques available for study. These techniques of electron spin resonance will also be used in this investigation to provide new information about surface states of semiconductors and insulators.

*Minnesota Univ., Minneapolis.
ELECTRON SPIN RESONANCE STUDIES OF SOLID DEFECTS, J. E. Wertz. Project 9760(802), Contract AF 49(638)-683; AFOSR, DSS.

The purpose of this work is: (1) to identify the defects giving rise to electron spin resonance lines presently observed but unassigned; (2) to seek evidence for centers presumed to occur but not experimentally established; (3) to relate optical absorption, luminescence properties and electrical conductivity of magnesium oxide samples to defects identified by electron spin resonance; (4) to observe electron resonance of defects from helium temperatures up to the point where vacancies are mobile and electronic conductivity is appreciable. The study of magnesium oxide, with a melting point over 2800°C, is particularly advantageous because it has excellent insulating properties at room temperature and high conductivity above 2000°C.

*Detroit Univ., Michigan
ELECTRONIC CHARGE DISTRIBUTION IN VALENCE CRYSTALS, H. Payne, S. J. Czyzak. Project 9763(802), Contract AF 49(638)-852; AFOSR, DSS.

The purpose of this research is to study the electronic charge distribution in valence crystals with the objective of a more detailed understanding of their fundamental properties and of the crystalline state in general. It is expected that this can be accomplished effectively through the study of the electronic charge distribution and the crystal potential derived from it, and from the variations that would occur by the application of an external electric field, the application of a stress, and vibrations in the crystal lattice. The variation of the electronic charge distribution with the application of an electric field is expected to account for the dielectric coefficient of the crystal. The variation due to the displacement of a nucleus from its equilibrium position in the crystal should yield the atomic force constants of the crystal. From this latter information may be deduced the manner in which the crystal potential varies because of lattice vibrations, which is necessary for the determination of the electronic scattering by phonons. Further from this the electrical and thermal conductivities can be obtained when the crystal exhibits semiconducting properties. The experimental effort will be complemented with an appropriate theoretical effort based on recently devised techniques for a more simple and more accurate calculation of the binding energy of a crystal.

*Illinois Univ., Urbana.
ELECTRONIC PROPERTIES OF NON-METALLIC CRYSTALS, R. J. Mauer. Project 9760(802), Contract AF 49(638)-529; AFOSR, DSS.

Research will be conducted upon the intrinsic photoconductivity of the alkali halides for the purpose of determining the energy gap between the valence and conduction bands and other details of the energy level structure. The trapping of electrons and holes in alkali halides will be investigated with emphasis upon the details of the trapping centers as revealed by magnetic resonance techniques. Studies will be made to determine the potential value of thermoelectric power measurements on ionic crystals such as the alkali and silver halides and may be followed by measurements of thermoelectric power of pure and doped ionic crystals.

*Illinois Univ., Urbana.
ELECTRONIC PROPERTIES OF SILVER AND ALKALI HALIDES, F. C. Brown. Project 9760(802), Contract AF 49(638)-579; AFOSR, DSS.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

The electronic properties of suitably prepared silver and alkali halides will be studied by means of electronic and optical measurements at temperatures down to liquid helium. The purpose of this investigation will be to obtain an understanding of such aspects of the electronic properties of the halides as trapping mechanisms, energy levels of traps, factors affecting mobility of electrons and holes, and scattering processes. The value of such research at the present time rests on the fact that these halides all have the same crystal structure, are ionic solids, and are relatively well understood. They now provide a convenient medium for a detailed elucidation of electronic properties of solids. At the present time attention is being directed toward the properties of holes in silver bromide, the magneto-resistance for photoelectrons in silver bromide, scattering in silver chloride and potassium chloride, conductivity in potassium chloride resulting from ion bombardment, and attempts to measure cyclotron resonance in silver bromide.

*Advanced studies Office, RADC, Rome, N. Y.
F CENTERS IN CRYSTALLINE SOLIDS, C. D. Mollenhauer. Project 8503(803), Internal.

An experimental and theoretical program involving the study of relaxation mechanisms and anisotropy of g-factors in single crystals of irradiated calcium carbonate has been initiated. Of interest also are any electron-nuclear spin coupling mechanisms which may exist. The existence of a three energy level scheme will be investigated. Crystals other than calcium carbonate may also be studied.

*Michigan State Univ., East Lansing.
INTERACTION OF ELECTROMAGNETIC RADIATION WITH CRYSTAL LATTICES, D. J. Montgomery, W. G. Hammerle. Project 9760(802), Contract AF 49(368)-622; AFOSR, DSS.

This is primarily a study of lattice vibrations to be carried out through the measurement of infrared absorption and reflection. The interest in this subject has arisen from a recently renewed appreciation by solid state scientists that the factors governing atomic vibrations in solids are not well understood. Materials which are being studied to good purpose now are ionic salts containing separated isotopes such as Li^6F^{19}, LiH, LiD, etc.

*Stanford Research Inst., Menlo Park, Calif.
LUMINESCENCE OF SOLIDS PRODUCED BY SURFACE RECOMBINATION OF ATOMS, H. Wise. Project 9760(802), Contract AF 49(638)-353; AFOSR, DSS.

This program is a study of the conversion of the heterogeneous recombination energy of free-atoms (and other unstable species) into luminescent energy of a solid surface composed of a phosphor such as calcium oxide. Free atoms are produced by passing an electrodeless r.f. discharge through the gas being studied. These free atoms are then allowed to diffuse to the surface of the phosphor, and the resulting luminescence of the phosphor is observed as a function of time, surface temperature, physical and chemical constitution of the phosphor, identity and pressure of the gas, and power input to the discharge. The basic phosphor used in the work to date has been calcium oxide; this substance has been studied both in combination with small additions (typically 0.005 mol fraction) of sensitizers such as Bi, Sb or Mn, and without any sensitizer. Magnesium oxide is also being studied. The following experimental results have been obtained: (1) initial luminescence decreases with increasing temperature of the phosphor; (2) luminescence tends to decrease with increasing time under otherwise fixed conditions, but the nature of the decrease depends on the temperature, and (3) luminescence varies with gas pressure in a manner strongly dependent on electrical power input to the discharge.

*Pittsburgh Univ., Pa.
MAGNETIC AND STRUCTURAL PROPERTIES OF SOLIDS AND SOLUTIONS, F. Keffer, C. Dean, J. Anderson. Project 9760(802), Contract AF 49(638)-323; AFOSR, DSS.

The purpose of this research is to gain an understanding of the relationship between the electronic and magnetic properties of solids and their crystal structures. Specific subjects of investigation include: (1) optical and microwave studies of F-centers in the alkali halides; (2) studies of electron and hole collision processes in semi-conductors and properties of plasma oscillations; (3) splitting of nuclear quadrupole spectra in single crystals; (4) nuclear quadrupole resonance in solid solutions; (5) nature of bonding in wurtzite structures; (6) electromechanical properties of wurtzite structures; (7) studies of antiferromagnetic transitions; and (8) effect of dipolar interactions.

*Georgia Inst. of Tech., Atlanta.
MECHANISM OF THE PHASE TRANSITIONS IN QUARTZ, R. A. Young. Project 9760(802), Contract AF 49(638)-624; AFOSR, DSS.

It is proposed to make a definitive study, by x-ray means, of the mechanism of the alpha beta transition in quartz. The approach proposed involves the temperature dependence of the intensities of diffracted x-rays. These data will be used to determine the changes in the thermal vibrations and positional parameters of the individual atoms as the temperature is gradually varied through the phase transition point. Detailed electron density maps prepared from data obtained at different specimen temperatures are expected to constitute an important means of data analysis. The principal result expected is an understanding of the mechanism of the phase transition adequate for an explanation of the known behavior of other properties, such as the piezoelectric coefficients, at and in the neighborhood of the phase transition temperature.

*Utah Univ., Salt Lake City.
METASTABLE STRUCTURES OF SOLID MATERIALS, P. Gibbs. Project 7021(802), Contract AF 33(616)-5016; ARL, MCB.

High pressures will be used as an additional physical variable in the study of the fundamental behavior of solid materials. Apparatus is being designed and constructed for conducting carefully controlled fundamental mechanical and physical tests at temperatures up to 1000°C and hydrostatic pressure up to 20,000 atmospheres. Studies will be made of creep, electrical resistivity, defect structure, chemical reactivity and many additional phenomena as a function of pressure.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Yale Univ., New Haven, Conn.
OPTICAL ABSORPTION EDGE OF INSULATING CRYSTALS AT LOW TEMPERATURES, R. G. Wheeler. Project 9760(802), Contract AF 49(638)-503; AFOSR, DSS.

The investigation will proceed along the following general lines: (1) group theoretical analysis of the group of the wave vector applicable to the particular space group to which the insulating crystal belongs; (2) optical reflection and absorption interpreted in light of group theoretical selection rules; (3) observation of line spectra near the absorption edge interpreted in light of possible exciton states as predicted from the group theory; (4) magnetic optical effects, Zeeman splitting of exciton lines and the observation of the Landau levels associated with the valence and conduction bands; and (5) further magnetic measurements, such as susceptibility which offers the possibility of eliminating the ambiguities of the location within the zone of the band extrema. The group theoretical selection rules when compared with experiment are not always able to establish definitely the location of the band extrema. Susceptibility sometimes can determine the number of equivalent extrema, thus usually eliminating the ambiguity determined only from selection rules.

*Rochester Univ., N. Y.
OPTICAL AND ELECTRICAL PROPERTIES OF SOLIDS, D. L. Dexter. Project 9761(802), Contract AF 49(638)-433; AFOSR, DSS.

This research may be described broadly as an experimental investigation of those phenomena, for example, photoconductivity and luminescence, associated with the electronic transfer of energy in ionic crystals. The questions of particular interest include the nature of the optical excitation processes, the mechanism of energy transfer, and the role of the simpler kinds of lattice imperfections. The experimental work will consist of such measurements as the spectral dependence of optical absorption and of excitation and quenching of photoconductivity and luminescence; temperature dependence of thermal charge release, of infrared quenching of photoconductivity, and of "fundamental" optical absorption, effects of imperfections introduced by irradiation, annealing, cold work, or addition of impurities; excitation and emission spectra of "fundamental" luminescence; study of the role of intergrain potential barriers in the mechanism of photoconductivity; and a study of hole excitation in photoconducting crystals. These studies will be performed primarily on single crystals of the alkali halides, and such others as the silver halides and cadmium sulfide and lead sulfide.

*Technion Research and Development Foundation, Israel.
OPTICAL PROPERTIES OF ATOMIC DISPERSION IN INERT MATRICES, O. Schnepp. Project 9763(802), Contract AF 61(052)-428; AFOSR, DSS.

Dispersions of metal atoms or ions in such matrix materials as solidified rare gases, solid ammonia, and ice will be prepared and studied. The crystal field perturbation of the metal atoms and ions will be investigated by spectroscopic methods. In the case of ionized metal atoms the electronic properties of the dispersions will be examined. Such research will lead to an improved understanding of how the electron shell of a metal atom is distorted by the electric fields in solids. When the electric fields are strong enough, the shell will be broken and a metal ion and free electron may be formed. Study of the electronic and optical properties of these dispersions may lead to development of new and unusual materials not now in existence.

*Pennsylvania State Univ., University Park.
CRYSTAL-STRUCTURAL BASES FOR SOME PHYSICAL PROPERTIES OF CRYSTALLINE SOLIDS, R. Pepinsky. Project 9763(802), Contract AF 18(603)-35; AFOSR, DSS.

The scope of the research includes: (a) x-ray and neutron diffraction studies of high dielectric, piezoelectric, ferro- and antiferro-electric crystals; (b) x-ray and neutron diffraction studies of other types of polymorphic transitions; (c) search for new materials with advantageous physical properties. The detailed structure analyses of a number of ferroelectrics both by the x-ray and neutron techniques have been of prime importance in elucidating the crystal-structural mechanisms of a good number of ferroelectrics, and have provided sound foundation for theoretical treatment of the behavior of this important class of solids.

*Polytechnic Inst. of Brooklyn, N. Y.
PHASE TRANSFORMATIONS EFFECTS ON THE PROPERTIES OF SOLIDS, E. Banks, B. Post. Project 9760(802), Contract AF 49(638)-827; AFOSR, DSS.

Materials such as low alkali bronzes of hexagonal or tetragonal structure, the transition metallic silicides and germanides, including rare earths, and the ordering effects of substituting aluminum and antimony for silicon and germanium will be studied from the viewpoint of their effects on the electrical resistivity of the material. Phase transformations in single crystals of alkali halides, such as cesium chloride, will be investigated also. In general emphasis will be given to the interpretation of the relationships between the mechanisms of the atomic transformations to the accompanying changes in the electrical, optical and thermal properties of the crystalline substance.

*Pennsylvania Univ., Philadelphia.
PHENOMENA IN VARIOUS SOLID STATE MATERIALS, M. E. Caspari. Project 4150(803), Contract AF 33(616)-6640; WADD, ETL.

The current objectives are to obtain experimental data and theoretical interpretation of: (1) the interaction of electromagnetic waves with semiconductors by means of a microwave Faraday rotation apparatus; (2) the defects produced in alkali halides by vacuum ultraviolet irradiation and their influences on high electric field breakdown; and (3) paramagnetic resonance properties of dilute alloys of various rare earth metals.

*Maryland Univ., College Park.
COLOR CENTERS IN ALKALI HALIDES, W. G. Maisch. Project 9760(802), Contract AF 49(638)-627; AFOSR, DSS.

The proposed research is concerned with the study of the effect of pressure and temperature on various optical absorption bands in irradiated and additively colored alkali halides. The absorption spectrum of certain colored alkali halides will be studied at pressures up to 50,000 atmospheres using recently developed high pressure apparatus. Correlation of frequency shift, band widths, bleaching, dichroism, and the appearance of new bands with fundamental properties of the lattice will be attempted.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Brandeis Univ., Waltham, Mass.
PHOTO-IONIZATION, ELECTRON TRAPPING AND PHOTO-CONDUCTIVITY IN GLASSES, H. Linschitz. Project 5620(802), Contract AF 19(604)-4536; ERD, EML.

This program has as its object the study of trapped or conduction electrons in glassy media. The use of photo-ionization methods to prepare solvated electrons opens up a much larger class of possible media in which to study the behavior of the solvated charge. Studies on the trapping of electrons in dipolar media will be useful in deepening the basic understanding of the trapping of electrons in ionic lattices (F centers) as well. The project will provide information on such matters as binding energy, adsorption spectrum, mobility, mechanism of movement, and chemical reactivity as a function of solvent composition. Moreover, quite apart from the trapped electron problem, study of the photo-ionization process itself provides important chemical information. The primary effects of the interaction of light radiation and matter are the creation of ion-pairs (electrons and ionized molecules) and excited molecules.

*General Electric Co., Schenectady, N. Y.
PHYSICAL PROPERTIES OF LARGE DISLOCATION FREE CRYSTALS, W. C. Dash. Project 7024(802), Contract AF 33(616)-6780; ARL, MCB.

Large dislocation-free crystals of silicon and germanium have been prepared. These crystals will be studied as a function of temperature to determine their tensile, optical and electrical properties. In addition an attempt will be made to produce similar crystals of some of the more common metals.

*Illinois Univ., Urbana.
PHYSICS OF SOLIDS, F. Seitz. Project 9763(802), Contract AF 49(638)-528; AFOSR, DSS.

Investigation of the properties of insulating and conducting solids, in order to obtain further information on the behavior of the electronic states of these substances, is being continued. Particular attention is devoted to the influence of imperfections in nearly perfect crystals, such imperfections consisting of phonons, excitons, free electrons and holes, dislocations, vacancies and interstitial atoms, and impurity atoms. Principal attention is placed on theoretical aspects. In particular, calculations are made of the energies and other properties associated with imperfections in crystalline solids.

*Johns Hopkins Univ., Baltimore, Md.
ROTATIONAL AND VIBRATIONAL ENERGY LEVELS OF SYSTEMS OF ATOMS, J. B. Newman. Project 4150(803), Contract AF 33(616)-6457; WADD, ETL.

The investigation of molecular crystals and dilute solutions of molecular crystals is being carried on. A literature survey of the field has been completed and theoretical and experimental work is being started. The work has been divided into three aspects: (1) the properties of molecules having internal rotors, (2) the possibilities of the existence of rotational energy levels in solids for which there are allowed transitions, and (3) the combination modes of inter- and intra-molecular vibrations in solids. Currently, work is in progress on the properties of internal rotors in molecules and on the existence of rotational states and selection rules for transitions among them. Detailed studies of the normal modes of crystal lattices of various symmetries, and of particular molecules at the lattice sites, will also be included so that at least for a given model of a molecular crystal some appraisal of possible combination modes may be made. The principal problem to be solved in the experimental work is the production of crystals of sufficient size to make measurements. Growth of iodoform, brucite and portlandite is being attempted.

*Electronic Material Sciences Lab., ERD, AFCRL
Bedford, Mass.
SINGLE CRYSTAL EVALUATION, W. G. Field. Project 5620(802), Internal.

New experimental equipment for the determination of the properties of both ferrimagnetic and paramagnetic materials now permits the rapid and accurate evaluation of the important microwave properties of magnetic materials. Techniques and new equipment for conduction experiments to temperatures near absolute zero are also in use. Coding of the Center's digital computer permits solution of several problems associated with the above outlined experimental program. This not only increases the speed with which data can be obtained but also permits the solution of problems which cannot be handled by ordinary means. Measuring techniques will be utilized for the evaluation of various materials and new measuring techniques will be developed as the state-of-the-art demands.

*Illinois Univ., Urbana
SINGLE DISLOCATIONS IN SOLIDS UNDER STATIC AND DYNAMIC CONDITIONS, R. M. Thomson. Project 9760(802), Contract AF 49(638)-819; AFOSR, DSS.

The purpose of this research is to conduct a combined theoretical and experimental investigation of single dislocations in solids under both static and dynamic conditions. The experimental work is intended to elucidate the electrical properties of a dislocation, the process by which climb of a dislocation occurs, and the details of how a single dislocation moves under a force. The theoretical effort is intended to balance and complement the experimental work by treating problems such as the details of the "field" aspects of the dislocations and the details of the crystal or core effects and their consequences on the dislocation motion.

*Mellon Inst. of Industrial Research
Pittsburgh, Pa.
SOLID STATE STUDIES ON RARE GAS SOLID SOLUTIONS, H. P. Klug. Project 9760(802), Contract AF 49(638)-575; AFOSR, DSS.

Rare gas atoms most nearly of all elements approach the uncomplicated characteristics ascribed to atoms in simple theories of solids. Therefore, a study of the crystals of these rare gases, both pure and mixed, should provide valuable information about some of the basic assumptions of solid state theories. An x-ray diffraction examination to test for adherence to Vegard's law and the presence of ordering of binary mixtures is planned. At the same time, a thermodynamic investigation will provide further information on the energy of formation of crystals of these elements. A special x-ray spectrogoniometer cryostat will be constructed during the first half of this program.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Maryland Univ., College Park.
SOLID STATE THEORY. R. D. Myers. Project 9763 (802), Contract AF 49(638)-399; AFOSR, DSS.

This is a theoretical investigation of several fundamental problems of the solid state. These problems are: (1) Electron-electron and electron-lattice interactions, involving methods for the calculation of correlation energies and specific heats of the electron gas. This phase of the general investigation also includes the relativistic theory of electron interactions and the theory of the pair distribution function. These studies are not only related to the central problems of solid state physics but may have possible application to problems of stellar structure and various plasma problems. (2) The effect of local variations in periodic lattices on the low temperature thermodynamic properties of crystals. (3) The effect of long range interactions between particles on the nature of the vibrational frequency spectrum of sodium chloride, to be studied with the aid of a high speed computer. (4) Using mathematical techniques developed in the study of the effects of localized imperfections on vibrational properties of crystal lattices, calculations of the vibrational contribution to the low temperature equation of state, compressibility and elastic constants will be carried out. (5) A quantum mechanical calculation of the interatomic force constants in monovalent metals using the techniques for studying the effects of localized perturbations on electronic energy levels developed by Koster and Slater.

*Instituto Elettrotechnico Nazionale, Italy.
SPECIAL PROPERTIES AND STRUCTURES OF SOLIDS, G. Bonfiglioli. Project 9760(802), Contract AF 61(052)-328; AFOSR, DSS.

The purpose of this research is to conduct further refined studies of the surface electrical conduction, distribution of traps and the grain "sub-structure" of selected solids such as metals, silicon, germanium, and Al/Cu solid solutions. Specifically, the work is divided into three problem areas: (1) the fundamental process involved in F-center thermoluminescence will be studied and the role played by electron and hole radiative recombination in this phenomenon will be investigated, experiments will be carried out to determine the effects of both DC and AC external electric fields on the thermoluminescent problem; (2) a study will be made of a bombardment induced disarrangement of single crystal surfaces, using transmission electron microscopy techniques, and the structure of the distorted regions of the crystal structure will be investigated; and (3) investigations will be started to determine the effects of temperature on F-center thermoluminescence.

*Brandeis Univ., Waltham, Mass.
THEORETICAL INVESTIGATION OF RELAXATION PHENOMENA IN PARAMAGNETIC CRYSTALS. D. L. Falkoff, J. I. Kaplan. Project 5621(802), Contract AF 19(604)-4107; ERD, EML.

The dissipation of microwave energy contained in the spin system of a paramagnetic crystal is being studied by theoretically examining the spin-spin, spin-phonon, phonon-phonon interactions in paramagnetic crystals. Specific topics include the following: (a) Relaxation of the spin system in solids which relax to several intermediate equilibria before finally coming to equilibrium with the lattice; (b) study of spin-spin cross saturation, making use of better perturbation techniques such as those used in current many-body problems; implications of this work for line shapes and widths will be considered, and (c) Study of the effect of coherence and phase in relaxation by the quantum mechanical density matrix.

*Syracuse Univ. Research Inst., N. Y.
THEORETICAL STUDIES OF PROPERTIES OF QUARTZ AND CARBONS, H. Kaplan. Project 9760(802), Contract AF 49(638)-642; AFOSR, DSS.

A theoretical analysis of nuclear motions rather than electronic structure will be undertaken to relate measured properties with the chemical binding in such crystals as quartz and graphite. It is proposed to determine the extent of the covalent character of the binding and to incorporate the measured elastic and piezoelectric constants and peak infrared vibrational absorption frequencies in a model of the vibration spectrum of quartz. An idealized small crystal of carbon will be used as a model for the calculation of thermal conductivity as a function of crystal size. The thermal conductivity of a perfect two-dimensional Graphite lattice will be computed. These theoretical studies should make it possible to establish quantitative relationships between many and varied physical parameters of such solids as quartz and graphite and to establish a value for the ionic or the covalent character of the binding which will be more accurate than values available at present.

*Brandeis Univ., Waltham, Mass.
CRYSTAL FIELDS AND EXCHANGE INTERACTION BY MEANS OF ELECTRON PARAMAGNETIC RESONANCE, P. Dorain. Project 7021(802), Contract AF 33(616)-6401; ARL, SSB.

An investigation is being carried out by electron paramagnetic resonance of crystals of various dilutions to gain information on the exchange energy and thereby, its contribution, to the stability of the compound.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
CRYSTAL MEASUREMENTS, H. G. Lipson. Project 5621(802), Internal.

A perkin-Elmer Model 112 double pass spectrometer equipped with auxiliary sources and detectors is in operation between 2300 Angstrom unit in the ultraviolet to 25 microns in the infrared. Mirror optics for imaging the beam at external equipment to allow transmission and reflection measurements over a range of temperatures has been designed and constructed. Infrared transmission measurements between 14 and 25 microns at room temperature on samples of hexagonal and cubic silicon carbide, impurity absorption, lattice vibration spectra and the absorption edge have been studied in this material. The high transmission of pure samples of silicon carbide has made possible the identification of some lattice bands which were not previously observed. Also, a difference in the absorption edge between the polytypes of hexagonal silicon carbide has been observed. Measurements have also been made on samples of bismuth telluride and germanium-silicon alloys.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
THEORY OF SINGLE CRYSTAL PROPERTIES, E. Czerlinsky. Project 5620(802), Internal.

The crystalline field acting upon the iron ions in the tetrahedral and octahedral positions of garnets depends on the positions of the oxygen ions, which are being

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

determined by a refined method of X-ray diffractometry. Efforts are in progress to obrain precise expressions for the energy levels of the 3d ions in paramagnetic crystals, such as sapphires and rutiles. A novel type of instability phenomenon of magnetic resonance in single crystals of garnets was discovered and theoretically explained as being caused by a change of anisotropy versus temperature.

*Radio Corp. of America, Princeton, N. J.
THERMAL AND ELECTRICAL PROPERTIES OF SELECTED TRANSITION METAL COMPOUNDS, SEMI-CONDUCTORS AND INSULATORS, F. Rosi. Project 7021(802), Contract AF 33(616)-6165; ARL, MCR.

This research shall be planned and conducted with the ultimate goal of learning more of the principles causing thermoelectric behavior and of the ways these principles can be directed to the control of the thermoelectric characteristics of any material. The principles studied shall include the relative importance of phonon-phonon and phonon-impurity scattering in semiconducting alloys; the relative importance of the various electronic heat conductivity processes (normal electronic heat conduction, ambipolar conduction, and excitons); the relationship between the phonon thermal conductivity and such quantities as the Debye temperature, the coefficient of thermal expansion, and cohesive energy; the temperature dependence of thermoelectric power and the carrier mobility; and the effects of alloying on these parameters.

Fatigue

TR 5623.
SIX TON SCHENCK FATIGUE TESTING MACHINE. A. J. Herzog, Dr., Ing. August 1947.

Univ of Illinois. TR 5726, Parts 1 and 2 (In one volume).
PART 1 - THE EFFECT OF SIZE AND NOTCH SENSITIVITY ON FATIGUE CHARACTERISTICS OF TWO METALLIC MATERIALS. PART 1 - ALUMINUM ALLOY 75S-T. H. F. Moore. W33-038-ac-9225(1482) October 1948.

TR 5775, Parts 1 and 2.
REVERSED BENDING FATIGUE CHARACTERISTICS OF STEEL AND HIGH STRENGTH ALUMINUM ALLOYS AS AFFECTED BY TYPE OF SPECIMEN. PART 1 - EXTRUDED ALUMINUM ALLOYS AND ROLLED 4130 STEEL PLATE. PART 2 - ALUMINUM ALLOY 75S-T6 PLATE. Ture T. Oberg and Robert J. Rooney. July 1949.

TR 52-148.
FATIGUE TESTING UNDER PROGRESSIVE LOADING - A NEW TECHNIQUE FOR TESTING MATERIALS. E. Marcel Prot; E. J. Ward, Captain, Translator. September 1952. PB 109814. Order from LC, Mi $2.40 Ph $3.30.

A method of accelerated fatigue testing is described. Specimens are tested under cyclic stress increasing with time; the increase continuing until failure occurs. The failure stresses are plotted against the square root of the rate of the increase of load. It is proposed that a straight line drawn through the plotted points will intersect the ordinate of zero rate of increase at the endurance limit.

TR 52-234
INVESTIGATION OF PROT ACCELERATED FATIGUE TEST. E. J. Ward, Captain and D. C. Schwartz, Captain. November 1952. PB 122869. Order from LC, Mi $3.00 Ph $6.30.

Prot (reference 1) has proposed a method of accelerated fatigue testing. The method consists of continuously increasing the stress on a test specimen until failure occurs. Specimens are tested at several rates of increasing stress. Then the failure stresses are plotted against the square root of the rate of increasing stress. Prot then proposes that a straight line drawn through the failure stresses will intersect the zero rate of increasing stress at the endurance limit.

This project has investigated the validity of Prot's proposal for SAE 4340 steel and also for flash welded points in SAE 4340. The method was extended in an attempt to utilize statistical analysis of the data to estimate scatter of stress at the endurance limit.

Prot's method gave an excellent estimate of the mean endurance limit and the scatter of the endurance limit of the SAE 4340 steel. However, for the flash welded points, although a fair estimate of the endurance limit was obtained, the estimate of the scatter was considered unsatisfactory.

Before Prot's method can be recommended for general use, even for steel, more extensive investigation must be conducted to confirm this preliminary study.

Univ. of Minnesota. TR 53-122
REVIEW OF PREVIOUS WORK ON SHORT-TIME TESTS FOR PREDICTING FATIGUE PROPERTIES OF MATERIALS. Franz H. Vitovec and Benjamin J. Lazan. AF 33(038)-20840. August 1953. PB 111374.

Experimental observations relating to the fatigue process and theories of fatigue are briefly reviewed. Short-time fatigue testing methods are systematically reviewed and critically discussed. The classification of the methods is based on the relationship of fatigue properties to static properties, to stress-strain characteristics under reversed stress, and to other physical properties. Other methods discussed involve assumptions regarding the shape of the S-N curve. Also tests utilizing special loading conditions are reviewed. Advantages and applicability of the different methods are discussed.

Univ. of Minnesota. TR 53-167.
THE EFFECT OF SPECIMEN SURFACE AS A DISCONTINUITY IN FATIGUE PHENOMENA. F. H. Vitovec. AF 33(038)-20840. September 1953. PB 120309. Order from LC, Mi $3.90 Ph $10.80.

Experimental observations relating to the fatigue process and theories of fatigue are discussed. A picture of the mechanism of fatigue considering all experimental observations is developed. The influence of the free surface of the specimen on the strength of surface grains is discussed. Considering the effect of the free specimen surface, the influence of size of specimen, shape of cross section, and the effect of notches on fatigue strength are

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

explained. The effect of sharp notches in a material is shown in relationship to the propagation of fatigue cracks.

Ontario Research Foundation. TR 53-184.
FATIGUE PREDICTION BY MEANS OF THE CYCLOGRAPH. R. L. Cavanagh. AF 33(038)-19151. November 1953. PB 123641. Order from LC, Mi $6.90 Ph $22.80.

Tests were conducted on SAE 4340 samples in different heat treated conditions to determine whether changes in magnetic and electrical properties at low field strengths could be correlated with fatigue damage. To indicate the changes in magnetic and electrical properties, the Cyclograph was used. This is a high frequency, nondestructive, magnetic test instrument. Some tests were also conducted to evaluate the application of the method for a short fatigue test to obtain the endurance limit.

Test equipment, specimens and procedures are described in detail. Test results are presented in the form of tables and curves. The static bend test in combination with the fatigue test seems to be the best and most convenient test of those investigated to attempt to determine fatigue damage. It can generally be concluded that it is possible to determine whether a steel part has been loaded above or below the endurance limit while it is in service and that by application of low static loads in the same sense as the fatigue load it appears that the extent of fatigue damage can be approximated.

National Bureau of Standards. TR 53-393, Part 1.
DEVELOPMENT OF A FATIGUE DAMAGE INDICATOR: PART I - PRELIMINARY INVESTIGATION. Darnley M. Howard. AF 33(038)-51-4061. December 1953. ASTIA Document No. AD 27588. PB 134586. Order from LC, Mi $3.30, Ph $7.80

This report describes an investigation that was conducted in an attempt to develop a fatigue damage indicator which can be used in aircraft to warn of impending fatigue failure. Five different methods of indicating fatigue damage were investigated; four were subsequently abandoned as all tests on these four methods were unsuccessful. The fifth method investigated is based on Miner's theory of cumulative damage and measures fatigue damage by means of a fine aluminum alloy wire bonded to an aluminum alloy fatigue specimen. The wire is subjected to a known amount of fatigue damage before it is attached to the specimen and is therefore expected to fail in fatigue before the specimen and give warning of impending failure in the specimen. The results of tests on this method indicate that further tests might produce a workable fatigue damage indicator.

Univ. of Michigan. TR 53-437.
THE INFLUENCE OF SURFACE TREATMENT ON THE FATIGUE PROPERTIES OF TITANIUM AND TITANIUM ALLOYS. Lars Thomassen, Maurice J. Sinnott and Albert W. Demmler, Jr. AF 33(616)-26. February 1954

The fatigue properties of commercially pure titanium (Ti-75A) and a titanium alloy (RC-130B), as determined by a rotating beam type of test, have been evaluated as a function of several surface conditions. A shot-peened surface improves the fatigue properties of both alloys although the effect on RC-130B is noticeable only at the higher stress levels. A ground surface on both alloys showed the poorest fatigue properties of any of the surfaces that were prepared by mechanical methods. Four other types of surfaces prepared in different ways, such as hand finishing, rough machining, and electro-polishing showed some significant differences in fatigue properties but generally were quite similar. Oxidizing or nitriding the surface of both alloys markedly lowers the fatigue strength.

Univ. of Minnesota. TR 54-20.
FATIGUE FAILURE UNDER RESONANT VIBRATION CONDITIONS. B. J. Lazan. AF 33(038)-20840. March 1954. ASTIA Document No. AD 39439. PB 12286. Order from LC, Mi $3.60, Ph $7.30.

The nature of resonant vibration and the accompanying amplification of fatigue stress are discussed in relationship to the damping energy absorbed by a vibrating system. The resonance amplification factor is defined as a measure of the severity of a resonant condition. The sources of damping in a vibrating system are discussed and classified according to whether they are external (structural) or internal (material). Data on the internal damping properties of a variety of structural materials are presented and the generalized behavior is discussed. In cases where internal damping is significant, the importance of both fatigue strength and damping properties of materials as joint criteria for resonant strength is demonstrated and quantitatively expressed. The analyses are made in terms of the resonant strength constant for the material (the material factor) and the volume-stress function of the part (the part factor).

Univ. of Minnesota. TR 54-64.
FRICTIONAL DAMPING AND RESONANT VIBRATION CHARACTERISTICS OF AN AXIAL SLIP LAP JOINT. James H. Klumpp and Benjamin J. Lazan. AF 33(038)-20840. March 1954. ASTIA Document No. AD 31155. PB 132285. Order from LC, Mi $3.30, Ph $7.80.

A brief summary of the various methods of damping analysis is presented. Data are procured on the frictional properties of mild steel in reciprocating sliding motion. The variation of the kinetic coefficient of friction as a function of normal load, lubrication, and number of cycles of motion is studied. The friction testing apparatus is considered as a vibrating system with Coulomb damping and its frequency response and damping energy are analysed.

Univ. of Minnesota. WADC TR 54-200.
PRELIMINARY INVESTIGATION OF THE EMANATION FROM COLD-WORKED METALS. F. H. Vitovec. AF 33(038)-20840. July 1954. ASTIA Document AD 41227 PB 130367. Order from LC, Mi $3.00, Ph $6.30.

The disturbance of metal surfaces causes an emanation which can be detected by either Geiger-Mueller counter (Kramer effect) or by photographic emulsions (Russell effect). It was found that the photographic action is caused by the formation of hydrogen peroxide. Various explanations of the origin of the Kramer effect have been attempted, but none of them are completely satisfactory.

Univ. of Michigan. WADC TR 53-437.
THE INFLUENCE OF SURFACE TREATMENT ON THE FATIGUE PROPERTIES OF TITANIUM AND TITANIUM ALLOYS. Lars Thomassen, Maurice J. Sinnott, Albert W. Demmler, Jr. AF 33(616)-26. October 1954.

The fatigue properties of a commercial all-alpha alloy, an experimental all-alpha alloy, and an experimental all-beta alloy, as determined by a R. R. Moore rotating beam type of test, have been evaluated as a function of three different surface conditions. Shot peening improves the fatigue life while grinding decreases the fatigue life, as compared to a hand finished surface. The extent of the increase or decrease varies with the fatigue life range.

Notch fatigue properties of alloys Ti-75A, RC-130B, A-110AT, and experimental all-alpha and all-beta alloys have been determined. Notch types and methods of notch

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preparation have been evaluated. Rolling operations produce notches that do not adversely affect the fatigue lives. Grinding of notches, either carefully controlled diamond grinding or commercial grinding, very markedly decreases the fatigue life.

Univ. of Minnesota. WADC TR 55-86.
FATIGUE CRACK DETECTION METHODS. L. J. Demer. AF 33(038)-20840. January 1955.

The factors involved in fatigue crack detection are discussed and the various nondestructive and destructive methods available for the purpose are described. Emphasis is placed on the use of these in laboratory fatigue tests on machined specimens. Numerous references are given both to literature dealing with the methods themselves and to investigations in which these methods have been employed. A critical evaluation is made of the various methods and the characteristic of each are set forth.

WADC TR 54-462
AN ANALYSIS OF NOTCHED UNIAXIAL FATIGUE DATA. William E. Dirkes. March 1955.

An analysis of the failure trends of fatigue data is presented in terms of the nominal applied stresses. Fatigue failure stresses are related to the maximum tensile stresses applied to test specimens. The concepts presented are used as a means of expressing notched specimen test results as functions of unnotched specimen characteristics.

Test data for 2014-T6(14S-T6) and 2024-T4(24S-T4) aluminum alloys and SAE 4130 steel were taken from WADC TR 52-307 and NACA TN 2639 for use in this analysis. The test data available are in agreement with the concepts presented; however, additional data are required to prove the analysis.

Data for failure of unnotched fatigue specimens are used as the basis for the development of a method for predicting fatigue test data for notched specimens by a semigraphical method. Fatigue strengths for notched specimens can be predicted for various mean strengths from tensile test data, fatigue data for unnotched specimens, and a single SN curve for notched specimens.

The method as presented is limited to ductile materials tested with axially applied loads and is applicable for fatigue strengths determined for large numbers of cycles.

Curtiss-Wright Corp. WADC TR 54-531.
INVESTIGATION OF MATERIALS FATIGUE PROBLEMS APPLICABLE TO PROPELLER DESIGN. H. N. Cummings, F. B. Stulen, W. C. Schulte. AF 33(616)-493. May 1955.

SAE 4340 steel, aircraft quality, from a single heat, at three high-hardness levels, was tested in rotating bending, and statistically determined S-N curves of constant probability of strength for smooth and for notched specimens were established. In addition, exploratory tests were made, by the Prot method, on the 4340 steel, and, by conventional methods, on ultra-high strength 4350 steel, for the mean endurance limit. The effect of inclusions on fatigue strength was studied, not only for the above tests but also for a small lot of vacuum-melted "inclusion-free" steel. A preliminary study was made of the possibility of using ultrasonic techniques for locating and measuring inclusions, and for detecting and measuring early fatigue cracks.

Univ. of Minnesota. WADC TN 55-232.
ANALYSIS OF SLIP DAMPING WITH REFERENCE TO TURBINE BLADE VIBRATION. L. E. Goodman, J. H. Klumpp. AF 33(038)-20840. June 1955.

Energy of vibration may be dissipated by microscopic slip on interfaces where machine elements are joined in a press fit. In this report slip damping is studied as an agent in reducing turbine blade resonant stresses and prolonging turbine life. A general theory of slip damping is developed and an expression for the energy loss per cycle of oscillation is found. The predictions of the theory are compared with the results of controlled experiments. It appears that the theory is in satisfactory agreement with experiment and with measurements made on turbine blades elsewhere in this country and abroad

The implications of the general theory in the design of turbine blades are discussed. It appears that slip damping is capable of being an effective agent in reducing resonant stresses, especially in the "stall-flutter" condition where aerodynamic damping is inadquate. The design of a slip damping joint which would achieve theoretically possible energy decrements much larger than are present in existing commercial construction is shown to depend on the maintenance of an optimum contract pressure.

Columbia Univ. WADC TN 55-273 Part 1.
CUMULATIVE FATIGUE DAMAGE OF AIRCRAFT STRUCTURAL MATERIALS - PART 1: 2024 and 7075 ALUMINUM ALLOY. A. M. Freudenthal, R. A. Heller, P. J. O'Leary. AF 33(616)-2274. June 1955.

The object of this investigation is to determine the effect of randomly varying stress-amplitudes representing gust or maneuver load sequences encountered in flight on the fatigue life of AA 7075 and AA 2024 aluminum by testing small rotating beam specimens subject to random time series of stress-amplitudes derived from specific frequency distributions of stress-amplitudes. The tests were carried out on specially built random fatigue testing machines of a type recently developed in the Civil Engineering Research Laboratories of Columbia University.

Over a thousand specimens were tested. Three sets of six stress levels were used for each material with which nine different load distributions were constructed. In addition, conventional constant amplitude tests were carried out to determine the S-N diagrams. The results were statistically analyzed and presented on the basis of two and three parameter extreme value distributions. While a detailed evaluation of the test results will be presented in the second part of this report, the tentative statement can be made that according to the test results at present available, only for the least severe stress distributions within the low range of stresses does the sum of the cycle ratios itself provide a rough estimate of the cumulative damage under varying stress-amplitudes.

Univ. of Minnesota. WADC TR 55-86 Part 2
FATIGUE CRACK DETECTION METHODS. L. J. Demer. AF 33(038)-20840. June 1955. ASTIA Document AD 75478 PB 143877. Order from LC, Mi $4.50 Ph $12.30

Current fatigue literature contains numerous references to a few recent studies in which fatigue cracks have been detected very early in the specimen life. This paper attempts to clarify such isolated observations by making a detailed review of the available experimental data in relation to crack initiation and growth in laboratory fatigue test specimens. The data are analysed considering the nature of the test material, the character of the specimen, the stress level of the test, and the manner of testing. Conclusions are reached in regard to the effect of these factors on the percentage of fracture cycles at which fatigue cracks are detected. The formula of

A. K. Head for the actual growth of fatigue cracks is reviewed and its agreement with experimental results is discussed.

Univ. of Minnesota. WADC TR 55-225.
STRENGTH, DAMPING, AND ELASTICITY OF MATERIALS UNDER INCREASING REVERSED STRESS WITH REFERENCE TO ACCELERATED FATIGUE TESTING. F. H. Vitovec, B. J. Lazan. AF 33(038)-20840. June 1955. ASTIA Document No. AD 75853. PB 132275 Order from LC, Mi $3.00, Ph $6.30.

The purpose of the work was to investigate the damping, stress-strain, and failure properties under uniformly increasing stress amplitude and to determine the relation of these properties to conventionally determined fatigue strength. Data are presented on SAE 1020 Steel, 24S-T4 Aluminum Alloy, SAE 4340 Steel, and RC-55 Titanium under rotating bending stress amplitudes which (a) are progressively increased during the test and (b) are held constant as in conventional fatigue tests. The Gough dynamic proportional limit method and the Lehr damping intercept under uniformly increasing stress amplitude were found to agree with the conventional fatigue strength only for certain materials and to be misleading in other cases. The failure stress at different rates of stress increase and different starting stresses was determined to evaluate the reliability of the Prot short-time fatigue testing method. For the materials tested Prot starting stress below the cyclic stress sensitivity limit (in the region where damping is unchanged by stress history) has practically no effect on the Prot failure stress. In general, the test results for the four materials indicate that the Prot method indicates the conventional fatigue strength with a reliability of 10 per cent. The use of modified Prot methods which utilize exponents n other than 0.5 were not significantly better for indicating fatigue strength.

Univ. of Minnesota. WADC TN 284.
EFFECT OF MATERIAL DAMPING AND STRESS DISTRIBUTION ON THE RESONANT FATIGUE STRENGTH OF PARTS. Egons R. Podnieks, Benjamin J. Lazan. AF 33(038)-20840. September 1955.

The behavior of various types of parts under resonant vibrations is reviewed considering material hysteresis as the only form of damping present. Three different criteria are introduced for comparing materials and parts for resonant operation: total damping - D_d oc, resonance amplification factor - A_r ($A_{\frac{1}{2}}$), and resonance exciting stress - S_g ($S_{\frac{1}{2}}$). Graphical and analytical procedures are derived for determining the unitless damping energy oc and the unitless elastic strain energy β. Both relate the effect of the shape of the part and the stress distribution to the resonant behavior of a part. The relationship of stress distribution to the shape of the damping energy curves and the resultant effect of the above criteria is discussed. The influence of the cyclic stress history on resonant behavior is also indicated. In comparative examples the relative merits of five different types of structural materials (type 403 alloy, titanium - RC55 annealed, RC 55 cold-worked, RC130B, and glass fabric laminate) based on above criteria are discussed in connection and with application to various types of parts. Resonant fatigue curves are introduced and discussed for the same examples.

Univ. of Minnesota. WADC TN 55-460.
EFFECT OF PRIOR CREEP ON THE REVERSED STRESS FATIGUE PROPERTIES OF TEMPERATURE RESISTANT ALLOYS. Franz H. Vitovec, Benjamin J. Lazan. AF 33(616)-2803. September 1955.

The purpose of this work was to investigate the effect of prior creep on the fatigue properties of the temperature resistant alloys, N-155, S-816, and Stellite 21. In order to separate the effects of stress, time and temperature variables, four different types of tests have been performed: conventional axial reversed stress fatigue tests, creep tests, Prot tests with specimens aged unstressed for various times, and Prot tests with specimens which were subjected to various amounts of creep prestrain.

Increasing amounts of prior creep caused first a slight increase and then a rapid decrease of the Prot fatigue strength of N-155 and S-816. For Stellite 21, the Prot failure stress decreased continuously with increasing prestrain due to aging effects during the creep prestrain. The criterion found to most accurately predict fatigue damage was the percent creep rather than the state of creep.

Univ. of Minnesota. WADC TN 55-527.
A REVIEW OF OBSERVATIONS ON THE CRACKING CHARACTERISTICS AND FRACTURES OF LABORATORY FATIGUE SPECIMENS. L. J. Demer. AF 33(038)-20840. September 1955.

This review examines experimental observations relating to characteristics of the fatigue cracking and fracture of laboratory specimens both of single crystals of metals and polycrystalline metallic structures of various crystal habits. The sections deal with the formation and the paths of fatigue macrocracks, the occurence of fatigue macrocracks, the characteristics of fatigue fractures and certain associated phenomena, and the path of fatigue macrocracks under combined stresses. The object of the report is to provide background for further study of the mechanism of the fatigue process in metals.

Curtiss-Wright Corp. WADC TR 55-456.
RELATION OF INCLUSIONS TO THE FATIGUE PROPERTIES OF SAE 4340 STEEL. H. N. Cummings, F. B. Stulen, W. C. Schulte. AF 33(616)-493. October 1955.

From studies of an unusually large number of fatigue data, certain trends in the relation of inclusions to fatigue strength of SAE 4340 steel were brought to light. After discussing these trends in some detail, an explanation concerning the mechanism of fatigue is proposed and various predictions are made based on the proposed explanation.

Cornell Aeronautical Laboratory, Inc.
WADC TR 55-226
EFFECT OF CYCLIC LOAD FREQUENCY ON THE CREEP-RUPTURE AND FATIGUE PROPERTIES OF JET ENGINE MATERIALS. L. A. Yerkovich, G. J. Guarnieri AF 33(616)-42. December 1955. ASTIA Document AD 92492. PB 133925. Order from LC, Mi $4.80, Ph $13.80.

An investigation has been conducted to evaluate the effects of cyclic loading and load frequency on the elevated temperature creep-rupture properties of several jet engine sheet materials. Specifically the behaviors of low carbon N-155, type 321 stainless steel and Inconel X were studied, when exposed to various combined steady and cyclic stresses at various stress amplitudes and temperatures within a wide range of test frequencies.

Data for selected static and dynamic test conditions are presented in various tabular and chart forms to illustrate the influence of direct fluctuating stresses on the creep and rupture characteristics of the test alloys. These data demonstrate that the static load high temperature creep and rupture behavior of N-155, type 321 stainless steel and Inconel X are not always altered by the

superposition of cyclic stresses; however, damage may be accelerated or retarded depending upon temperature, static stress level and the frequency of the cyclic stress component.

Univ. of Minnesota. WADC TR 56-37. DAMPING, ELASTICITY, AND FATIGUE PROPERTIES OF TITANIUM ALLOYS, HIGH TEMPERATURE ALLOYS, STAINLESS STEELS, AND GLASS LAMINATE AT ROOM AND ELEVATED TEMPERATURES. E. R. Podnieks, B. J. Lazan. AF 33(038)-20840. March 1956. PB 128211 Order from LC, Mi $5.40, Ph $15.30.

The significance of the damping, elasticity, and fatigue properties of a material on the resonant behavior of a part is reviewed. The basic unit for expressing damping energy and a procedure for its determination are discussed. Data on damping, elasticity, and fatigue properties at room and elevated temperatures are presented for four types of materials: titanium alloys, high temperature alloys, stainless steels, and glass fabric laminate. The general behavior of these dynamic properties is summarized with regard to the significance of the more important variables. The resonant fatigue properties in form of resonant fatigue curves are determined for several characteristic types of parts by using the above materials. A comparison is made for the materials under different design criteria.

Forest Products Laboratory. WADC TR 55-389. FATIGUE PROPERTIES OF VARIOUS GLASS-FIBER-REINFORCED PLASTIC LAMINATES. K. H. Boller. DO 33(616)-54-14. May 1956. PB 121500. Order from OTS $2.50.

Fatigue strength values are presented for 6 standard and 4 heat-resistant resin laminates reinforced with glass fibers. Fifty-three S-N curves, representing fatigue data between 1 thousand and 10 million cycles, show the effect of fatigue strength of a notch, moisture, fabrics, resins, mean stress levels, angles to warp, and temperatures up to 500°F.

Univ. of Minnesota. WADC TR 56-180. BIBLIOGRAPHY OF THE MATERIAL DAMPING FIELD (With Abstracts and Punched Card Codings). L. J. Demer AF 33(616)-2803. June 1956. PB 121437. Order from OTS $2.75.

This bibliography was compiled as an aid in furthering current and future research in the field of the damping of materials and structures. Consisting of almost 900 entries, it is believed to be the most extensive such collection presently available. An abstract of each reference is included in all but a few cases. A detailed classification system for the Damping Field is described which differs from the ASM-SLA Metallurgical Literature Classification only in the Processes and Property Index. Directions for use of the Damping Field classification system and ASM-SLA punched filing cards in conjunction with the present bibliography are given. Punched card codings are included with each reference in the bibliography so that the entries in this report copy may be used in constructing a personal punched card filing system of the damping field for the individual user.

Univ. of Minnesota. WADC TR 55-497. THE EFFECT OF STATIC MEAN STRESS ON THE DAMPING PROPERTIES OF MATERIALS. Neal L. Person, Benjamin J. Lazan. AF 33(616)-2803. July 1956 ASTIA Document No. AD 97123. PB 121522.

Vibrational stresses resulting from near-resonant operation are often encountered in current engineering practice under conditions which superimpose static mean stresses, such as, jet engine compressor and turbine blades. One method of minimizing vibration amplitude under near-resonant operating conditions is by the use of damping. Therefore, investigation of the damping properties of materials was undertaken.

New bending vibration decay equipment was developed to determine the effect of static mean stress on the damping associated with a given alternating stress. Tests were performed on SAE 1020 steel, 2024-T aluminum, J-1 magnesium, annealed RC-55 titanium, S-816 alloy, glass laminate plastic, and 403 stainless steel. In all cases the maximum stress on the test specimens was kept below the cyclic stress sensitivity limit, below which damping is unchanged by stress history.

403 was the only material that displated a significant change in the damping due to the superimposed static mean stress. Whereas for the other materials the change was less than 30 percent, for 403 (a magneto-mechanical alloy) the specific damping energy decreased 90 percent when the mean stress was increased from zero to 40,000 psi.

Univ. of Minnesota. WADC TR 56-122. EFFECT OF FATIGUE STRESS HISTORY ON ELASTICITY PROPERTIES AND STRESS DISTRIBUTION UNDER ROTATING BENDING. Leonard C. Lidstrom, Benjamin J. Lazan. AF 33(616)-2803. August 1956. ASTIA Document No. AD 97186. PB 121523.

Data are presented on the effect of fatigue stress amplitude and number of cycles on the stress-strain properties of mild steel under reversed axial (tension-compression) stress. Cyclic stress near the fatigue limit is shown to have a significant effect on the stress-strain properties and secant modulus of the material. The general significance of the observed changes in modulus properties on the stress distribution in a rotating beam are discussed.

An analytical method is presented for determining the actual or specific stress-strain relationship from the moment-strain data of rotating beam fatigue specimens. This method is used to calculate the specific stress-strain relationships for mild steel, and these are compared with experimentally determined axial stress-strain data. The agreement is found to be good.

The actual stress distributions in rotating cantilever-beam fatigue specimens are determined from the specific stress-strain relationships. The errors associated with nominal stress based on the accepted linear stress-strain relationship are analyzed. The effects of magnitude of stress and number of stress cycles on stress distribution are discussed. Fatigue data are presented on solid and hollow rotating beams and under axial stress. These data, compared on the basis of nominal stress, show poor agreement (12 to 30%). However, if compared on the basis of specific or true stress, determined by the methods presented, the agreement is reasonably good (3 to 7%).

Univ. of Minnesota. WADC TR 56-289. EFFECTS OF SPECIMEN PREPARATION ON FATIGUE. Franz H. Vitovec, Harold F. Binder. AF 33(616)-2803. August 1956. ASTIA Document No. AD 97211. PB 121576

The effect of surface preparation methods for plain specimens on the fatigue strength is reviewed. The various effects caused by machining, grinding and mechanical and electrolytical polishing are analyzed. A study of the penetration of plastic deformation caused by the notch preparation is presented. Data on the effect of lapping procedure on the Prot failure stress of SAE B1113 steel and SAE 1020 steel are reported. The direct stress fatigue properties of notched specimens from these two

steels were not affected by the direction of lapping.

Univ. of Minnesota. WADC TR 56-181.
FATIGUE, CREEP, AND RUPTURE PROPERTIES OF HEAT RESISTANT MATERIALS. F. H. Vitovec, B. J. Lazan. AF 33(616)-2803. August 1956. ASTIA Document No. AD 97240. PB 121580.

Fatigue, rupture, and creep data at various temperatures obtained under various combinations of mean and alternating stress are presented for the alloys Stellite 31, S-816; 6.3% Mo-Waspalloy, 7% Mo-Waspalloy, M-252, Inconel X-550, 16-25-6 Timken, Crucible 422, Lapelloy and Stainless type 403. Tests were performed under axial stress on unnotched specimens and specimens having theoretical stress concentration factors of 2.4 and 3.4. The data are presented as S-N curves and stress range diagrams to show the effect on the fatigue and creep properties of specimen notch, temperature, ratio of alternating-to-mean stress, and stress magnitude. The role of both creep and fatigue as design factors and the relation of notch sensitivity to loading conditions are discussed with particular reference to temperature and ratio of alternating-to-mean stress.

Ohio State Univ. Research Foundation.
WADC TR 55-483
STATISTICAL EVALUATION OF VARIATION IN ENDURANCE LIMIT AMONG SEVERAL HEATS OF PROPELLER TYPE STEEL. W. L. Starkey, S. M. Marco, R. R. Gatts. AF 33(616)-2468. August 1956. ASTIA Document No. AD 97190. PB 121654.

Samples from three heats of SAE 4330, five heats of SAE 4340, and four heats of SAE 4350 aircraft quality steel were subjected to Prot-type rotating bending endurance tests. All specimens were heat treated to approximately Rockwell C 43 hardness. Separate estimates of the arithmetic average and the standard deviation of the endurance limit were determined for each heat from specimens selected parallel to the direction of mill rolling and from specimens selected perpendicular to that direction. An evaluation was made of the extent of agreement between the data obtained and a normal frequency distribution of the endurance limits of individual specimens. On the basis of normal distribution, confidence limits of 95% probability were established for each estimate of average endurance limit. The effects of various parameters on the average and on the standard deviation of specimen endurance limits were studied. These parameters included the carbon content, anisotropy, nonmetallic inclusion count, hardness, tensile strength, type of fatigue fracture, and origin of heat. Additional endurance tests were performed to compare the results of Prot-type tests with conventional endurance tests.

WADC TR 56-296
NUCLEAR RADIATION OF REINFORCED PLASTIC RADOME MATERIALS. Robert C. Tomashot, Douglas G. Harvey, 1/Lt. September 1956. ASTIA Document No. AD 97254. PB 121936.

Nine different reinforced plastic laminate materials, each made with a different laminating resin, and one alkyd-isocyanate foam core-glass fabric faced sandwich material were subjected to integrated gamma radiation dosages up to 109 roentgens. After completion of the radiation esposures, the materials were tested to determine the flexural, tensile, and compression strengths under both standard and wet conditions. The mechanical properties of the heat resistant plastic laminates were also determined at elevated temperature.

Dielectric constant and loss tangent measurements were conducted on irradiated samples to determine the effect of radiation on the electrical properties of these materials.

Data obtained from these tests show that the mechanical properties were not significantly affected except for one epoxy type resin laminate. None of the materials showed any significant change in electrical properties due to radiation.

Columbia Univ. WADC TN 55-273 Pt. 2.
CUMULATIVE FATIGUE DAMAGE OF AIRCRAFT STRUCTURAL MATERIALS - PART 2. 2024 AND 7075 ALUMINUM ALLOY ADDITIONAL DATA AND EVALUATION. Alfred M. Freudenthal, Robert A. Heller. AF 33(616)-2274. October 1956. ASTIA Document No. AD 110491. PB 121909.

The object of the investigation is to determine the effect of randomly varying stress-amplitudes representing gust or manoeuver load sequences encountered in flight on the fatigue life of 7075 and 2024 aluminum alloys by testing small rotating beam specimens subject to random time series of stress-amplitudes derived from specific frequency distributions of stress-amplitudes. The tests were carried out on specially built random fatigue testing machines of a type developed in the Civil Engineering Research Laboratories of Columbia University.

Most of the test results have been presented in Part 1 of the Technical Note. Some recent results are included in the present report. An approximate non-linear cumulative damage theory based on a thermal mechanism of fatigue supported by a purely statistical approach is developed. The theory approximates test results reasonably well for the unnotched specimens used in the investigation. Its validity, for specimens having stress concentrations, is currently being tested.

Univ. of Minnesota. WADC TR 56-127.
EFFECT OF CHANGING CYCLIC MODULUS ON BENDING FATIGUE STRENGTH. A. A. Blatherwick, B. J. Lazan. AF 33(616)-2803. October 1956. ASTIA Document No. AD 110492. PB 121816.

The implications of changing cyclic properties of a material are analyzed by means of hypothetical cyclic stress-strain curves. The effects of cross-sectional shape of specimen and of loading conditions on fatigue strength are investigated through calculations based on the assumed material behavior. Two types of material are considered, one of which has a strain-hardening characteristic, while the other becomes softer under repeated cycling. In the second phase of the work, actual materials are tested in reversed-flexure fatigue to provide experimental verification of the results obtained in the hypothetical analysis. It is concluded that the type of material is exceedingly important in determining the effect of specimen shape and type of test on fatigue strength.

Curtiss-Wright Corp. WADC TR 54-531 Supp. 1.
INVESTIGATION OF MATERIALS FATIGUE PROBLEMS APPLICABLE TO PROPELLER DESIGN. H. N. Cummings, F. B. Stulen, W. C. Schulte. AF 33(616)-493. October 1956.

WADC Technical Report 54-531 reported the fatigue characteristics of SAE 4340 steel as a guide for propeller and rotor designers. Stress levels for probabilities of 10, 50 and 90% survival under repeated loading were determined for a constant life. The knowledge, however, that 90% of a material will survive a given stress for, say, ten million cycles is not adequate for the design engineer. What he would like to know is at what stress 100% of the material will survive. Therefore, the tests

reported in WADC Technical Report 54-531 have been supplemented by additional fatigue tests in the long-life region in order to determine higher survival probabilities, in the order of 99%. Inclusion studies have revealed variability within the steel ingot that caused greater scatter in strength than was predicted by the tests reported in WADC TR 54-531.

Univ. of Minnesota. WADC TR 56-408. INTERRELATION OF FATIGUE CRACKING DAMPING AND NOTCH SENSITIVITY. L. J. Demer. AF 33(616)-2803. March 1957. ASTIA Document No. AD 118157. PB 131025.

The materials tested in this study were the heat resistant alloy N-155, a high carbon steel in two conditions of heat treatment, gray iron, aluminum alloy, J-1 magnesium alloy, and SAE 1020 steel. The number of cycles to initiation of macrocracking and to fracture were determined for both unnotched and notched specimens during fatigue tests performed over a wide range of stress levels. Crack detection was principally by the moist coating and deflection methods. Other supplementary techniques were also employed. The damping and stiffness behaviors of the specimens were observed during the fatigue tests to determine the characteristic changes taking place at the higher stress levels both prior to, and following the initiation of cracking.

Comparisons of the damping and stiffness properties of the specimens prior to cracking are made to determine the correlations existing with the static properties of the materials. Analyses are also made of the tendency toward ease of crack initiation and also of the rates of crack growth in the various materials. The dependence of these properties on the type of material is shown. Relationships are indicated between the damping and fatigue strength properties of both unnotched and notched specimen types. Variations of the fatigue strength reduction factors with number of stress cycles are determined both for fracture data and also for crack initiation data. A possible relation is indicated between the damping and the stiffness properties and the notch sensitivity properties of the materials. In addition, observations are presented on the cracking and fracture behavior of unnotched and notched specimens of the materials tested and the variations in these characteristics with stress level of the tests.

Curtiss-Wright Corp. WADC TR 56-611 INVESTIGATION OF MATERIALS FATIGUE PROBLEMS. H. N. Cummings, F. B. Stulen, W. C. Schulte. AF 33(616)-2876. March 1957. PB 131288. Order from OTS $5.50

The studies of high-hardness steels that were begun under Contract AF 33(616)-493 were continued. S-N curves were obtained for SAE 4340 air-melted steel of 230 ksi UTS and vacuum melted steel of 190 ksi UTS. Several steels of 300 ksi UTS were investigated by Prot tests, and S-N curves were obtained for a 4350 steel of the same UTS. Studies of the relation of non-metallic inclusions to fatigue life and strength of the steels were continued and exploratory studies were made of crack initiation and propagation. The applicability of the Prot method of testing to four non-ferrous metals was also included in the work done under the present contract.

WADC TR 57-310. THE EFFECT OF VARIOUS MACHINING PROCESSES ON THE REVERSED-BENDING FATIGUE STRENGTH OF A-110 AT TITANIUM ALLOY SHEET. Robert J. Rooney. November 1957. ASTIA Document No. AD142118. PB131606.

The results of reversed cantilever bending fatigue tests on A-110 AT (5% AL - 2.5% Sn) titanium alloy sheet, machined by various processes, are presented. The machining processes employed were the following: Ultrasonic, slab milling, chem-milling, grinding and electrical discharge machining. The effect of shot-peening on the fatigue strength of the "as-rolled" alloy is also presented. The effects of the various machining processes, as well as that of shot-peening, are shown by comparing the fatigue strengths obtained under these conditions with the fatigue strength of the material in the "as-rolled" condition. Results of the measurements of residual surface stress in the "as-rolled" material are also presented.

Curtiss-Wright Corp. WADC TR 57-589. FATIGUE STRENGTH REDUCTION FACTORS FOR INCLUSIONS IN HIGH STRENGTH STEELS. H. N. Cummings, F. B. Stulen, W. C. Schulte. AF 33(616)-5182. April 1958. ASTIA Document No. AD 151162. PB 131816.

Tentative values of Fatigue Strength Reduction Factors for non-metallic non-malleable inclusions in single-nucleus fractures of R. R. Moore rotating beam specimens are determined by two methods. Data for the computations are taken from tests on 309 specimens of SAE 4340 and 4350 steel, of 140, 190, 230, 260 and 300 ksi UTS. Quantitative results are thought to be somewhere near the correct order of magnitude. Qualitatively, it is concluded that the values of the factors depend upon the size of the inclusions and upon the hardness level of the steel. Also, it is thought that for very small inclusions (less than 0.00025 inch) other inhomogeneities inherent in the steel itself dominate the failure of a specimen.

Columbia Univ. WADC TR 58-69 Pt I. ON STRESS INTERACTION IN FATIGUE AND A CUMULATIVE DAMAGE RULE - PART I - 2024 ALUMINUM AND SAE 4340 STEEL ALLOYS. Alfred M. Freudenthal, Robert A. Heller. AF 33(616)-3982. June 1958. ASTIA Document No. AD 155687. PB 140816. Order from LC, Mi $3.60, Ph $9.30.

The object of this investigation was to determine the effects of stress-interaction, under randomly varied exponentially distributed stress amplitudes representing gust and maneuver loads on aircraft wings, on the fatigue life of smooth 2024 aluminum and SAE 4340 steel alloy specimens. On this basis a quasi-linear cumulative damage theory supported by numerous test results is developed. The lack of significance of the conventional endurance limit under random loading is demonstrated.

The tests were performed on specially built rotating bending random load fatigue machines.

Univ. of Minnesota. WADC TR 58-214. EFFECT OF STATIC PRESTRAIN ON THE PROT-FATIGUE PROPERTIES OF UNNOTCHED AND NOTCHED MATERIALS AT ROOM AND ELEVATED TEMPERATURE F. H. Vitovec. AF 33(616)-2803. July 1958. ASTIA Document No. AD 155738. PB 151280. Order from OTS $1.75.

A study was conducted on the effect of short time and creep prestrain on the fatigue properties of unnotched and notched specimens of the alloys 7075-T6 extruded, 2024-T4 extruded, 16-25-6 hot-cold-worked, and S-816 solution treated and aged. Fatigue properties were determined using a Prot-type test with a single loading rate of 0.01 psi per cycle. The unnotched and notched specimens were prestrained at test temperature and then immediately subjected to a Prot-type fatigue test at the same temperature and in the same testing machine. The test

temperature for the short time prestrain and creep prestrain respectively were 75 and 300°F for 7075-T6, 300 and 500°F for 2024-T4, 1200°F for 16-25-6, and 1500°F for S-816. Short time prestrains up to 10 percent were applied. Stresses used for the creep prestrains corresponded to those which produced rupture in 100 to 200 hours at test temperature.

The main factors influencing the Prot-failure stress were residual stresses, metallurgical reactions, and cracks produced by prestraining. Strain hardening and relaxation of residual stress appeared to be of secondary importance for the materials and range of testing variables investigated.

Univ. of Minnesota. WADC TR 58-36.
ENERGY DISSIPATION IN LONGITUDINAL VIBRATION.
C. S. Chang, L. E. Goodman. AF 33(616)-2803. July 1958. ASTIA Document No. AD 155705. PB 151312. Order from OTS $1.25.

The problem of the longitudinal vibrations in a finite prismatic bar with a terminal viscous damper is solved. The complete boundary value problem (forced vibration with arbitrary initial conditions) is first split into two parts according to the method developed by Mindlin and Goodman. One of these parts yields the steady-state solution and the other represents a free-vibration problem, which is then solved by the method of Boussinesq.

Examples of the applications of the general solutions are given and the amount of mechanical energy dissipated through the damper is computed.

Columbia Univ. WADC TR 58-72.
VARIOUS ASPECTS OF THE DISTRIBUTION OF FATIGUE LIVES. E. J. Gumbel. AF 33(616)-3982. July 1958. ASTIA Document No. AD 155747. PB 151211. Order from OTS $1.25.

The report deals with the following aspects of the asymptotic probability function of limited smallest values that is being used for the representation and interpretation of both constant stress-amplitude and random fatigue tests and for the estimate of the minimum fatigue life:

(a) Transformation of the probability functions for large values of the scale parameter;
(b) Probability function for the relative life defined as the fatigue life divided by the characteristic value;
(c) Relation between scale-parameter and minimum life;
(d) Nomograms for the rapid estimation of the parameters of the probability function.

Crane Co. WADC TR 58-128.
HEAT TREATMENT RESPONSE, MECHANICAL PROPERTIES AND STABILITY OF TITANIUM SHEET ALLOYS
E. A. Sticha, F. W. Richards. AF 33(616)-3586. August 1958. ASTIA Document No. AD 155851.

Tensile and bend test data indicate that the three titanium sheet alloys studied in this investigation have formability, when solution treated and quenched from an intermediate temperature in the alpha-beta phase field, equal to or better than annealed materials of these same compositions. Formability is not likely to be improved by heating to moderate temperature but less power would be required for forming. Best ductility is exhibited by the alloys of low interstitial content. Compositions with the greatest total alloy content give the highest strength and lowest ductility.

It is possible to obtain high strength with satisfactory ductility by application of a solution treat, quench and age type of heat treatment to these alloys. The heat treatment for optimum properties varies with the interstitial content and, possibly, with nature of the interstitial elements. However, the interstitial elements seem to contribute to strength properties without detracting from ductility in the heat treated alloys. The solution temperature for maximum strength with satisfactory ductility is sometimes the same as that for maximum formability. Strain influences response to the subsequent aging treatment and results in properties which suggest overaging.

Short-time strength decreases with increasing temperature and, somewhat anomalously, so do ductility values. The effect of interstitial elements persists at high temperatures. The materials exhibit rather good creep resistance at 800°F with Ti: 6Al: 4V alloy having the highest strength and the Ti: 4Al complex alloy the lowest. This is the same order of strength as determined by elevated temperature tensile testing. Exposure at 800°F may produce some embrittlement in the Ti: 6Al: 4V alloys and the high interstitial Ti: 4Al complex alloy but the results are not conclusive.

Curtiss-Wright Corporation. WADC TR 58-43.
RESEARCH ON FERROUS MATERIALS FATIGUE.
Harold N. Cummings, Foster B. Stulen, William C. Schulte. AF 33(616)-5182. August 1958. ASTIA Document No. AD 155772. PB 151243.

Three studies of the effect of fatigue stressing on steel R. R. Moore rotating beam specimens were made, - (1) the rate of propagation of fatigue cracks, (2) the effect, on fatigue strength, of surface hardening by shot peening and plating, by carburizing, and by nitriding, and (3) the transverse properties of SAE 4340 steel. An equation is proposed expressing fatigue crack length as a function of fatigue life. The increased strength produced by surface hardening is indicated and certain limitations on the use of Prot method testing are incidentally brought to light and discussed. Some decrease in transverse strength as compared with longitudinal strength is revealed and discussed for rolled and forged SAE 4340 steel.

Univ. of Minnesota. WADC TR 58-539.
GRAIN SIZE EFFECTS ON FATIGUE AND THEIR RELATIONSHIP TO NOTCH GEOMETRY, STRESS GRADIENT AND SIZE OF SPECIMENS. F. H. Vitovec. AF 33(616)-5449. December 1958. ASTIA Document No. AD 206665. PB 151598. Order from OTS $1.00.

A study was conducted on the effect of grain size, size of specimen and stress gradient on the direct stress fatigue strength of a 2.5 Al-magnesium alloy at room temperature. Specimens with three different grain sizes and seven different notch sizes were used in this investigation. The data are analyzed with regard to relationships between grain size, size of notch, relative stress gradient, stress gradient per grain, and theoretical strength of notched specimens.

Armour Research Foundation. WADC TR 58-430.
INVESTIGATION OF STATIC AND FATIGUE STRENGTH OF METALS SUBJECTED TO TRIAXIAL STRESSES.
William Lehrer & Harry Schwartzbart. AF 33(616)-3237. December 1958. ASTIA Document No. AD 206384. PB 151 594. Order from OTS $2.00.

The objectives of this program were: (1) the determination of the effect of triaxial stresses on fracture in static and dynamic loading and (2) determination of the brittle fracture strength of metal in the absence of any preceding plastic flow. To achieve these objectives, use was made of a composite brazed specimen consisting of a thin disk of soft metal between cylinders of hard metal.

When a load is applied to the brazed bars, a triaxial stress condition is created due to the plastic restraint on the metal by the harder steel cylinder interfaces.

Although base metals of mild steel and 4340 steel, and filler metals of lead, cadmium, and silver were utilized, significant data were obtained only with the silver filler metal and 4340 base metal.

Experimental procedures and equipment were developed for producing sound and voidless brazements over a range of joint thicknesses for accurately measuring joint thickness and for determining the tensile and fatigue properties of such brazements with a minimum of scatter.

The static tensile strength of silver butt brazements in 4340 steel increases with decreasing joint thickness because of increasing triaxiality of stress, reaches a maximum at a joint thickness of ~1.5 x 10^{-4} in., and falls to a relatively constant value of 26,000 psi at thicknesses less than 4 x 10^{-5} in. The maximum strength is greater than 110,000 psi, some 7 1/2 times the strength of cast silver in uniaxial tension. The ultimate tensile strength of pure cast silver has been determined to be approximately 15,000 psi.

The fatigue life of silver-brazed butt joints in 4340 steel increases continuously with decreasing joint thickness for both fluctuating tension and completely reversed axial loading over the entire range of joint thicknesses investigated, 3 x 10^{-4} in. to 1.8 x 10^{-2} in. Also determined and included with these data are the fatigue properties of pure cast silver to simulate the infinitely thick joint.

Relationships between the appearance of the fracture surfaces and the mechanical properties are drawn, where possible.

Parsons Corporation and Battelle Memorial Institute. WADC TR 58-461.
A SYSTEM FOR AUTOMATIC PROCESSING OF FATIGUE DATA. A. J. Belfour, W. S. Hyler. AF 33(616)-3300 and AF 33(616)-3301. January 1959. ASTIA Document No. AD 207792. PB 151596. Order from OTS $3.00.

This report presents and discusses a system for coding of fatigue information on IBM cards for subsequent automatic processing of the data. Certain codes are needed for some types of entries; the design and preliminary development of these codes are included.

The card system devised will permit coding not only the basic fatigue information (stress and lifetime) but also pertinent information describing: (1) the basic material and fabrication, (2) mechanical properties of the materials, (3) specimen design and methods of preparation, and (4) type of test and associated testing procedure. The system is useful in coding information from basic fatigue studies of materials as well as component and structural fatigue studies.

Armour Research Foundation. WADC TR 58-289.
THE EFFECT OF METALLURGICAL VARIABLES ON THE FATIGUE PROPERTIES OF AISI 4340 STEEL HEAT TREATED IN THE TENSILE STRENGTH RANGE 260,000-310,000 PSI. James I. Fisher & J. P. Sheehan. AF 33(616)-3299. February 1959. ASTIA Document No. AD 208321. PB 151661. Order from OTS $3.00.

Eight heats of AISI 4340 were melted by several different methods in an effort to produce a variety of nonmetallic inclusions. The fatigue properties of these steels were studied at the 260,000-310,000 psi strength level by the rotating beam method using both standard and cylindrical R. R. Moore specimens. The endurance limits were determined by the Prot method and by the constant stress method. It was found, after a study of the size of the nonmetallic inclusions in each heat, that a fair correlation existed between the mean width of the largest inclusions and the ratio of endurance limit to ultimate tensile strength. The smaller the mean diameter of inclusions, the higher the endurance limit/tensile strength ratio.

Experiments were also performed in an effort to determine the effect of reducing the amount of retained austenite and dissolved hydrogen and nitrogen on the fatigue properties. No effect was observed, possibly because of the overriding influence of large inclusions in the heat tested.

Univ. of Minnesota. WADC TR 56-127 Pt II.
EFFECT OF CHANGING CYCLIC MODULUS ON BENDING FATIGUE STRENGTH. A. A. Blatherwick, B. J. Lazan, AF 33(616)-5449. May 1959. ASTIA Document No. AD 214385. PB 151897. Order from OTS $1.00.

The influence of the observed increasing cyclic modulus in super-purity aliminum on stress distribution in bending-fatigue specimens is discussed. The resulting stress redistribution is examined for the various types of fatigue tests in common use. Differences in published fatigue data are partially explained in the light of this stress redistribution. It is concluded that for a cyclic strain hardening material, such as high purity aluminum, constant strain (or deflection) tests are more severe than constant moment tests, and the latter in turn are more severe than constant stress tests. Some observations of the development of slip bands and fatigue cracks are presented.

Univ. of Minnesota. WADC TR 58-570.
THE RANDOM VIBRATION OF ELASTIC STRINGS--THEORETICAL. Richard H. Lyon. AF 33(616) 5426. May 1959. ASTIA Document No. AD 214383. PB 151885. Order from OTS $0.75.

The response of a perfectly flexible string with longitudinal deformation to random excitation is studied in some detail. The equations of motion are essentially those of Carrier (Quart. App. Math., 3, 157-165). The modified mean square response for the "elastic" strings is discussed and it is shown that the mean square deflection is diminished from the linear case. From a study of the fourth moments, it also appears that the shape of probability distributions of the transverse displacement are altered, the response to gaussian noise being in general non-gaussian.

*Rutgers Univ., New Brunswick, N. J.
EARLY DETECTION OF MICRO-CRACKS RESULTING FROM FATIGUE OF METALS, S. Weissmann, J. J. Slade. Project 9763(802), Contract AF 49(638)-17; AFOSR, DSS.

Considerable theoretical work has already been carried out under this contract, considering various possible combinations of lattice defects leading to the formation of microcracks. The experimental x-ray work in support of this theoretical study is currently underway. The experimental x-ray studies will comprise the investigation of single crystals as well as that of polycrystalline specimens. Fatiguing of single crystals of silver and aluminum will be carried out in a special fatigue machine. Analysis of the fatigued specimens will be made by an x-ray double-crystal diffractometer method combined with x-ray microscopy and optical microscopy, also by the Guinier-Tennevin focussing Laue method, metallographic etch-pit techniques, and electron microscopy. Fatigued polycrystalline specimens of aluminum and copper will be analyzed by a special x-ray method combining x-ray diffraction analysis with x-ray microscopy and optical microscopy, as well as

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

by double-Bragg reflection techniques. Theoretical studies will continue and will involve mathematical models of lattice discontinuities to represent microcracks and consideration of the dynamics of failure.

*Metallurgy and Ceramics Research Branch, ARL, Dayton, Ohio.
EFFECTS OF NEUTRON IRRADIATION ON THE FATIGUE BEHAVIOR OF METALS, H. A. Lipsitt. Project 7024 (802), Internal.

The neutron irradiation of steel is known to increase the elastic limit of this material. Since the fatigue limit is really a dynamic elastic limit, it would be anticipated that the fatigue limit would, as well, be increased. The available data in this area are very few and do not clearly indicate the validity of our hypothesis; this research, then, is an attempt to study the variation of fatigue properties when a steel is irradiated, and determine the validity of the hypothesis. This is a cooperative program with Mr. W. A. Trapp of the Materials Laboratory, WADD.

*Midwest Research Inst., Kansas City, Mo.
FATIGUE PHENOMENA IN ULTRA SMALL SPECIMENS USING MICROPHYSICAL TECHNIQUES, J. C. Gross-Krentz. Project 7021(802), Contract AF 33(616)-6383; WADD, MC.

The objective is to investigate the micro-physical changes in ultra-small specimens under cyclic loading, using the electron microscope, X-ray diffraction, and other scientific methods, for the purpose of exploring fatigue mechanism. The microphysical changes accompanying cyclic stress have been investigated in copper and aluminum samples by use of X-ray and electron microscope techniques. Small angle X-ray scattering revealed that formation of misoriented subgrains is the most pronounced structural change and that this formation is essentially completed within the first 10 percent of the fatigue life. It was further demonstrated that high temperature annealing does not remove this polygonized structure, but does promote some growth and consolidation of the subgrains. The X-ray showed no evidence of formation of voids in the volume of metal during cyclic stressing. Electron micrographs revealed the formation of deep intrusions in the surface of the copper specimens which may have served as nuclei for crack initiation. Studies were also made of the energy dissipation in the specimens during cyclic load tests. In the current program, a study of volume versus surface effects on fatigue mechanism will be conducted. In addition to this study the following effects will be studied: (1) the effect of cold working and irradiation on the freedom of movement of dislocations; (2) the role of subgrain formation and polygonization in fatigue; and (3) the effect of temperature on the accumulation of dislocations.

*Metallurgy and Ceramics Research Branch, ARL, Dayton, Ohio.
THE MECHANISM OF THE FATIGUE LIMIT IN METALS, H. A. Lipsitt. Project 7024(802), Internal.

The fatigue limit behavior of steel has been shown to be a direct consequence of strain aging which occurs under alternating stress. The present research employs the purest titanium and three dilute alloys containing C, N, and O, respectively. The data gathered to date indicate a marked dependence of fatigue behavior on the amount and type of interstitial element present. This research is being extended to both higher and lower temperatures to study the effects of increasing or decreasing the amount of strain aging that is allowed to occur.

*Birmingham Univ., Gt. Brit.
STRAIN AGEING AND HARDENING AND SOFTENING OF METALS BY FATIGUE, T. Broom. Project 9760(802), Contract AF 6(514)-1182; AFOSR, DSS.

(a) The objective of the first part of this research is to gain a fundamental understanding of the processes which control fatigue behavior. This will be ultimately useful in the development of new alloys of both high tensile and fatigue strengths. This work will draw upon and logically extend a detailed study, just concluded, of the mechanism and kinetics of strain-aging of aluminum magnesium alloys after tensile deformation. The technique to be used will include the accurate determination of stress-strain relationships after strain-aging treatments at various temperatures and for various times. This research will be continued by substituting fatigue cycling for the plastic strain. (b) The objective of the second part of this research is to elucidate a major part--hitherto largely neglected--of the fundamental mechanism of fatigue in annealed and cold-worked pure metals. It is required to obtain reliable data for both hardening and softening, using single crystals and polycrystalline specimens fatigued under a variety of conditions. An analysis of the extremely local conditions which give rise to the formation and growth of a fatigue crack in a softened or hardened matrix will be attempted.

*Massachusetts Inst. of Tech., Cambridge.
ULTRASONIC DETECTION OF FATIGUE DEVELOPMENT, P. Brosens. Project 7063(806), Contract AF 33(616)-6469; ARL, MRB.

The study of metal structures by means of acoustical phenomena has proved very effective in the detection of macroscopic defects, using either reflective or transmission type instruments. However, these techniques are not sufficiently sensitive to warn of impending fatigue. MIT has conducted preliminary experiments which indicate that metals, after a substantial number of stress cycles, will exhibit a marked change in transmission attenuation prior to development of a visual crack, for signals in the low ultrasonic region (below 1.5M cps). Therefore, in this effort, theoretical and experimental studies will be conducted in the application of ultrasonic energy transmission techniques for the detection of fatigue development in materials used for aircraft and missile structures.

Hydrogen Evolution and Embrittlement

Technische Hochschule, Munich, Germany.
WADC TR 59-687
RESEARCHES ON HYDROGEN EVOLUTION. C. A. Knorr. AF 61(052)-142. April 1960. PB 161880. Order from OTS $1.25.

Part I - Investigation of adsorption phenomena on platinum, rhodium, iridium, palladium, and gold, by means of the potentiostatic method of applying a triangular voltage. Formation and decomposition of hydrogen and oxygen coverages as a function of the potential. Determination of the retardation of charge transfers.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

Part II - Investigation of the reactions of hydrogen occluded in Pd-wires with H-acceptors like chromic acid, potassium ferricyanide, and hydrogen peroxide, by the measurement of the longitudinal resistance. Study of the oxide and cyanide layers on the electrode surface with regard to the H^{+} permeability and the influence of poisoning on the decomposition reaction.

WADD TN 60-115.
RESEARCHES ON HYDROGEN OVERVOLTAGES ON METALLIC SINGLE CRYSTALS: TIN. L. Peraldo Bicelli, A LaVecchia, Milano, Italy. AF 61(052)-144. July 1960.

Hydrogen overvoltage on tin single crystal cathodes, oriented following the: (001), (100), (110) planes and on polycrystalline tin, has been measured in perchloric acid solutions with two different concentrations and in hydrochloric acid solutions.

Tafel law holds true; while the parameters are different for the different electrodes.

WADD TR 60-275.
HYDROGEN EMBRITTLEMENT OF TITANIUM ALLOYS. A. E. Riesen, D. H. Kah. October 1960. PB 171394. Order from OTS $1.25.

The effect of various hydrogen interstitial contents between 60 parts per million and 312 ppm on the notch sensitivity and low strain rate embrittlement of four titanium alloys was investigated at room temperature. The materials investigated included three alpha-beta alloys: Ti-6Al-4V, Ti-2Al-6Mo, and Ti-2.5Al-16V; and one all alpha alloy, Ti-6Al-2.5Sn, which were all heat treated per manufacturers recommended schedules. An optimum embrittling effect was encountered between 180 to 220 ppm hydrogen content in the alpha-beta alloys. Over the range of hydrogen content investigated the alpha alloy was not appreciably embrittled.

Politecnico di Milano, Milano, Italy.
WADD TR 60-769.
RESEARCH ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS. R. Piontelli, L. Peraldo Bicelli, A. LaVecchia. AF 61(052)-144. February 1961. PB 171913. Order from OTS $1.75.

Hydrogen overvoltage on silver, lead, nickel, tin and cadmium single crystals has been measured in different experimental conditions. The results have been discussed and it has been observed that the Tafel law is generally followed.

Laboratori di Ellectrochimica, Milano, Italy.
WADD TN 60-269.
RESEARCHES ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS: CADMIUM. L. Peraldo Bicelli, A. LaVecchia, N. Sala Cataffo. AF 61(052)-144. February 1961.

Hydrogen overvoltage on cadmium polycrystalline and single crystal cathodes, oriented following the (001), (1010) and (1120) planes, has been measured in perchloric acid solutions.

The Tafel law holds nearly; the parameters being different for the different electrodes.

Laboratori di Ellectrochimica, Milano, Italy.
WADD TN 60-270.
RESEARCHES ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS: NICKEL. L. Peraldo Bicelli, A. LaVecchia. AF 61 (052)-144. February 1961. ASTIA Document No. AD 258587.

Hydrogen overvoltage on nickel single crystal cathodes, oriented following the (100), (110) and (111) planes has been measured in perchloric acid, hydrochloric acid and in sulfamic acid solutions.

The Tafel law holds nearly; the parameters being different for the different electrodes.

*Politenico di Milano, Italy.
HYDROGEN EVOLUTION. R. Piontelli. Project 7022 (802), Contract AF 61(052)-144; WADD, MC.

The electrochemical evolution of hydrogen from metal cathodes is being studied as a function of crystallographic orientation. Various metals from the three groups: noble, transition, and soft, are to be studied on their major crystal planes in various electrolytes employing high purity techniques. Work is in progress on Zn, Cd, and Ni and essentially completed on Sn crystals.

*National Bureau of Standards, Washington, D. C.
MECHANISM OF HYDROGEN EMBRITTLEMENT OF STEEL USING ISOTOPES. A. M. Adair. Project 7021(802), Contract AF 33(616) 58-18; ARL, MCB.

The relationship between the amount of hydrogen or its isotope electrolytically introduced into steel and the severity of the resulting embrittlement will be investigated under a variety of conditions of electrolysis. The main emphasis will be on determining the hydrogen content required for embrittlement. If the passage of hydrogen through the metal is by ordinary diffusion, and the changes in the distribution of hydrogen within the microstructure is due to the application of stress, the mechanism by which promotors increase the rate of passage of hydrogen through steel will also be studied by determining the effect of promotors on the relative concentrations of hydrogen and deuterium electrolytically evolved. The fundamental data obtained will be used to verify or challenge existing theories of the mechanism of hydrogen embrittlement in ferrous alloys.

*Technische Hochschule, Munich, Germany.
HYDROGEN EVOLUTION. C. A. Knorr. Project 7022(802), Contract AF 61(052)-305; WADD, MC.

The general area of the mechanism of hydrogen reaction at metallic surfaces shall be studied by using pulse techniques to investigate the types and extent of surface layers present and the effects of these on the hydrogen reactions at metal-solution interfaces. Among the factors to be considered are the effects of foreign atoms or ions, the effects of extensive solution clean up, the effects of electrode surface treatment, both anodic and cathodic conditions, and the effects of prior hydrogen penetration into the electrodes. Previous work has emphasized the noble metals, current work will emphasize Fe, Cd, Zn, Ti, Ni. Initial results on platinum have established the potential regions over which the various layers are stable, and the extent of surface coverages. The rate of formation and destruction of these layers has established that the adsorption of hydrogen or oxygen on platinum is irreversible.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

Univ. of Minnesota. WADC TR 58-547
DAMPING ENERGY DISSIPATED BY INTERFACES IN BEAM AND PLATE SUPPORTS AND IN SANDWICH CORES. Theodore J. Mentel. AF 33(616)-5426. December 1958. ASTIA Document No. AD 206667. PB 151611. Order from OTS $1.00.

The maximum energy dissipation which can result from both viscous and dry friction damping between the longitudinal interfaces at the supports of built-in beams and plates is compared with the total material damping. This comparison shows that this type of support damping is relatively unimportant in the vibration attenuation problem for beams, but may be extremely important in the case of thin plates. An alternative method for the damping of beams, that of using sandwich construction with an energy dissipating central core, is found to provide an effective damping mechanism in the cases where support damping becomes ineffective.

Univ. of Minnesota. WADC TR 59-545.
ON THE UTILIZATION OF THE CONCEPT OF BOUNDARY IMPEDANCE IN VIBRATIONS OF BARS. F. J. Wilmers and R. F. Lambert. AF 33(616)-5426. February 1959. PB 161740. Order from OTS $1.25.

This report deals with the utilization of boundary impedance in describing conditions at the extreme boundaries of a bar and its measurement by employing techniques derived from electrical transmission line theory. Important similarities and differences between mechanical and electrical transmission lines are discussed. The concepts are applied to the measurement of a reactive termination and to the development of a "complex conjugate" matching section. These concepts also present a convenient specification of boundary conditions in applications involving random vibrations and fatigue.

Univ. of Michigan Research Institute.
WADC TR 59-339.
EFFECT OF PRIOR CREEP ON SHORT-TIME MECHANICAL PROPERTIES OF 17-7 PH STAINLESS STEEL (RH 950 Condition Compared to TH 1050 Condition). Jeremy V. Gluck, James W. Freeman. AF 33(616)-3368. March 1959. PB 161296. Order from OTS $2.25.

A study was carried out on the effect of elevated temperature creep exposure on the short-time mechanical properties of 17-7 Ph stainless steel in the RH 950 condition of heat treatment. The results were correlated with an earlier study of the TH 1050 condition of the alloy. Exposures were conducted for times of 10, 50, or 100 hours either unstressed or at stresses causing up to 2 percent creep deformation at 600°F, 800°F, or 900°F.

Following the exposures, short-time tension, compression, or tension-impact tests were conducted at either room temperature or the temperature of exposure. A substantial loss in ductility was observed in the room temperature tests following the creep-exposures of the RH 950 condition. At either room temperature or 600°F, a substantial Bauschinger effect was observed in the material subjected to 600°F creep-exposure. This caused an increase in the tension yield strength and a decrease in the compression yield strength as the amount of creep was increased. The ultimate strength was also increased following creep-exposure at 600°F. Little change was found in the other mechanical properties as the result of exposure to creep.

Compared to the TH 1050 condition, the RH 950 condition was initially stronger and maintained its strength better after creep-exposure. The RH 950 condition had a greater loss in room temperature ductility following 800° or 900°F exposure than the TH 1050 condition.

The changes in properties are believed due principally to an aging reaction caused by the continuation of the precipitation of an aluminum-nickel compound under the influence of stress and/or temperature. Plastic strain was also a factor as it produced the Bauschinger effect. Any effects due to strain hardening were minor.

Univ. of California. WADC TR 58-507.
THE EFFECT OF STRESS ON THE CREEP BEHAVIOR OF HIGH PURITY ALUMINUM IN THE REGION OF DISLOCATION CLIMB. A. Bayce, W. Ludemann, L. A. Shepard, J. E. Dorn. AF 33(616)-3860. March 1959. ASTIA Document No. AD 211850. PB 151783. Order from OTS $1.00.

The effect of stress on the creep rate in the dislocation climb region was investigated by increasing and decreasing the stress abruptly during the course of primary creep of high purity aluminum. These investigations revealed that a simple functional relationship between creep rate and stress does not exist; the effect of stress on the creep rate depended on the substructure as well as the stress. In general the creep of aluminum under conditions of changes in stress can be resolved qualitatively in terms of dislocation climb models for creep.

WADC TN 58-369.
OBSERVATIONS ON THE EFFECT OF SURFACE AND STRUCTURE ON THE TENSILE STRENGTH OF IRON WHISKERS. Helmut Weik. May 1959. ASTIA Document No. 212151. PB 151904. Order from OTS $0.75.

Investigations were made of iron whiskers grown by reduction of iron chloride. Characteristic whisker shapes were found to be dependent on the growth conditions. Observations of the surface of the whiskers after the test revealed normal slipping on slip planes connected with low tensile strength in the case where the whiskers were grown from wet chloride. In the case where they were grown by reduction of dried chloride in hydrogen and argon, however, much higher values for the tensile strength were obtained. The strength increases with decreasing whisker diameter. On the surface of some whiskers from this series shearing of surface layers could be observed giving the impression that the whisker surface has a layer or shell structure. By means of microstructure investigations the layers observed in the surface were also discovered in the whisker core. The distance between the layers was found to be constant and about 1200 to 1500 atoms thick. It may be concluded that the layers are a structural element of the whiskers grown under the conditions above mentioned.

Nickel coating of the whiskers increased the tensile strength. In the case of a 100 atoms thick nickel coating the amount of increase was about 100% compared to the strength value of the uncoated whisker. That means that surface effects are important in considering the reasons for the extremely high tensile strength of very thin whiskers.

Univ. of Minnesota. WADC TR 58-570.
THE RANDOM VIBRATION OF ELASTIC STRINGS - THEORETICAL. Richard H. Lyon. AF 33(616)-5426. May 1959. ASTIA Document No. AD 214383. PB 151885. Order from OTS $0.75.

The response of a perfectly flexible string with longitudinal deformation to random excitation is studied in some detail. The equations of motion are essentially those of Carrier (Quart. App. Math., 3, 157-165). The

modified mean square response for the "elastic" strings is discussed and it is shown that the mean square deflection is diminished from the linear case. From a study of the fourth moments, it also appears that the shape of probability distributions of the transverse displacement are altered, the response to gaussian noise being in general non-gaussian.

Univ. of Minnesota. WADC TR 58-569.
RESPONSE OF BARS (WITH INTERNAL AND BOUNDARY DAMPING) TO TRANSIENT AND RANDOM EXCITATION. Robert F. Lambert. AF 33(616)-5426. May 1959. ASTIA Document No. AD 214382. PB 151884. Order from OTS $1.00.

Theoretical analysis of the effects of internal and boundary damping on the forced vibrations of a uniform bar is carried out using perturbation techniques. A first order correction to the poles of the response function is obtained and the dependence upon frequency noted. It turns out that the real part (damping factor) will in general vary with frequency and hence mode number. In the case of internal damping it varies as approximately n^2 where n is the mode number. In the case of a low boundary damping parameter, the damping constant varies as n^2 while for high boundary damping it has roughly an n^4 dependence. These results virtually insure that the response functions (displacement, moment, strain) for random excitation obtained using a generalized Fourier analysis will converge for Grownian motion type excitation. Relationships showing the mode shape corrections for finite boundary impendances are obtained from the perturbation theory. Several experimental studies are suggested which would contribute to our understanding of these damping mechanisms and their relative importance in studies involving random excitation of bars and plates.

Pennsylvania State Univ. WADC TR 57-755.
CORRELATIONS BETWEEN TRANSVERSE AND TORSIONAL SHEAR CREEP DEFORMATIONS. Joseph Marin. AF 33(616)-2729. June 1959. ASTIA Document No. AD 245210.

The creep deformation of a rivet subjected to transverse shear depends, among other factors, upon the rivet diameter and rivet length. This influence of size means that each particular rivet configuration must be tested to obtain the transverse shear-creep deformation. A more basic approach would be to predict the transverse shear-creep of a rivet in terms of the more fundamental case of pure shear as represented by torsion. This report develops a theory for predicting the transverse shear-creep deformation of a rivet in terms of pure torsional shear-creep. The correlation of transverse shear and pure torsion shear is made based on both the torsion of a solid circular bar and the torsion of a thin-walled circular tube. From the theory developed, it is also possible to obtain the creep deflection of a rivet from the creep constants in pure shear or simple tension.

Pennsylvania State Univ. WADC TR 57-757
CREEP DEFORMATION IN A SINGLE RIVETED STRUCTURAL JOINT UNDER AXIAL TENSION. Joseph Marin. AF 33(616)-2729. June 1959. ASTIA Document AD 216712. PB 161036. Order from OTS $0.50.

A review and study of the literature (1 to 12) shows that efforts to determine the creep of riveted joints have been confined primarily to experimental investigations on the overall creep in various types of riveted joints. Little attention has been given to the theoretical prediction of the creep deformation of a riveted joint from the basic simple tension creep strain-stress relation for the material. It is the purpose of this report to outline a procedure for theoretically predicting the creep deformation of a simple riveted joint. This procedure attempts to consider the creep of the component parts in a riveted joint.

Univ. of Minnesota. WADC TR 59-96.
DAMPING ENERGY DISSIPATION AT SUPPORT INTERFACES OF SQUARE PLATES. T. J. Mentel, C. C. Fu. AF 33(616)-5426. June 1959. ASTIA Document No. AD 216530. PB 151972. Order from OTS $0.75.

The energy dissipation due to viscous shear forces between support interfaces of built-in square plates is obtained for simple harmonic transverse vibration of the plates. A comparison is made with the energy dissipation due to material damping within the plates and it is shown that the interface damping mechanism can have an overriding effect for thin plates. A preliminary design curve is presented which allows rapid evaluation of structural and material parameters which will maximize interface damping.

Univ. of Minnesota. WADC TR 58-568.
THE EFFECT OF CONFIGURATIONAL ADDITIONS USING VISCO-ELASTIC INTERFACES ON THE DAMPING OF A CANTILEVER BEAM. James S. Whittier. AF 33 (616)-5449. June 1959. ASTIA Document No. AD 214381. PB 151883. Order from OTS $1.75.

An analysis of the damping of a cantilever beam by an added configuration involving viscoelastic interfaces is presented. The damping is obtained after first calculating the response of the system to low frequency cycling under constant sinusoidal force amplitude. Calculations are made for optimum values of damping due to viscoelastic interfaces. These optimum values are compared with calculated values of damping due to hysteresis of the structural materials of a plain cantilever beam.

Results of an experimental check of the configurational damping theory are presented. The experimental data show fair agreement with the predicted values both as to order of magnitude and trends toward optimum values. The limitations of the theory and reasons for discrepancies between theory and experiment are discussed. Possible extensions and refinements of the theory are also discussed.

Bolt, Beranek and Newman, Inc. WADC TR 58-577.
PRELIMINARY SURVEY OF HIGH-SPEED IMPACT INFORMATION. Peter A. Franken. AF 33(616)-5730. June 1959. ASTIA Document No. AD 216029. PB 151947. Order from OTS $0.75.

This report summarizes studies of high-speed impact now under way. Experimental facilities for obtaining high velocities are considered, and advantages and disadvantages associated with the various propulsion systems are discussed. A separate classified table lists capabilities of existing propulsion systems. Methods of analyzing high-speed impact data are reviewed.

Pennsylvania State Univ. WADC TR 57-756.
STRESS DISTRIBUTION IN A PLATE WITH A HOLE SUBJECTED TO AN AXIAL LOAD AND CREEP. Joseph Marin. AF 33(616)-2729. June 1959.

In evaluating the creep in a riveted joint, the creep of the component parts of the joint must be considered. That is, the creep of a riveted joint is made up

of the creep in the plates plus the creep of the rivets. Since the creep of the plates is influenced by the presence of holes in the plates, it was considered necessary to determine the creep deformations in a plate with a hole when subjected to axial tension (Fig 1). In order to obtain these creep deformations, it is necessary first to determine the creep stresses in an axially loaded plate with a hole. This report gives an approximate analysis of these stresses.

Royal Institute of Technology. WADC TR 59-78, Part I.
MEASURING ACCURACY IN CREEP TESTS PART I. Influence of Eccentricity of Load. Arne Mellgren. AF 61(052)-05. July 1959. ASTIA Document No. AD 216357. PB 151928. Order from OTS $0.75.

In the course of an ordinary uniaxial creep test, several sources of error may be operative, resulting in test data that are not representative of the real properties of the test material. A primary source for error is loading eccentricity which is the subject of Part I of this report series.

In this report, the author determines the influence that each of several types of loading eccentricities has upon the observed secondary creep rate. Consideration is given to non-uniform creep due to eccentrically applied loads which includes initial and time dependent eccentricity, curved test specimens, and asymmetric location of the extensometer. Non-uniform creep due to inhomogeneous materials and non-uniform temperature distribution across the specimen cross section causing eccentric loading, is also presented. The error introduced by each is formulated and discussed.

Univ. of Minnesota. WADC TR 59-121.
STEADY STATE RESPONSE OF A SIMPLE SYSTEM WITH A HYSTERETIC SPRING. Edward R. Rang. AF 33(616)-5426. July 1959. ASTIA Document No. AD 227099. PB 161091. Order from OTS $0.75.

The response of a simple spring-mass system is studied. The spring characteristics are chosen to simulate hysteresis loops found for systems in which the damping is due to the energy dissipation of materials or fabricated joints. Procedures for approximate calculation are outlined and an example is given.

Univ. of Minnesota. WADC TR 59-141.
STEADY STATE UNDAMPED VIBRATIONS OF A CLASS OF NONLINEAR DISCRETE SYSTEMS. P. R. Sethna, B. E. Fristedt, V. G. Harvester. AF 33(616)-5449. August 1959. ASTIA Document No. AD 228512. PB 161129. Order from OTS $1.00.

Steady state vibrations of a class of nonlinear discrete systems with an arbitrary number of degrees of freedom are studied. The coordinates of the system are first transformed to the principal coordinates corresponding to the linear part of the system. A perturbation scheme is used to obtain the solutions. Some special effects of the ratios of the linear natural frequencies on the qualitative nature of the solutions are demonstrated. Solutions are obtained for some specific problems and the results are checked against those obtained from an analog type computer.

Curtiss-Wright Corp. WADC TR 59-227.
INVESTIGATION OF FATIGUE PROPERTIES AT ROOM TEMPERATURE OF HIGH STRENGTH STEELS HAVING HIGH TEMPERING TEMPERATURES. Harold N. Cummings, Foster B. Stulen, William C. Schulte. AF 33(616)-5182. September 1959. ASTIA Document No. AD 228517. PB 161144. Order from OTS $2.50.

High strength steels exhibiting some promise of good fatigue strength at temperatures up to about 1000°F were investigated. The tests reported herein were made at room temperature, as preliminary to later testing, under temperature, of the steels studied or modifications of them as suggested by the test results. Steels for (1) general structural use, (2) use for springs, (3) use for the surface of space vehicles, and (4) use for bearings were investigated. Possibilities of improved fatigue performance were indicated provided the inclusion content could be lowered and the inclusion size restricted to below half a thousandth of an inch.

Curtiss-Wright Corp. WADC TR 59-230.
QUALITATIVE ASPECTS OF FATIGUE OF MATERIALS. Harold N. Cummings. AF 33(616)-5182. September 1959. PB 161145. Order from OTS $4.00.

This report discusses qualitatively the variables that affect the fatigue life and strength of structural metals. Although the table of contents purports to list variables separately, the text reveals the fact that many variables are themselves functions of other variables. This requires that extra-polation, from effects discussed under any set of circumstances to those that might occur under different circumstances, be done with extreme caution.

Some of the theories of the mechanism of fatigue are discussed briefly at the end of the report.

Midwest Research Institute. WADC TR 59-192.
RESEARCH ON THE MECHANISMS OF FATIGUE. J. C. Grosskreutz, Fred R. Rollins. AF 33(616)-5065. September 1959. PB 161412. Order from OTS $2.00.

The microphysical changes accompanying cyclic stress have been investigated in copper and aluminum samples by the use of X-ray and electron microscope techniques. Small angle X-ray scattering reveals that the formation of misoriented subgrains is the most pronounced structural change and this formation is essentially completed within the first 10 per cent of the fatigue life. It is further demonstrated that high temperature annealing does not remove this polygonized structure, but does promote some growth and consolidation of the subgrains. The effect of annealing on fatigue life is discussed in the light of these results.

No evidence has been found for the formation of voids in the volume of copper and aluminum during cyclic stressing. This result strongly favors the hypothesis that all cracks originate at the surface of a fatigue sample. Electron micrographs have shown the existence of deep intrusion which are possible sources of fatigue cracks on the surface of copper samples run to fatigue. Apparatus for the study of atmospheric effects on crack initiation has been built and experiments initiated on copper and aluminum samples.

Energy dissipation during fatigue has been measured and found to follow the classic stages of fatigue: initial hardening, a long quiescent period in which dissipation gradually increases, and onset of fracture. Reasons are discussed for discarding this description of fatigue in favor of a simple picture of crack initiation and crack propagation.

A comprehensive state-of-the-art survey has been made and attached as an Appendix.

Bockamollan, Brosarps Station, Sweden.
WADC TR 59-400, Part I.
STATISTICAL EVALUATION OF DATA FROM FATIGUE AND CREEP-RUPTURE TESTS, Part I. Fundamental

Concepts and General Methods. Waloddi Weibull. AF 61(514)-1208. September 1959. PB 161297. Order from OTS $2.00.

Fatigue tests are classified into three types with consideration to the appropriate method of evaluating the data. Fundamental statistical concepts, general methods, and useful tools are presented.

The possibilities of estimating distribution parameters have been examined. Starting from the concept of information available in a sample, various methods of estimating the parameters of location, scale, and shape are discussed, completed by a comparison of the efficiency of various estimates.

General principles of fitting curves to observations are outlined and applied to the methods of maximum likelihood, linear regression, and best linear estimators.

Royal Institute of Technology. WADC TR 59-78, Part II.

MEASURING ACCURACY IN CREEP TESTS PART II. Influence of Thermal Stresses. Arne Mellgren. AF 61 (052)-05. October 1959. PB 161356. Order from OTS $1.50.

In an ordinary, uniaxial, elevated temperature creep test inaccuracy arises from various sources. One such source of inaccuracy is eccentricity of loading which has been reported in WADC TR 59-78. Another source, presented herein, is thermal stress considered in conjunction with its influence upon the creep rate.

In this report, the author calculates the thermal stresses induced in cylinders and plates by non-uniform surface temperatures. The relations of thermal stresses to creep are then determined for cylindrical and sheet creep specimens. These relations are expressed mathematically for sinusoidal and square wave surface temperature gradients. Numerical examples are presented and discussed.

Univ. of Minnesota. WADC TR 59-76.

BEAM VIBRATIONS WITH QUASI-ORTHOGONAL BOUNDARY CONDITIONS. Y. C. Das, L. E. Goodman, A. R. Robinson. AF 33(616)-5426. November 1959. PB 161348. Order from OTS $1.25.

One of the ways in which the development of destructive structural vibrations may be prevented is the use of energy-absorbing material at the boundary of the panel or stringer. Rational estimation of the benefits to be expected from such damping devices has been hindered by the absence of a suitable analytical method for predicting the dynamic response of structures with energy-absorbing boundary conditions. Under these circumstances the characteristic functions which define the mode shapes are not orthogonal and the conventional analytical tools are ineffective. The present report describes a new analytical technique which overcomes these difficulties. The case of a cantilevered beam has been used as a vehicle for conveying essential ideas but the method, of course, is not limited to beams.

The technique described for the analysis of linear structures subject to linear (velocity) damping at boundaries is an extension of the work of Bulgakov which has been paralleled independently by that of Foss. The unusual feature of this method is that the displacement and velocity are considered as two independent functions and the kinematic relation between them is not immediately imposed. Quasi-orthogonality relations which are obtained for any set of two modes permit expansion of any sufficiently smooth function in a series of eigenfunctions. This fact makes it possible to complete the solution.

Bolt Beranek and Newman, Inc.
WADC TR 59-509.

DAMPING OF FLEXURAL VIBRATIONS BY ALTERNATE VISCO-ELASTIC AND ELASTIC LAYERS. Eric E. Ungar, Donald Ross, Edward M. Kerwin, Jr. AF 33(616)-5426. November 1959. PB 161425. Order from OTS $1.25.

Previous work dealing with the damping of flexural vibrations by application of single "damping tapes" consisting of metal foils and dissipative adhesives is summarized and extended to multiple tapes. A general analysis of damping due to N equal tapes is presented; the effect of using non-equal tapes is investigated for double tape applications. Suitable dimensionless parameters are used where possible in order to maintain generality.

It is shown that additional tapes provide a considerable increase in damping at low frequencies, but only a very small increase at high frequencies. It is found that multiple tapes and single tapes incorporating an equivalent amount of metal provide nearly the same damping, a fact which results in great design flexibility.

Experimental and theoretically predicted results are shown to be in reasonably good agreement.

Univ. of Michigan Research Institute.
WADC TR 59-454.

EFFECT OF PRIOR CREEP ON THE MECHANICAL PROPERTIES OF A HIGH-STRENGTH HEAT-TREATABLE TITANIUM ALLOY: Ti-16V-2.5Al. Jeremy V. Gluck, James W. Freeman. AF 33(616)-3368. November 1959. PB 161490. Order from OTS $2.00.

A study was carried out of the effect of exposure to elevated temperature creep conditions on the short-time mechanical properties of a high-strength, heat-treatable titanium alloy, Ti-16V-2.5Al. Exposures were conducted for 10 or 100 hours either unstressed or at stresses causing up to 2 percent creep deformation at temperatures between 600° and 900°F. The specimens were taken parallel to the sheet rolling direction.

Following the exposures, short-time tension, compression or tension-impact tests were run at room temperature or the temperature of exposure. Prior creep at 600°F raised the ultimate tensile strength and tensile yield strength considerably and the compressive yield strength and tensile elongation were substantially decreased. Exposure to temperature alone caused increases in strength indicative of an age-strengthening reaction. The changes in mechanical properties are attributed mainly to a combination of stress-accelerated age-strengthening and a Bauschinger effect. A lesser change in properties was noted for creep exposures conducted at 700°F. Peak properties from the age-strengthening reaction were noted in the unstressed exposures conducted at 800°F and over-aging with a consequent drop-off in strength was obtained from the 900°F creep-exposures. Metallographic evidence tended to confirm the presence of stress-accelerated aging.

New York Univ. WADC TR 59-416.

INVESTIGATION OF CREEP BUCKLING OF COLUMNS AND PLATES. Ralph Papirno, George Gerard. AF 33(616)-4807. November 1959. PB 161488. Order from OTS $1.00.

As a first phase of an investigation of the Creep Buckling of Columns and Plates, materials properties tests were conducted on the sponsor chosen test material Ti-7Al-4Mo Titanium Alloy in both "as received annealed" and test treated conditions. Bulk specimen material was heat treated according to the following schedule to obtain optimum creep performance: 1450°F-1 hr., furnace cool to 1050°F, air cool. This phase of the investigation was necessary because of the paucity of published materials

property data on this alloy, especially in compression.

A new technique for the collection of compression creep data from sheet specimens was developed in which both the short time stress-strain properties prior to creep and the creep data itself are autographically recorded.

The test results include values of the modulus of elasticity and 0.2 percent yield strength in both tension and compression at room temperature, 750°F, 850°F, and 950°F. Compressive creep data were collected for a number of stress levels at 850°F and 950°F.

Metals Research Laboratory, Brown Univ. WADC TR 59-389.
THE USE OF ULTRASONIC METHODS TO DETERMINE FATIGUE EFFECTS IN METALS. John Truell, Bruce Chick, Amos Picker and George Anderson. AF 33(616)-5884. November 1959. ASTIA Document No. AD 226098. PB 161487. Order from OTS $0.75.

This report discusses observed changes in ultrasonic attenuation and velocity in commercially pure 1100 aluminum (Alcoa) when subjected to slow continued stress cycling (1 to 6000 cycles) and a delayed recovery phenomenon that appears when the cycling is interrupted. In addition, there is a description of an instrument to measure and record ultrasonic attenuation changes.

Univ. of California. WADC TR 59-131.
THE EFFECT OF HIGH TEMPERATURE RECOVERY ON THE CREEP OF POLYCRYSTALLINE ALUMINUM IN THE DISLOCATION CLIMB REGION OF TEMPERATURES. AF 33(616)-3860. December 1959. PB 161422. Order from OTS $1.00.

Recovery of the creep resistance of 99.99% pure aluminum was studied at temperatures of 540°, 573°, 600°, and 611°K. Polycrystalline specimens crept under a stress of 950 psi to a strain of 5.5% were allowed to recover for periods of from one minute to sixteen days under a residual stress of 4.4 psi. Increased creep rates upon reapplication of the 950 psi stress evidenced softening of the material.

The activation energy for the recovery process was found to be 64,000 cal/mole. Metallographic studies of the specimens showed relief of local elastic strains by coalescence of dislocations into subgrain boundaries upon recovery, and sub-boundary migration. Indications of minor grain growth were also found. The activation energy of 64,000 cal/mole indicates that recovery of creep resistance was due primarily to the sweeping out of dislocations within the grains by the migration of subboundaries.

WADC TR 59-433.
RAPID LOADING OF ALUMINUM ALLOY RIVETED JOINTS. Lt. R. T. Ault. December 1959. PB 161434. Order from OTS $0.75.

Slow and rapid loading tensile shear tests were conducted at room temperature to determine the failure strength of multiple riveted lap and butt joints. Both concentric and eccentric loading conditions were used. The time to failure for the rapid loading tests ranged from 0.02 to 0.08 seconds. All failures were by rivet shear.

The results indicate that for both eccentric and concentric loading conditions, rapid loading has no significant effect on the strength of the joint.

Midwest Research Institute. WADC TR 59-561.
STUDY OF METHODS FOR NONDESTRUCTIVE MEASUREMENT OF RESIDUAL STRESS. Fred R. Rollins. AF 33(616)-5932. December 1959. PB 161436. Order from OTS $1.25.

A rather thorough search for new techniques of measuring residual stresses has been conducted. This search has included a study of various physical phenomena which exhibit some stress dependent relationship. Of the many phenomena studied, ultrasonic was chosen for further experimental investigation. It has been definitely established that residual stresses in metals can cause double refraction of a polarized shear wave. A technique based on the double refraction of shear waves, for measuring the average stress through certain specimens is described and sources of error are discussed. The interaction of two ultrasonic waves in metals has been investigated also.

Kaiser Aluminum & Chemical Corp. & Materials Laboratory. WADC TR 59-591.
FATIGUE BEHAVIOR OF 2014-T6, 7075-T6 and 7079-T6 ALUMINUM ALLOY REGULAR HAND FORGINGS. D. A. Paul, D. Y. Wang. January 1960. PB 161500. Order from OTS $1.00.

This report presents the test procedures and results of a fatigue investigation on regular hand forgings of the aluminum alloys 2014-T6, 7075-T6 and 7079-T6. The effects of forging direction on fatigue strength are investigated in the unnotched and notched condition.

The unnotched fatigue strength of the three alloys ranged from 20,000 to 25,000 psi at 2×10^7 cycles, with the 7079-T6 alloy being slightly lower than 7075-T6 and about the same as that of the 2014-T6 alloy.

The fatigue strength in the short transverse direction is consistently lower than in the longitudinal direction; however, the difference becomes less in the notched condition.

WADD TR 59-26.
AN INVESTIGATION OF THE RELATIONSHIP OF HOT-HARDNESS TO THE ELEVATED TEMPERATURE EXTRUSION BEHAVIOR OF SELECTED ARC-CAST MOLYBDENUM BASE ALLOYS. Lt. John R. Giancola. February 1960. AD 208665. PB 151958. Order from OTS $1.25.

A study was conducted to determine the relationship between the hot hardness and the required extrusion forces and percent yield of sound material for molybdenum and molybdenum base alloys. Un-alloyed molybdenum, molybdenum - Ti, and Mo-Ti-Zr billets were sectioned, examined, weighed and extruded at different temperatures from 1000° to 2900°F. The extrusion force was measured while other controllable variables were held constant. The extruded billet was examined visually, machined, weighed, and prepared for metallographic analysis. The extrusion dies were also prepared for metallographic examination.

It was found that no definite relationship between hot hardness and the extrusion forces, either maximum or average, could be established. Also, it was impossible to establish a relationship between hot hardness and the yield of sound metal. However, it was possible to relate both the average extrusion force and the percent yield to the extrusion temperature. The extrusion force decreased with increased temperature while the yield increased to maximum and then decreased as the temperature increased. The latter relationship indicates a possibility of hot shortness in the molybdenum alloys.

Although a number of variables in the extrusion process were controlled, certain factors are not always constant. Among these are the hardness of strain hardening accomplished. These variables could make it impossible to establish any definite relationship between hot

hardness and the extrudability of molybdenum and molybdenum alloys.

Columbia Univ. WADC TR 59-399.
CREEP AND PLASTIC DEFORMATION IN CYLINDRICAL SHELLS. Alfred M. Freudenthal, Maciej P. Bieniek. AF 33(616)-6112. March 1960. PB 161706. Order from OTS $0.75.

In the first part of this report, the problem of creep deformation and bending moments in the vicinity of a freely supported or fixed end of a long cylindrical shell is solved under conditions of stationary (second stage) creep, using an approximate method based on extremum principles of dissipative media which are valid for the assumed uni-axial and non-linear creep law $\dot{\varepsilon} = K\sigma^n$. The results of analysis are checked by experiment.

In the second part of this report, results of tests of steel cylinders under internal pressure, producing large plastic deformation are given, illustrating the relation between problems of non-linear creep and plastic deformation.

Univ. of Minnesota. WADC TR 59-544.
DAMPING OF RECTANGULAR PLATE VIBRATIONS. T. S. Lundgren, C. C. Chang, Y. C. Whang. AF 33 (616)-5426. March 1960. PB 161755. Order from OTS $0.75.

In Part A, an analysis is made of the effect of an impact type damping mechanism on a vibrating square plate. The force which the damping mechanism exerts on the plate is idealized as a series of impulses acting at the center of the plate. It is found that the device analyzed does not make a very effective damper.

In Part B, an analysis is made of the effect of a damping device on the vibrations of a rectangular plate. The damper considered is a free piston in a closed cylinder which is attached to the center of the plate. When the leakage through the gap between the piston and the cylinder is small, the piston is caused to oscillate on the "spring" of air in the cylinder. It is found that this device can effectively damp out the resonant mode of the plate vibration.

WADC TN 59-340.
ELASTIC LIMIT BY THE RESIDUAL STRAIN METHOD USING SR 4 AND TUCKERMAN GAGES ON ALUMINUM ALLOY. Alton W. Brisbane, R. F. Klinger. March 1960.

The "elastic limit" of 7075-T6 aluminum alloy was defined in terms of SR 4 and Tuckerman gage indications through the use of residual microstrains following tensile and compressive loading and unloading. Residual strain vs. applied load relationships were developed as functions of reference stress level and material and gage history. Some qualitative theoretical and experimental explanation of residual microstrain indications were attempted.

The results indicate there is considerable interaction between SR4 gage characteristics and specimen material properties in the region of residual microstrain considered and that a better understanding of the strain indications should be obtained before they are used to explain microdeformations of metals.

Univ. of Michigan Research Institute.
WADC TR 59-470.
NOTCH SENSITIVITY OF HIGH-TEMPERATURE ALLOYS. Howard R. Voorhees, James W. Freeman. AF 33(616)-5775. March 1960. PB 161796. Order from OTS $1.50.

Critical examination of all available results on creep-rupture of notched specimens disclosed: (1) notch strengthening for all alloys under proper conditions, (2) maximum strengthening for intermediate notch acuities, (3) two general patterns of stress-rupture time data relative to the smooth-bar curve.

Analysis of observed behavior suggests notch strengthening requires stress redistribution by yielding and creep, and is associated with the multi-axial stress pattern produced by the notch.

Both maximum principal stress and the shear-stress invariant are hypothesized to influence rupture of notched specimens through their respective effects on crack initiation and propagation.

Research is proposed on crack development during creep-rupture in notched tension bars and in biaxial plate specimens, to verify the suggested explanation for notch behavior.

Univ. of Minnesota. WADC TR 59-543.
STEADY STATE DAMPED VIBRATIONS AND STABILITY OF A CLASS OF NONLINEAR DISCRETE SYSTEMS. S. T. Chow, P. R. Sethna. AF 33(616)-5426. March 1960. PB 161748. Order from OTS $1.50.

A class of nonlinear discrete systems with an arbitrary number of degrees of freedom are studied for their steady state vibrations. The coordinates are first transformed to the principal coordinates corresponding to the linear part of the system. An iteration scheme is used to obtain the desired solution. Some special effects of the relations between the linear natural frequencies on the qualitative nature of the solutions are demonstrated. It is shown that if the linear natural frequencies do not possess certain relations, the system can be treated in a manner similar to that for a system of a single degree of freedom. In other cases the procedure gets more complicated. The various solutions are then examined for their stability. Poincare's theory of singularities in the phase plane is used to study the stability of those problems that can be treated in a manner similar to that for a single degree of freedom system. In all other cases the stability is examined by applying the Routh-Hurwitz criterion to a transformed set of equations. Solution for one specific problem is obtained and checked against those obtained from an analog type computer.

American Machine & Foundry Co.
WADC TR 59-762, Part I.
ULTRA-SHORT-TIME CREEP RUPTURE EQUIPMENT MANUAL. Joseph S. Ives, Jr. AF 33(616)-5557. April 1960. ASTIA Document No. AD 237830. PB 161789. Order from OTS $1.50.

The purpose of this program was to develop a method of heating a sample of sheet metal to a pre-determined temperature in a short period of time and to measure the creep characteristics of this mateiral at the developed temperature.

Described in this report are the special equipments designed to carry out the work together with the necessary operating instructions. Also included are the drawings required to produce the equipment at the contractor's facilities.

Magnetic Materials

Wyandotte Chemicals Corp. WADC TR 56-495.
DEVELOPMENT OF NON-TOXIC ORGANIC SOLVENTS OR SOLVENT SYSTEMS FOR GENERAL CLEANING APPLICATIONS. Kenneth K. Aoki, Arthur J. LaRue, Brock G.

Peacock. AF 33(616)-2775. July 1957. ASTIA Document No. AD 130888.

A search has been made for new solvents or solvent combinations which could satisfactorily replace carbon tetrachloride in general cleaning applications. Inhibited methychloroform and combinations of methylchloroform with up to 15 volume percent of Freon 112 were formulated. Freon 112 is used to reduce the evaporation rate of methylchloroform which is slightly higher than that of carbon tetrachloride. Although the corrosion inhibition properties of the formulated mixtures cannot be considered satisfactory, the target specifications appear to have been met in respect to minimum flash point, decrease in flash point during evaporation and toxicity. The solvents are essentially equal to or better than carbon tetrachloride in respect to deleterious action on various electrical insulators, plastic laminates, and organic coatings. The recommended solvents can be economically produced in the required volume. Although the present limited production of Freon 112 and its consequent high cost may limit the proportion which can be used in the solvent combination, there is reason to anticipate materially reduced costs under volume production. Areas in which continuing development work is needed are the further refinement and proving of several possible inhibitor systems for optimum results, long term storage and stability tests under anticipated storage conditions, practical application tests, and final clearance in respect to toxicity by Air Force medical personnel.

WADC TR 53-147 Suppl. 1.
NON TOXIC SOLVENTS FOR CLEANING AIRCRAFT ENGINES AT EXTREMELY LOW TEMPERATURES. Robert E. Cretcher. September 1957. ASTIA Document No. AD 131072.

Two reciprocating aircraft engine degreasing formulae consisting essentially of methylene chloride and aliphatic petroleum hydrocarbon, suitably inhibited, had been recommended on the basis of laboratory evaluations as being suitable for use under arctic conditions. Service tests were conducted by three operational activities of the Alaskan Air Command during the winter seasons of 1954-1955, and 1955-1956. The results of the operational usage of the two degreaser formulae and a control solvent indicate generally that either of the test formulae would be suitable for the purpose at temperatures above -20°F. One of the test materials evaporates more slowly than the other but may be used as both cleaning and rinse solvent. While the second material evaporates more rapidly, a straight petroleum solvent is required to rinse the cleaning solvent from the treated surfaces. Test results are discussed and the proposed specification requirements for the recommended degreaser are presented.

Denver Research Institute. WADC TR 57-606.
ORGANIC COMPOUNDS OF GALLIUM - A SUMMARY OF THE LITERATURE. Andrew J. Frank, Richard W. Sullivan, Virgil W. Lichtenberg. AF 33(616)-2939. November 1957. ASTIA Document No. AD 142158. PB 131625.

The literature concerning organic compounds containing gallium has been surveyed, and the properties of all organo-metallic and organic coordination compounds of gallium are summarized. An annotated bibliography of all references is provided.

Foster D. Snell, Inc. WADC TR 57-187.
LOW TEMPERATURE AIRCRAFT SURFACE CLEANERS. Elias Kimmel, Bernard Berkeley, Daniel Schoenholz. AF 33(600)-32244. March 1958. ASTIA Document No. AD 151073.

Physical and chemical characteristics of different types of soil normally encountered on aircraft surfaces were studied. Laboratory soil removal tests were developed and used to evaluate all important classes of organic solvents. Seventeen different amine oleates were examined for their ability to form dilute water-in-naphtha emulsions. A combination of diethylamine oleate and triethanolamine oleate allowed the incorporation of 1.5 parts of water in naphtha and naphtha-chlorinated solvent blends. Emulsifier systems were compounded to render hydrocarbon and hydrocarbon-chlorinated solvent blends readily rinsable with diluted deicing fluids. Field tests at Loring A. F. B. and Mitchell A. F. B. established the efficacy of water-containing and self-emulsifiable cleaners. Two cleaners conforming to all target requirements are recommended for removal of oily, and traffic film, type soils from aircraft surfaces at temperatures of 0° to 32°F. One cleaner is used in conjunction with a naphtha rinse; the other is flushed with either naphtha or a glycol-alcohol-water fluid. Raw material costs, methods of preparation, sources of supplies and ingredient specifications are reported for both recommended cleaners.

Pennsalt Chemicals Corp. WADC TR 57-193.
DEVELOPMENT OF NON-CRESYLIC CARBON REMOVERS. Rubin Weiner, J. Walter Carroll. AF 33(600)31890. March 1958. ASTIA Document No. AD 151022.

The usual carbon remover formulations now in use contain chlorinated hydrocarbons, cresols, and wetting agents. As such they present a serious toxicity and waste disposal problem and are therefore unsuitable for field use where only limited facilities are available. Major Air Force overhaul depots are successfully removing engine varnish (polymerized oils) and loosening carbon deposits from reciprocating engine parts with this material. However in this laboratory, they have been found unsatisfactory for removing carbon deposits from either jet engine or reciprocating engine parts.

Two formulations, which this laboratory developed, appear to be much more effective in removing carbon deposits than the present specification compositions. Laboratory tests indicate that these two formulations are satisfactory with respect to corrosivity and toxicity, present no serious waste disposal problems, and are not expensive to manufacture.

*Ohio State Univ., Columbus
BEHAVIOR OF SOLIDS IN ALTERNATING AND CONSTANT MAGNETIC FIELDS. J. Korringa. Project 9760(802), Contract AF 49(638)-264; AFOSR, DSS.

This research is directed toward the behavior of solids in alternating and constant magnetic fields and is specifically concerned with: (1) the development of mathematical expressions for the magnetization and aero-field energy of paramagnetic substances as influenced by interactions between the magnetic ions; (2) the theory of paramagnetic relaxation in crystals, in particular the influence of dipole and exchange interaction of the magnetic ions at low temperature; (3) investigation of the electron band theory of alloys and its implications for their magnetic behavior; and (4) the influence of lattice deformations

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

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induced by nearest neighbor interaction on the thermodynamic properties of spin systems. The orientation of metallic nuclei by saturation of the electron resonance in a magnetic field, predicted by Overhauser, has its counterpart in paramagnetic substances. In the case of metals, the essential points are: (1) one of the processes contributing to the electron relaxation is the coupling with the nucleus, which conserves the total spin vector; (2) this coupling is the predominant one in the nuclear relaxation; (3) the energy in such a process can be conserved because the states of the conduction electrons form a continuum; (4) in the saturated situation, the Pauli principle provides electron transitions with decrease of magnetic energy with a larger a priori probability than the inverse transitions.

*Massachusetts Inst. of Tech., Cambridge.
ELECTRIC AND MAGNETIC PROPERTIES OF MATTER. A von Hippel. Project 4571(803), Contract Nonr 1841(10); AFOSR, DPS.

The Joint Services program at the Laboratory for Insulation Research covers broadly the field of molecular science and engineering and covers areas such as: molecular electronics; dielectric spectroscopy; excitation, conduction, and breakdown; ferroelectrics and ferromagnetics; and single crystal and ceramics research.

*Harvard Univ., Cambridge, Mass.
FUNDAMENTAL PHYSICS OF FERROMAGNETIC AND FERROELECTRIC MATERIALS. R. V. Jones. Project 5633(803), Contract AF 19(604)-5487; ERD, ERL.

The dependence of ferrimagnetic properties on high hydrostatic pressure (up to 12000 atmospheres) due to changes in atomic spacing in the crystal lattice is being investigated. Low temperature measurements of the specific heat of magnetic systems are being made to obtain information on the energy level structures and lattice properties of these materials. New barium ferrite magnetic systems are being investigated. Characterized by high anisotropy fields, these materials have high potential for microwave applications due to the reduction in external magnet requirements. Studies of other new magnetic systems which may conceivably be both magnetic and either piezoelectric or ferroelectric are commencing. Theoretical and experimental investigations of the ultimate reversal time in ferrite core switching are also being carried on.

*Michigan Univ., Ann Arbor.
MAGNETIC AND ELECTRIC PHENOMENA IN FERRIMAGNETIC MATERIAL. D. M. Grimes. Project 9760(802), Contract AF 49(638)-779; AFOSR, DSS.

The objective of this research is to obtain a better understanding of the source of the magnetic and electric properties of ferrimagnetic materials and to determine possible means of controlling them. The approach is two-fold. First, it is necessary to understand how the solid material reacts to arrive at its final form, which involves the use and the understanding of reactions between solids. In this endeavor use will be made of the techniques of differential thermal analysis, optical and electron microscopy, X-ray analysis, density and heat-capacity determinations as appropriate. Secondly, it is necessary to understand the electric and magnetic properties of a given material. For this purpose measurements will be made of the electric and magnetic properties as a function of biasing magnetic field, the magnetization, and the temperature. The spontaneous magnetic moment and magnetic anisotropy will be measured from helium to room temperature.

*Massachusetts Inst. of Tech., Cambridge.
MAGNETIC LOSS PHENOMENA IN FERROMAGNETIC MATERIALS. D. J. Epstein. Project 5620(802), Contract AF 19(604)-5482; ERD, EML.

Studies of the magnetic switching of polycrystalline samples of yttrium iron garnet at low temperature indicate that two distinct switching modes are present with time constant of several minutes and within the order of milliseconds respectively. Hysteresis loops were measured at liquid-nitrogen temperatures over a range of peak fields and at different frequencies. No clear cut analysis of the results of the experiments has yet been made.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
MAGNETIC MATERIAL MEASUREMENTS, W. G. Field. Project 5621(802), Internal.

A new method was developed for the determination of ferrimagnetic resonance utilizing a special pick-up loop placed in a standard wave guide, and led to the discovery of a new type thermal instability in rare earth-iron garnet materials. The measurement and evaluation of paramagnetic materials has improved due to the development of an improved electron paramagnetic resonance spectrometer. The resolution of the instrument has been increased and the data is now automatically recorded. Very small spherical samples are required for use in the ferrimagnetic resonance spectrometer. The technique for the preparation of these samples has been improved. The facilities for other magnetic and X-ray diffraction measurements have also been improved during the past year. These improvements have in general been limited to increasing the ease of operation and the accuracy of data.

*Franklin Inst., Philadelphia, Pa.
MAGNETIC PROPERTIES OF FINE PARTICLES OF MAGNETIC MATERIALS. A. E. Berkowitz. Project 7021(802), Contract AF 33(616)-6922; ARL, MCB.

The major objective is to observe the principal features and differences in a large number of fine particles in order to develop a comprehensive method for analyzing the properties of any type of fine powder. Emphasis will be placed on providing reliable data on, and rigorous analysis of the size dependence of the magnetic properties of fine particles in the superparamagnetic single domain and multidomain size ranges. The study of particles of narrow size distribution will be stressed. X-ray diffraction measurements will be used to determine composition, structure and line broadening due to size effects. If apparatus is available, some measurements of magnetic characteristics will be made at liquid helium temperatures. Techniques will be developed for production of fine particles of additional metals, alloys, ferrites, garnets and other oxides. The general structural and magnetic properties of these materials will be investigated.

*Minnesota Univ., Minneapolis.
MAGNETIC RESONANCE IN STRONGLY COUPLED DIPOLE SYSTEMS. A. H. Morrish. Project 9760(802), Contract AF 49(638)-803; AFOSR, DSS.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

The purpose of this research is to investigate the magnetic properties of strongly coupled dipole systems, such as ferrimagnetic garnets by means of magnetic resonance techniques. In particular, ferrimagnetic garnets will be investigated since these materials have some unusual properties. Since the garnets are optically transparent it should be possible to perform experiments such as those involving magneto-optical resonance.

*Franklin Inst., Philadelphia, Pa.
MAGNETIC AND STRUCTURAL PROPERTIES OF PRECIPITATING FERROMAGNETIC SYSTEMS. A. E. Berkowitz. Project 9760(802), Contract AF 49(638)-159; AFOSR, DSS.

The purpose of this investigation is to clarify some of the relationships between various magnetic and structural properties of systems in which ferromagnetic precipitates appear in a non-magnetic matrix. This research is concerned with the kinetics and structural features of the nucleation and growth of precipitates in these systems. This work should result in a more rigorous understanding of the magnetic behavior of systems of ferromagnetic fine particles. In particular, it will contribute to knowledge of the origins of the magnetic properties of permanent magnet alloys such as Alnico V. Since alloys such as Au-Ni, Cu-Co, Beta brass-Fe, and Au-Co are prototypes of the precipitation hardening permanent magnet materials, their study will provide insight into the mechanisms responsible for the superior properties of these systems.

*Washington Univ., Seattle.
NUCLEAR MAGNETIC RELAXATION TIMES. E. A. Uehling. Project 9760(802), Contract AF 49(638)-92; AFOSR, DSS.

Present efforts are directed toward further study of ferroelectric materials with attention being extended to potassium dihydrogen arsenate and to the study of acoustic excitation of nuclear spin transitions in ionic crystals. In this investigation it is desired to learn how acoustic standing wave energy is dissipated as waves of different frequency and as heat. There is need for an intensive study of such problems in view of our lack of understanding of heat conduction processes in solids.

*Oxford Univ., Great Britain.
PARAMAGNETIC AND FERRIMAGNETIC MATERIALS. W. P. Wolf. Project 5620(802), Contract AF 61(052)-125; ERD, EML.

Yttrium-gallium, yttrium-aluminum and yttrium-iron garnet single crystals have been prepared containing small amounts of rare earths. Preliminary resonances have been observed in single crystals of 1% and 0.1% neodymium; 1% and 0.1% dysprosium; 1% and 0.1% erbium in yttrium-gallium garnet and 0.1% erbium and 0.1% dysprosium in yttrium-aluminum garnet at the temperatures of liquid hydrogen and liquid helium. The analysis of some of the observed paramagnetic resonance spectra gives information about the energy states of the rare earths ions in the host lattices.

*St. John's Univ., Jamaica, N. Y.
THEORETICAL STUDIES OF MAGNETIC PROPERTIES OF SOLIDS. H. A. Brown. Project 9760(802), Contract AF 49(638)-861; AFOSR, DSS.

Several features of the magnetic properties of solids will be studied from a theoretical point of view. The series expansion method of evaluating the partition function of a Heisenberg type of ferromagnet will be carried out to the sixth term. The semi-classical model of a ferromagnet has been utilized satisfactorily to calculate the effect of an applied magnet field on the transition temperature. It is proposed to investigate additional predictions of this model such as the magnetic specific heat contribution, the paramagnetic susceptibility, and its application to ferrites, etc. in order to appreciate more fully the applicability and the limitations of the model.

Mechanical Properties of Crystals

*Lessells and Associates, Inc., Boston, Mass.
ACOUSTIC PHENOMENA IN THE STRAINING OF METALS. B. H. Schofield. Project 7021(802), Contract AF 33(616)-5640; ARL, MCB.

An investigation is being conducted to determine the origin and significance of the acoustic vibrations emitted by metals when stressed. Polycrystal and single crystal specimens of pure aluminum are loaded under very precise conditions. High amplification and high speed tape recording techniques are used to record the acoustic signals for study. The emission takes the form of a shaped spectrum with increasing stress. The results indicate the possibility that the source of the emission is related to the generation and movement of dislocations within the aluminum. However, since the emission appears to consist of two primary effects, other factors may also be operative. The study is continuing to isolate and verify these factors.

*Stanford Univ., Calif.
BEHAVIOR OF GRAIN BOUNDARIES IN ALUMINUM BICRYSTALS AT HIGH TEMPERATURE. R. A. Huggins. Project 9761(802), Contract AF 18(603)-66; AFOSR, DSS.

The purpose of this research is to study the effect of solute elements upon the kinematics of grain boundary shear in metals at high temperatures. A number of phenomena occur during the deformation of metals including the movement of individual dislocations, subgrain movement, grain boundary sliding, and boundary migration. Further, the relative magnitudes of these phenomena have been shown to depend upon several variables, including temperature and strain rate. Much of the previous work on grain boundary behavior has been done on polycrystalline materials in which boundary movements are accompanied and complicated by other deformation phenomena. For this reason, the current program is concerned with the effect of solute content on grain boundary shear in simple bicrystals, utilizing high purity aluminum and aluminum alloys.

*Midwest Research Inst., Kansas City, Mo.
BEHAVIOR OF SOLID FILM LUBRICANTS. P. Bryant. Project 7022(802), Contract AF 33(616)-6277; WADD, MC.

The mechanism of friction and wear for single crystals of graphite and for graphite filaments shall be described experimentally and examined theoretically on the basis of knowledge of single crystal structure and the bonding energies of crystals.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Case Inst. of Tech., Cleveland, Ohio.
EFFECT OF INTERSTITIAL ALLOYING ELEMENTS ON THE MECHANICAL BEHAVIOR OF METALS. A. R. Troiano. Project 7021(802), Contract AF 33(616)-6419; ARL, MCB.

A number of critical experiments will be conducted which are designed to fill certain gaps in our existing knowledge of the behavior of interstitials in metals. These experiments include a determination of the possibility that hydrogen will cause strain aging in iron and its alloys, attempts to produce delayed brittle failure and static fatigue in iron alloys under the influence of carbon and/or nitrogen, a determination of the possibility that hydrogen will cause the formation of a tetragonal iron martensite, and a determination of the strain energy involved in the solution of interstitials in a defect lattice. Ultra-high purity metals of different crystal structures will be used with closely controlled interstitial contents. Later work will be concerned with the effect caused by the interaction of several interstitials (other than carbon combined with nitrogen) on the mechanical behavior of a given metal or alloy. As new scientific knowledge is obtained, theory will be developed, evaluated, modified or changed as required.

*Rias, Inc., Baltimore, Md.
EFFECT OF SURFACE FILMS ON THE MECHANICAL PROPERTIES OF METALS. I. R. Kramer. Project 7023(802), Contract AF 33(616)-6220; WADD, MC.

The contractor shall investigate the influence of surface-active agents on the mechanical behavior of single crystals of aluminum, iron, and zinc to provide a basis for comparing the influence of the surface-active agents on different crystallographic systems. The contractor shall use various type films such as thin metal oxides, thin metal films, or liquid surface-active agents to study their effects on the plastic deformation of the single crystals. The methods for investigating the inelastic behavior of the single crystals shall include various strain rates, temperature and X-ray diffraction patterns after various amounts of plastic strain to determine the resolved shear stress, work hardening characteristics and transition temperatures.

*Massachusetts Inst. of Tech., Cambridge.
FACTORS CONTROLLING THE PROPERTIES OF METAL-OXIDE SINTERED COMPACTS. N. J. Grant. Project 7021(802), Contract AF 33(616)-5424; ARL, MCL.

Research is being conducted to determine the fundamental aspects of dispersion strengthening. The systems under study are dispersions of a hard phase in a metallic matrix, prepared by mechanical mixing or by internal oxidation techniques. Supplies of ultra fine powders have been obtained and the experimental techniques have been developed so that particle sizes and dispersions obtained are within the range between those obtained by conventional chemical precipitation methods and by conventional powder metallurgy techniques. The effects of particle size and shape, and interparticle spacing have been studied using dispersion of alumina and silica in a copper matrix. An investigation is now under way to determine the relative contributions of stored energy strengthening and pure dispersion strengthening. The stored energy strengthening will be expressed by variations in mechanical property parameters as a function of amount of recrystallization. The results obtained in this way are expected to be vastly more meaningful than the calorimetric determinations of stored energy, which are difficult to determine accurately and difficult to interpret.

*California Univ., Berkeley.
HEAT CAPACITY OF ALLOYS. R. Hultgren. Project 9760(802), Contract AF 49(638)-83; AFOSR, DSS.

The objectives of this investigation are to determine the heat capacities of various alloy systems, to examine the validity of Kapp's Law of additivity of heat capacities of the components, and to correlate heat capacity data with the nature of bonding forces in metals and alloys. The heat capacity of alloys is an important field of knowledge in which comparatively little work has been done. Knowledge of heat capacities is essential if thermodynamic data, valid only at the temperature of measurement, are to be extended to other temperatures. Thermodynamic data and concepts are being applied to many important fields of study of metals and alloys. Most obviously, knowledge of stability of phases contributes to the establishment of a crystal chemistry of the metallic state. This makes possible predictions of phase diagrams and of phase changes with temperature which are most valuable in designing new alloys. The best theoretical studies of reaction rates in alloys lean heavily on thermodynamic concepts. The dislocation theory, which accounts for the most important property of all, the strength of metals, has progressed to the stage where thermodynamic data are most useful.

*Cambridge Univ., Great Britain.
MECHANICAL PROPERTIES OF METAL CRYSTALS. P. Hirsch. Project 9760(802), Contract AF 61(052)-98; AFOSR, DSS.

The aim of this research is the study of the mechanical properties of metal crystals containing particular distributions of dislocations. Practically no work has yet been done on the possibility of producing metal crystals with particular dislocation distributions. Recent work carried out in Cambridge on the type of dislocations present in deformed metals suggests ways in which crystals could be prepared in a form suitable for mechanical testing. Investigations will be made first on face-centered cubic metals and alloys and then on hexagonal and body-centered cubic metals. In addition similar studies will be made on specimens inrradiated with neutrons and after quenching treatments. Transmission electron microscopy will be employed to study directly the dislocation distribution. These observations will be correlated with measurements on the magnitude, strain rate and temperature dependence of the flow stress. Pure metals as well as metals with controlled amounts of impurities will be studied in both the single and polycrystalline form. The influence of dislocation distribution on the rate of work hardening of such crystals will be studied by flow stress measurements and electron microscopical observations.

*Battelle Memorial Inst., Columbus, Ohio.
MECHANISM OF STRENGTHENING HEAT TREATABLE Fe-Cr-Ni-Al ALLOYS. G. K. Manning. Project 7021 (802), Contract AF 33(616)-6521; ARL, MCB.

The presently unknown mechanism responsible for strengthening precipitation hardenable ferrous alloys will be identified by studies of the reaction kinetics, structure and composition. Quantitative measurements of the volume fraction of the various phases present will be made.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

Should order be detected, the degree and type will be studied as a function of aging time. Composition surveys across relevant areas of the microstructure will be made by means of microchemical analysis and the electron probe microanalyzer. These initial studies will attempt to clearly define the strengthening mechanism in an alloy of the nominal composition of the 17-7PH alloy. Once this existing mechanism is well understood, experimental alloys of high purity will be used to explore the changes occurring during hardening, their relative importance to overall properties, their sensitivity to minor composition variations, and their kinetics.

*Stanford Univ., Calif.
METALLURGICAL VARIABLES AFFECTING THE HIGH TEMPERATURE MODULUS OF ELASTICITY. O. D. Sherby. Project 7021(802), Contract AF 33(616)-6789; ARL, MCB.

Research of the effects of preferred orientation, magnetic state (in case of ferromagnetic alloys), and temperature on the high temperature modulus of elasticity of metals and alloys will be initiated. The Young's modulus of elasticity of metals (Mo, W, Ir, and Os) shall be measured by dynamic means at temperatures up to 1500° - 2000° C. The effects of crystal structure on the modulus shall be investigated, using Zr, Ti, Ca, etc. on the initial investigation. The effects of alloying elements on the high temperature modulus shall also be studied. Creep and dispersion hardness shall be used for correlation.

*Northwestern Univ., Evanston, Ill.
PHASE TRANSFORMATIONS IN SOLIDS. M. E. Fine. Project 9760(802), Contract AF 49(638)-524; AFOSR, DSS.

Three areas of research on phase transformations in solids will be investigated: (A) the effect of introduced crystal imperfections on the kinetics of age-hardening and properties of the final age-hardened state in aluminum base alloys containing Ag, Cu, or Zn will be studied; (B) in Ni base Al and Ni base Ti alloys, the kinetics of precipitation by measuring change in Curie temperature, the nature of precipitates using X-ray diffraction, and the strength and plasticity properties of age-hardened specimens will be determined; (C) using large single crystals of gray tin grown by the Ewald-Tufte method, the motion of the transformation interface when these transform to white tin will be studied, the orientation relations between parent and produce phases will be measured, and an attempt will be made to develop a model for the transformation mechanism.

*California Inst. of Tech., Pasadena.
PHYSICAL PROPERTIES OF METALS AND ALLOYS AT ELEVATED TEMPERATURES. P. Duwez. Project 9760(802), Contract AF 18(600)-1561; AFOSR, DSS.

To overcome present serious difficulties in the precise determination of physical properties of metals and alloys, a high vacuum-high temperature chamber has been designed that will enable the meaningful measurement of crystal lattice parameters of such important metals as titanium, zirconium, hafnium, thorium, uranium, and rare earths which are readily contaminated by the residual oxygen and nitrogen.

*Utah Univ., Salt Lake City.
PRESSURE DEPENDENCE OF MECHANICAL PROPERTIES OF METALS AND IONIC AND VALENCE CRYSTALS. P. Gibbs. Project 9760(802), Contract AF 49(638)-853; AFOSR, DSS.

The purpose of this research is to study experimentally the pressure, temperature, and imperfection dependence of creep, internal friction, and fracture in a series of metal and ionic and valence crystals. Experiment does appear to confirm the tendency for lattice vacancies, voids, and "empty places" to be squeezed out by pressure, particularly at high temperatures. Crude estimation suggests that the vacancy concentration may be reduced by more than an order of magnitude. In a similar fashion, pressure might have only a small direct effect on dislocation structure in metals. There is no dilatation associated with screw dislocations, and edge dislocations are symetrical in dilatation and compression in isotropic elastic media. According to widely accepted theories, any large change in dislocation energy should be reflected in the critical shear stress for deformation. As might be expected, Bridgman found pressure-induced changes only of the order of 10% of the critical shear stress of metals. Thus one expects that pressure may influence the vacancy distribution in a different way from the dislocation structure, and in principle may serve to distinguish between them. Of course, pressure also may influence the rates at which the expected effects take place, and this must be taken into account in the analysis of data.

*Materials Central, WADD, Dayton, Ohio.
SINGLE CRYSTAL FILAMENTS. H. Weik. Project 7023(805), Internal

The mechanism of growth correlated to the mechanical properties of single crystal filaments will be studied. The crystallographic orientation of single crystals grown in filaments has to be determined in order to interpret properly the strength obtained. A comparison between these results and others reported for larger single crystals of the same metal will be attempted to provide a full understanding of the crystal growth in the filament. Distribution, arrangement and movement of surface dislocations have to be considered for the interpretation of the elastic limit up to which these filaments could be used. In addition chemical plating of such filaments having the highest strengths should be attempted which should theoretically raise considerably their strength above the ultimate strength for uncoated filaments by blocking dislocation movements.

*Pennsylvania State Univ., University Park.
STUDY OF WHISKER-TIP SURFACES WITH FIELD EMISSION MICROSCOPE TECHNIQUES. E. Muller. Project 7023(802), Contract AF 33(616)-6397; WADC, MC.

The investigation of crystalline structure and imperfections of iron whiskers with the field emission microscope will be continued to gain insight into the mechanism of their growth and into possible correlations of their imperfections and their unusual physical (mechanical) properties. The techniques employed with the field emission microscope, which allows one to study structures on an atomic scale, are very exacting. The instrument designed for work on iron whiskers is complete. Interest will be centered on the whisker tips, but some cross sectional areas at stations along their length will also be considered.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*California Univ., Los Angeles.
TEXTURE AND INELASTIC PROPERTIES OF FACE-CENTERED CUBIC CRYSTALS BASED ON SLIPS. T. H. Lin. Project 9763(802), Contract AF 49(638)-20; AFOSR, DSS.

The research is to develop a mathematical theory to calculate the texture of the metal under tension and compression, and the stress-strain relationship under reversal of loading beyond elastic limit (Bauchinger effect). This theory is to be developed based on slips on crystallographic planes along crystal slip directions.

Radiation Effects on Metals

Stanford Univ. Electronics Research Lab. Calif. Technical Report No. 71. Also published in Jour. Appl. Phys., v. 24; 826-833, July 1953.
EXPERIMENTS ON RADIATION BY FAST ELECTRON BEAMS. H. Motz, W. H. Thon and R. N. Whitehurst. Sponsored jointly by Office of Naval Research, Signal Corps, and Air Force Office of Scientific Research under N6onr-25107. 1953. ASTIA Document No. AD 19030.

The results of some experiments on millimeter wave and light generation by means of an undulator are described. After a brief survey of the theoretical background the design of a magnet system is discussed. An experiment is described in which a 100-mev electron beam from the Stanford linear accelerator passed through the undulator. Light radiated by the beam was observed and the plane of polarization determined. A small linear accelerator with good bunching action was used for an experiment on millimeter wave generation. At a beam energy of 3 mev, radiation in a wavelength band below 1.9 mm was observed. A peak power output of the order of one watt was obtained. Millimeter waves generated in the accelerator tube were also observed.

Maryland Univ. Inst. for Fluid Dynamics and Applied Mathematics. College Park. Technical Note No. BN-36. AFOSR-TN-54-148. Also published in Summa Brasiliensis Math. Rio de Janeiro, v. 3, Fasc. 7:125-146, 1955.
THE GENERALIZED RADIATION PROBLEM AND THE EULER-POISSON-DARBOUX EQUATION. A. Weinstein. AF 18(600)-573. June 1954. ASTIA Document No. AD 35529.

The classical radiation problem for the wave equation is reformulated in such a way that a problem for the Euler-Poisson-Darboux equation in two independent variables includes as a special case the problem of Tricomi and more recently Germain and Bader (Office National d'Étude et de Récherche Aeronautique, No. 54, 1952, and Sur le probleme de Tricomi Rendiconti del Circolo Matematico de Palermo, Series 2, v. 2: 53, 1953). The latter problem is concerned with transonic flows in which arbitrary data are given on the sonic line and prescribed to be zero on a Mach line. Precise conditions are given under which the radiation problem can be solved, and new formulas are obtained for the solution of the problem for the Tricomi equation. (ASTIA abstract)

*Metallurgy and Ceramics Research Branch, ARL, Dayton, Ohio.
EFFECT OF NEUTRON IRRADIATION ON THE STRENGTH OF AGE HARDENING ALLOYS. A. M. Adair. Project 7024(802), Internal.

The drastic reduction in strength of age hardening aluminum alloys, resulting from exposure to high level neutron irradiation, is not as yet fully explained. The attendant ductility loss normally associated with high levels of neutron irradiation was overshadowed by the redistribution of solid solution elements.

Experiments are being conducted to determine whether the loss of properties can be explained by irradiation or whether a rise in temperature is also required. Attempts will be made to determine a temperature equivalence for neutron irradiation. Preliminary experiments indicate that a large number of line defects are formed by high levels of neutron irradiation.

*Westinghouse Electric Corp., Dayton, Ohio.
MAGNETIC MATERIALS. J. A. Osborn. Project 7021 (802), Contract AF 33(616)-5555; ARL, MCB.

The effects of irradiation on single crystals of magnetic materials are being studied to determine the mechanism by which their magnetic properties are altered in a radiation environment. In addition, the effects of elevated temperatures on the magnetic characteristics of commercially available magnetic alloys in silicon-iron, cobalt-iron, and aluminum-iron are being determined.

*Colorado Univ. Boulder.
NUCLEAR RESONANCE ON Al IN SYNTHETIC RUBY. W. H. Tanttila. Project 9763(802), Contract AF 49 (638)-611; AFOSR, DSS.

The primary aim of this research is to study the interaction between the Cr ion and the Al nuclei in synthetic ruby. This interaction will be studied as a function of temperature. The relaxation process of the Al should be governed at some low temperature by the relaxation of the Cr ions via their mutual interaction. By studying the temperature dependence of the relaxation time and the line width of the Al nuclear resonance below the temperature, one should be able to get information on the interaction energy and the Cr ion relaxation time. Additional information on spin conduction by the nuclear system will of course be possible to obtain. The approach will be as follows: First, the line width and quadrupole interaction energy will be studied as a function of temperature and orientation for several single crystals (not more than four) of different Cr ion concentration. These experiments will be done using continuous resonance absorption (marginal oscillator with phase sensitive detection). Following this, pulsed nuclear induction will be used to study the relaxation time as a function of temperature, crystal orientation, and Cr ion concentration. Following these experiments, it is possible that, from the results, continuous absorption experiments may be interesting on several of the samples to look at the line widths of the nuclear resonance. The samples will range in Cr ion concentration from about .1% to 5%. The pulsed experiments will be done on about 8 samples of different Cr ion concentration.

*Michigan State Univ. East Lansing.
PARAMAGNETIC RELAXATION AT VERY LOW TEMPERATURES. R. D. Spence, J. A. Cowen. Project

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

9763(802), Contract AF 49(638)-613; AFOSR, DSS.

This research includes investigation of the following: (1) electron spin-lattice and lattice heat bath relaxation by means of low field, low frequency paramagnetic resonance; (2) color centers or other radiation induced damage; (3) nuclear relaxation as a function of paramagnetic centers; (4) interaction between relaxation times of paramagnetic impurities and nuclear spin precession times when they are of similar duration; (5) antiferromagnetic relaxation examined by a probe nucleus or paramagnetic center; and (6) use of second sound below 2.2°K to conduct relaxation energy to a detector.

*Solid State Physics Research Branch, ARL, Dayton, Ohio.

PRODUCTION OF EDGE EMISSION CENTER IN CADMIUM SULFIDE BY NUCLEAR RADIATION. B. A. Kulp. Project 7112(802), Internal.

The present effort is being directed toward determining the edge emission center, the center responsible for the red continuum and the infrared quenching centers in cadmium sulfide crystals. This work is done on whiskers since they are structurally very perfect and void of many of these centers when grown. The centers are then produced by nuclear radiation bombardment. The present bombardments are being made primarily with electrons accelerated with a Cockroft-Walton generator. Determinations of the displacement energy for both sulfur and cadmium are being made and the cross sections for both reactions will be determined. Similar measurements will then be made on zinc sulfide, and other materials of interest to our internal effort. The center for edge emission is either a sulfur vacancy or a sulfur interstitial. The threshold for displacement of a sulfur atom from a lattice position has been measured to be about 9.8 ev., corresponding to 130 Kev electrons. Work is continuing on the effects of bombardment at both lower and higher energies.

*General Electric Co., Schenectady, N. Y.

RADIATION DAMAGE THRESHOLDS OF THE ELEMENTS. R. M. Walker. Project 5620(802), Contract AF 19(604)-5557; ERD, EML.

A study is being made of the radiation damage thresholds of various metals. Highly purified samples of copper, aluminum, zinc, tin, iron and nickel have been prepared and some irradiated. Onset of radiation damage is determined at liquid helium temperatures by resistivity. Annealing curves have been measured and damage production rate data are being measured. These data when analyzed will be compared to the low temperature neutron irradiation data. Radiation damage on cold worked and annealed metal samples is being studied.

*Battelle Memorial Inst., Columbus, Ohio.

RADIATION EFFECTS RESEARCH ON SEMICONDUCTOR MATERIALS. R. F. Willardson. Project 7112(802), Contract AF 33(616)-3747; ARL, SSB.

Radiation damage studies are conducted primarily on compound semiconductors such as GaAs, InSb, rather than monatomic materials, with particular emphasis being placed on determining the mechanism of the effects in these materials. Fast neutrons from a reactor are used for producing the effects. Annealing studies are made to assist in establishing a model to explain the radiation effects.

*Brown Univ. Providence, R. I.

RADIATION EFFECTS IN SOLIDS. R. Truell, C. Mylonas. Project 9763(802), Contract AF 49(638)-450; AFOSR, DSS.

Boron-containing solids, such as glass, will be irradiated with thermal neutrons. The neutron-boron interaction produces energetic heavy particles within the solid which in turn can bring about profound changes in the physical behavior of the solid. Ultrasonic and photoelastic methods will be used to measure such changes in the physical properties as density, strain, absorbed energy, scattering, etc., with respect to point defects and dislocations which occur. An immediate by-product of this work lies in the use of boron-containing glass or other materials containing boron as neutron irradiation detectors and dosimeters probably capable of measuring the total flux and the velocity distribution of neutron irradiation.

Pittsburgh Univ. Pa.

RADIATION EFFECTS USING CYCLOTRON IRRADIATIONS. J. Townsend. Project 5620(802), Contract AF 19(604)-3906; ERD, EML.

This project is directed towards learning the number of defects produced per unit energy absorbed from a cyclotron produced proton beam. The targets consist of polycrystalline wires, single crystals and whiskers of each of a number of elements. The defects introduced by the proton beam are detected as changes in resistivity and in Young's Modulus of Elasticity in this particular investigation. At present, the irradiations are carried out at room temperature, but a cryostat capable of maintaining liquid helium temperatures has been constructed. Annealing curves (resistivity as indicator) are being determined from room temperature to approximately 400°C. In the near future these annealing curves will be measured from liquid nitrogen, and then from liquid helium temperatures. A limited number of elements, tungsten, nickel, and copper have been worked with in order to develop technique. The data have been plugged into the appropriate equations and the differences are being examined. Ultimately, these data will be used to prepare a more exact mathematical model.

*Armour Research Foundation, Chicago, Ill.

STRUCTURE OF DEFECT CLUSTERS IN SOLIDS. R. H. Bragg. Project 9763(802), Contract AF 49(638)-829; AFOSR, DSS.

The contractor will undertake a theoretical and computational analysis relating measurements of small angle scattering of x-rays in solids to the sizes and shapes of particles in the solids; and apply this analysis to the study of particles in the solids, principally defect clusters produced by nuclear irradiation. This investigation will be centered around study of neutron damage in lithium fluoride crystals. The mechanism whereby the point imperfections resulting from irradiation are able to condense to form cavities and precipitates of impurity atoms will be considered.

Semiconductors

*Solid State Physics Research Branch, ARL, Dayton, Ohio.

CdS AND ZnS TYPE SEMICONDUCTORS. D. C. Reynolds.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

Project 7021(802), Internal.

This study has been concentrated on determining the nature of the electronic phenomena of synthetic CdS, ZnS and mixed Zn, CdS single crystals. The major effort has been expended on CdS crystals. Mechanically excited luminescence in CdS has been discovered. This phemomenon is one in which single crystals of CdS stimulated either by light or thermal energy at liquid N_2 temperature give off green emission when subjected to a mechanical impact. The threshold for light stimulation is 6900A°, the emitted light is approximately 5200A°. The temperature necessary for thermal stimulation is above -80°C. The green light can also be excited by infrared radiation. The stimulating radiation causes the conductivity of the sample to increase very markedly and this conductivity as well as the green light is stored indefinitely. The polarization of the emitted light as well as the stimulating and exciting energies allows one to use these phenomena to study the various transitions that occur in CdS and also to study the energy level scheme of this material.

*Pontificia Universidade Catolica du Rio de Janerio, Brazil.

CHARGE STORAGE AND IRRADIATION EFFECTS IN DIELECTRICS. B. Gross. Project 9763(802), Contract AFOSR 60-6; AFOSR, DSS.

A range of electrostatic phenomena will be studied in solid dielectrics with attention being directed to such topics as: (1) the properties of electrets, (2) storage of charge accumulated from electron bombardment, (3) charge storage after gamma irradiation, and (4) radiation induced conductivity of dielectrics. The first of these, study of electrets, will receive most attention. These are the electrostatic equivalent of the magnet, That is, they remain electrically polarized for decades without loss, the polarization intensity being limited only by the dielectric breakdown of air, about 10,000 volts per centimeter.

*Illinois Univ. Urbana.

DIFFUSION IN SEMICONDUCTORS. J. Bardeen. Project 9763(802), Contract AF 49(638)-417; AFOSR, DSS.

Investigations under the present program include: (1) measurement of the diffusion coefficient of groups III, IV, and V elements in silicon as a function of crystal preparation, in particular, the effects of dissolved gases such as O_2; (2) measurement of the diffusion coefficients of various transition elements in silicon; (3) a study of the various proposed diffusion mechanisms in semiconductors such as Ge and Si; (4) a study of the effects of large microwave electric fields on the mobility of carriers (hot electrons) in germanium; (5) a study of the ionization changes as a result of "heating" of electrons to higher than thermal energies, particularly with microwave fields; (6) a study of the mobility of trapping of carriers in amorphous semiconductors such as selenium, by measurement of the carrier lifetime in the milli-microsecond range; (7) study conduction energy transfer processes and optical absorption in organic semiconductors.

*Cornell Univ., Ithaca, N. Y.

EFFECT OF CRYSTALLINE IMPERFECTIONS UPON THE ELECTRICAL PROPERTIES OF SEMICONDUCTING INTERMETALLIC COMPOUNDS. C. W. Spencer. Project 9760(802), Contract AF 49(638)-480; AFOSR, DSS.

The semiconducting intermetallic compounds Bi_2Te_3 and Bi_2Se_3 are of interest from a theoretical standpoint because they offer an opportunity to study the interrelationship between crystal lattice defects and the electrical properties of semiconductors. It has been found that the electrical properties of Bi_2Te_3 crystals are markedly influenced by the rate at which they were grown and annealed just below the melting point. Also, it has been difficult to obtain material containing less than 10^{19} current carriers per cm^3. Crystalline imperfections are probably in large part responsible for the unusual properties and characteristics observed. Research so far has been concerned with the growth of high quality crystals of Bi_2Se_3. Material containing 10^{18} current carriers per cm^3 has so far been obtained. The nature of the reaction between the molton material and its environment has also been studied using a mass spectrometer to identify the molecular species produced. Finally, the effect of selenium vapor upon the electronic properties of the crystals is being examined.

*Batelle Memorial Inst., Columbus, Ohio.

EFFECTS OF VERY HIGH PRESSURE AND TEMPERATURE ON SEMI-CONDUCTIVITY AND INSULATING MATERIALS. C. M. Schwartz. Project 9763(802), Contract AF 49(638)-441; AFOSR, DSS.

The simultaneous application of both high pressure and high temperature has been employed recently as a means for the formation of new phases in materials. The research program will be directed towards obtaining fundamental knowledge of the structure and behavior of solids, as well as gaining new insight into profitable areas of future research in new materials which may be synthesized in an environment utilizing high pressures and temperatures. In situ measurements of electrical conductivity and thermoelectric power of such solids as NiO, BeO, etc., having marked differences in zone structure, may lead to the selection of additional oxides of the type RO, RO_2, R_2O_X, etc. From a systematic study of the effects of high pressure and temperature on such a series of oxides covering an extensive range of valence, thermodynamic stability, crystal structure, and energy gap, new knowledge of unusual characteristics and properties of solid state materials may direct further development of materials, devices, and equipment that can withstand extreme conditions of temperature and pressure.

*General Mills, Inc., Minneapolis, Minn.

ELECTRICAL CHARACTERISTICS OF GRAIN BOUNDARIES IN COMPOUND SEMICONDUCTORS. R. K. Mueller. Project 9761(802), Contract AF 49(638)-628; AFOSR, DSS.

This is an investigation of the electronic properties of grain boundaries, particularly very low angle grain boundaries which may be regarded as an array of edge dislocations. The material for study in Dr. Mueller's past work has been germanium, and he would like to extend this to other semiconductor materials. The contractor will conduct research on: (1) preparation of InSb; (2) preparation of precisely oriented bicrystals in n- and p-type material (tilt boundaries, twist boundaries, and coherent twin boundaries); (3) X-ray study of these grain boundaries; (4) study of their electrical and optical properties; and (5) theoretical studies and interpretation of experimental results.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Chicago Univ., Ill.
ELECTRONIC PROPERTIES OF GERMANIUM SEMICONDUCTORS AT HELIUM TEMPERATURES. H. Fritzsche. Project 9760(802), Contract AF 49(638)-802; AFOSR, DSS.

The object of this research is to elucidate the structure and the interaction of impurity centers in some of the semiconducting materials. Germanium will be investigated first, because the methods for its preparation and the theory of its properties are most highly developed. Controlled compensation and high compressional and tensile shear stresses will be used in the study of semiconduction at low temperatures as the novel features of this research. Although piezoresistance has been repeatedly used in the past to determine whether a semiconductor is of the multivalley type or not, the yield of quantitative information has been rather limited. The extension of the piezoresistance studies to higher stresses and to lower temperatures promises to yield previously unobtainable information about the deformation potentials and the structure and degeneracies of impurity states and important results concerning the conduction phenomena at low temperatures.

*California Univ., Berkeley.
ELECTRONIC STRUCTURE OF SEMICONDUCTOR AND METAL CRYSTALS. A. F. Kip. Project 9760(802), Contract AF 49(638)-600; AFOSR, DSS.

The principal object of the research under this contract is to determine the energy band and impurity level structure of solids, with particular emphasis on semiconductors. It is planned to exploit further the newly developed techniques for studying cyclotron resonance in metals and to extend the method to new metals and new semiconducting crystals. Such studies will add new detail to our knowledge of conduction processes. Low temperature studies will be extended of solids to higher microwave frequencies and to infrared wavelength regions which have been neglected so far.

*Wisconsin Univ., Madison.
ENERGY LEVELS IN SEMICONDUCTOR MATERIALS THROUGH CYCLOTRON RESONANCE (CdS/ZnS). R. Dexter. Project 7021(802), Contract AF 33(616)-6111; ARL, SSB.

Professor Dexter is utilizing microwave conductivity and cyclotron resonance techniques to obtain the band structure parameters in cadmium sulfide and zinc sulfide. The high degree of perfection of the whiskers and platelets grown by L. C. Green working in the in-house program of the Aeronautical Research Laboratory under this task have made these measurements possible.

*General Electric Co., Schnectady, N. Y.
FIELD ENHANCED DIFFUSION PROCESSES IN SEMICONDUCTORS. E. Pell. Project 5621(802), Contract AF 19(604)-5551; ERD, EML.

The work to be performed under this contract will include an investigation and study of the diffusion of such atoms as lithium, copper, gold and silver through semiconductor crystals. The stabilization of such fast-diffusing elements will be sought by addition of a compatible second element. Special emphasis will be placed on understanding and exploiting the motion imparted to some diffusion atoms by the application of an electronic field. This offers possibilities for making sharp, contoured electrical p-n junctions.

*Illinois Univ., Urbana.
METALLIC PROPERTIES OF TITANATE SEMICONDUCTORS. E. K. Weise. Project 9760(802), Contract AF 49(638)-212; AFOSR, DSS.

The objective of this research is to investigate the properties of highly reduced titanates, especially those of the second group of the periodic table, namely: magnesium dititanate, pseudo brookite structure; calcium metatitanate, perovskite structure; strontium metatitanate, perovskite structure; and barium metatitanate, several structures. The experimental phases of the research will include: (1) a determination of Ti^{3+} ions which act as electron sources; (2) a determination of the X-ray structure of the samples; (3) measurements of the electron conductivity as a function of both composition and temperature; (4) measurements of the magnetic susceptibility as a function of temperature, and, where possible, the magnetic anisotropy of single crystals; and (5) measurements of the specific heat, heat conductivity, and thermoelectric effects as a function of composition and temperature.

*Minnesota Univ., Minneapolis.
MICROWAVE STUDIES OF SEMICONDUCTOR CRYSTALS. K. S. Champlin. Project 9763(802), Contract AF 49(638)-747; AFOSR, DSS.

Microwave energy will be used in this research for two different purposes. First, a low energy electromagnetic wave will be used as a "probe" to measure the free carrier concentration in the sample. Second, high energy microwaves will be used to "heat" or energize the carriers and to create new carriers through impact ionization. Experiments involving energy transfer mechanisms, carrier transport, and recombination processes will be performed utilizing the above methods. In addition these techniques will be employed to measure the "hot" (energized) electron effects by completely electrodeless techniques. It is believed that electrodeless microwave measurements will prove superior to more familiar "ohmic" contact methods in experiments demanding a high degree of precision involving the following: (1) the study of the absolute properties of materials such as the III-V compound semiconductors; (2) experiments where surface ambients must be carefully controlled, such as in the study of recombination and trapping processes; (3) experiments requiring precise knowledge of the geometry and boundary conditions, such as in the study of transport processes; and (4) studies at low temperatures and high current densities where "ohmic" contacts show anomalous behavior, as for example the study of "hot" electrons.

*Clevite Corp., Cleveland, Ohio.
NEW HIGH TEMPERATURE SEMI-CONDUCTING MATERIALS. L. Shiozawa. Project 7021(802), Contract AF 33(616)-6865; ARL, SSB.

Research will be performed on methods of preparing materials of optimum purity. These high purity materials will be used to prepare single crystals of binary compounds and single crystals of solid solutions of mixed compounds. Optical and electric techniques will be used to study the fundamental properties of these materials. Studies will be made to determine whether phenomena occurring in binary compounds also occur in solid solutions of these compounds with related compounds. For example, the photovoltaic effect in cadmium sulfide has been observed. How is this modified when solid solutions of cadmium sulfide and cadmium selenide are formed? Quantitative spectroscopic analysis will be performed on samples of cadmium sulfide, zinc sulfide and solid solutions of these compounds.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Cornell Univ., Ithaca, N. Y
NEW SOLID STATE DEFECT STRUCTURES. M. J. Sienko. Project 9762(802), Contract AF 49(638)-191; AFOSR, DSS.

This is a study of non-stoichiometric compounds, specifically the tungsten bronzes. New materials such as copper, lead, and silver tungsten bronzes, and possibly vanadium bronzes, are being synthesized. Measurements of infrared absorption, thermal coefficient of conductivity, and Hall coefficient are being carried out to determine the electronic structure of these materials. These materials appear to be semi-metallic in character in that their electrical conductivity drops with an increase in temperature - the reverse of semiconductor behavior and of many other non-stoichiometric materials which have been studied. such research has a good chance of leading to the discovery of materials which are temperature insensitive semiconductors having high resistance to radiation damage. They also are chemically inert and may turn out to be of interest for thermoelectric power applications.

*Dayton Univ., Ohio.
OPTICAL PROPERTIES OF SEMI-CONDUCTING SINGLE CRYSTALS. W. R. Rambauske. Project 7021(802), Contract AF 33(616)-7500; ARL, SSB.

Experimental and theoretical research on the optical properties of II-VI compound semiconductor single crystals bearing on their band structure and interactions with electromagnetic radiation in the ultraviolet, visible and near infrared regions of the spectrum. Specifically, perform absorption, emission, reflection and photoconductivity measurements on CdS, ZnS, CdSe, ZnSe, ZnTe and other related semiconductor crystals, as a function of wavelength in the UV, visible and near IR regions, at various temperatures including that of liquid helium. The results of these measurements will be analyzed, in comparison with existent theories, in an attempt to obtain a more nearly complete understanding of the band structure of the materials investigated.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
ORGANIC SEMICONDUCTING COMPOUNDS. N. W. Silcox. Project 5620(802), Internal.

The object of this effort is the investigation of organic compounds for use as semiconducting materials and to study the properties of these compounds on a laboratory scale. Experimental investigations will be initiated beginning with the organic dyes. The melting point, energy gap, change of energy gap with temperatures, intrinsic resistivity, mobility and lifetime of such dyes as cynathrone, indanthrene (black), indanthrone, indantrazine, flavanthrone, isoviolanthrene, pyranthrone, pyranthrene, ovalene, isodibenzanthrone, anthracene, naphthacene, anthranthrene, methylene blue, coroene, and other such compounds will be determined as a first step. In addition, the physical chemistry of these compounds (vapor pressure, decomposition temperature, lattice parameters) will be studied. The importance of purity and defect structure on the semiconducting properties of these compounds will also be investigated.

*Armour Research Foundation, Chicago, Ill.
ORGANIC SEMICONDUCTORS. D. E. Laskowski. Project 9762(802), Contract AF 49(638)576; AFOSR, DSS.

This investigation is concerned with a review of current research on organic semiconductors, a theoretical study of electron donors and acceptors, and the experimental study of addition compounds and polymers which may conduct by coupled oxidation-reduction processes. Pairs of compounds forming addition compounds being studied include aromatics with halogen molecules, nitroaromatics, and quinones. The polymer, polytributylin methacrylate with addition of iodine molecules is being examined.

*Syracuse Univ., N. Y.
PARAMAGNETIC RESONANCE IN SEMICONDUCTORS. A. Honig. Project 9762(802), Contract AF 18(603)-50; AFOSR, DSS.

Investigations in paramagnetic resonance currently refer to the study of induced transitions between energy states which arise from the application of a magnetic field to atoms or ions with magnetic moments of the order of an electronic Bohr magneton. In semiconductors, such as silicon and germanium, which are doped with elements from group V or III of the periodic table, there is respectively an odd electron or hole associated with each of the impurity atoms; this odd electron or hole gives rise to the paramagnetism. The principal objective of this investigation is to conduct research on paramagnetic resonance in semiconductors with the view of corroborating further the existence of a high degree of nuclear polarization and of understanding better the mechanism involved. The experimental work on doped silicon includes: (a) a remeasurement of the intensity relations between neighboring hyperfine lines under better signal to noise conditions; (b) study of the simultaneous resonance of electrons and nuclei; (c) a corroboration of the existence of nuclear resonance by measuring the angular correlation of gamma rays in As^{73} doped silicon; (d) measurement of the energy levels of donors in silicon; (e) testing of theories of spin-lattice relaxation; and (f) measurement of the infrared spectral response in P, As, and Sb doped silicon.

*Association des Amis du Laboratoire de Physique de l'École Normale Supérieure, Alpens, Paris, France.
RECOMBINATION RADIATIONS FROM SEMICONDUCTORS. P. Aigrain. Project 9760(802), Contract AF 61(052)-370; AFOSR, DSS.

The study of recombination radiations (electron-hole) emitted from the semiconductors germanium and silicon has produced much information concerning the recombination process and other phenomena. The method is complementary to the study of absorption and magneto-absorption from these materials. However, in many instances, recombination radiation techniques are potentially much more accurate than absorption techniques. This has made possible the refined observation of infrared emmission of photon energy much smaller than gap energy from the recombination of hole-electron pairs in dislocated germanium and hence, a study of the mechanisms involved in the recombination process. A new model of the way dislocation produced recombination of hole-electron pairs take place in germanium has resulted. The purpose of this research is to further exploit these techniques to (1) study impurity induced radiations; (2) magneto-effects such as the Zeeman effect; and (3) observe and study unpinned dislocations in rather perfect crystals by their own infrared radiations.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Oklahoma A. and M. Coll., Stillwater.
SEMICONDUCTING PROPERTIES OF TYPE IIb DIAMONDS. W. V. Leivo. Project 9763(802), Contract AF 18(603)-40; AFOSR, DSS.

The present research is directed toward achievement of a basic understanding of the electronic properties of diamonds through study of natural type IIb and attempts to synthesize this type from ordinary diamonds. The following research is being performed: (1) Determination of rectification at the junction of various metals with the diamond and comparison with theoretical rectification equations; (2) Study of the absorption spectrum of the diamond; (3) Study of the phosphorescent and thermoluminescent properties of diamond; (4) Measurement of Hall effect as a function of temperature to determine activation energy of the carriers; (5) Measurement of photoconductivity and comparison with activation energy; (6) Determination, if possible, of carrier drift mobility; (7) Study of photovoltaic effect and carrier lifetime.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
SEMICONDUCTOR MATERIALS. R. F. Cornelissen. Project 5620(802), Internal.

The internal effort under this task has been as follows: (1) an accurate equilibrium phase diagram of the carbon-silicon system by use of physical chemical theories and statistical thermodynamics has been established. This diagram has been calculated for the 1700°K isotherm; with the accumulation of further experimentally determined physical constants, other isotherms will be calculated to complete the three dimensional (T, P, C) plot. An experimental furnace for growing SiC crystals from the vapor has been designed and built; trial runs will be started very soon. (2) The II-IV class of compounds has been theoretically investigated for their semiconductor properties, and found promising. Magnesium stannide has been prepared and single crystal techniques are being worked on. (3) New solid state phenomena associated with semiconductors are being theoretically investigated. These are the concept of negative effective mass, negative dielectric constant, and nonlinear dependence of mobility on frequency. These may form the basis of new active devices.

*Brown Univ., Providence, R. I.
SURFACE PROPERTIES OF SILICON AND OTHER SEMICONDUCTORS. H. E. Farnsworth, J. A. Dillon. Project 5621(802), Contract AF 19(604)-5986; ERD, EML.

Studies of the effects of ion bombardment on silicon and germanium crystals were completed. Results showed that etch pits which appeared on various crystalline faces are different. The etch pit formation is not sensitive to bombarding ambient. Study of diffusion of surface contamination into silicon was further carried out to substantiate the effectiveness of ion bombardment as a cleaning up procedure. Construction of a vacuum system for point-contact rectification between silicon and metal was completed. Preliminary studies of point-contact rectification between metal and silicon carbide were undertaken. By use of low energy electron diffraction techniques, the surface cleanliness of an ion bombarded and annealed sample of bismuth telluride was found equal to the cleanliness of a freshly cleaved surface of that compound. These freshly cleaned surfaces were found to have practically negligible chemisorption for oxygen and carbon monoxide, thus indicating complete saturation of surface bonds. Water vapor adsorption is now under study.

*Kansas State Coll., Manhattan.
THIN FILMS OF SEMICONDUCTORS. E. B. Dale. Project 5621(802), Contract AF 19(604)-7218; ERD, EML.

This work is a fundamental research backup to one approach to integrated curcuitry. This approach is through the utilization of thin single crystal films of semiconductors to realize compactness, ruggedness and ultra-miniaturization. A flash-anneal technique will be investigated as a method of producing single crystal films of both elemental and compound semiconductors on substrates of the respective materials. Crystal structure of such films as a function of various growth variables will be studied using X-ray and/or electron diffraction. The possibility of using surface magnetoresistance effects to determine carrier density and mean-free path in semiconductor films will be explored. Such determinations are at present difficult and unreliable. It is possible that a new tool may thus be provided for exploring surface phenomena in general.

*Kentucky Univ., Lexington.
TRANSFORMATION PROPERTIES OF SULFIDE SEMICONDUCTORS. L. Gildart. Project 9762(802), Contract AF 49(638)-90; AFOSR, DSS.

The physical properties of "semi-metal" compounds, such as antimony selenide, arsenic selenide and antimony telluride, are being investigated. These compounds may exist in either glassy or crystalline states and are interesting for study as semiconductors, photoconductors, infrared transmitters and filters, and thermoelectric elements. In addition to such applications, it is of importance to understand the nature of atomic and molecular bonding present in these compounds as a contribution to general solid state theory. One of the discoveries of this program is a field-induced switching cycle in stibnite, Sb_2S_3, containing excess antimony. That is, the resistance may be switched repeatedly between 10^4 and 10^{10} ohms. This semiconductor resistivity phenomenon has not previously been observed. Another part of this research is a study of solidification properties of some semiconducting materials which give vitreous as well as crystalline solids. This binary approach to the study of the same material gives promise of an understanding of the relationship between electronic properties and chemical bonds present.

Solid State Sciences

*Alfred Univ., N. Y.
DIELECTRIC AND MECHANICAL LOSSES IN SOLIDS. D. P. Detwiler. Project 9763(802), Contract AF 49(638)-87; AFOSR, DSS.

Although considerable work has been done on the study of internal friction in metals, relatively few measurements have been made on insulating crystals. Consideration of the mechanisms leading to the occurrence of internal friction in solids leads to the conclusion that in non-conducting materials many of the same mechanisms might be expected to produce dielectric losses. It has been suggested that there appears to be a close correlation between dielectric and mechanical losses in materials including crystalline oxides, sulfides, selenides, etc., but no detailed studies of these losses have been reported. The purpose of this study is to measure the mechanical and dielectric losses of aluminum oxides, magnesium oxide and aluminum sulfide;

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

to attempt to relate the observed dielectric losses to the internal structure and changes in internal structure of these materials; and to attempt to show that for non-conducting materials dielectric loss measurements yield the same information as do mechanical loss measurements.

*Northwestern Univ., Evanston, Ill.
DIELECTRIC AND PIEZOELECTRIC PROPERTIES OF SINGLE CRYSTALS OF CdS and ZnS. R. Frericks. Project 7021(802), Contract AF 33(616)-5625; ARL, SSB.

This contract supports experimental investigations of the dielectric and piezoelectric phenomena in cadmium sulfide and zinc sulfide and in mixed zinc-cadmium sulfide crystals.

*National Bureau of Standards, Washington, D.C.
ELECTROLUMINESCENCE AND THERMOELECTRICITY. G.G. Harman. Project 5621(802), Contract CSO and A 58-41; ERD, EML.

Research and development is being performed on methods of measuring the unique properties of electroluminescent and thermoelectric materials. Also included is an analysis of the band structure and an investigation of minority carrier lifetime in silicon carbide. The latter measurement being made through use of the electroluminescent properties in the case of silicon carbide.

*Paris Univ., France
ENERGY TRANSPORT PHENOMENA IN CADMIUM SULFIDE CRYSTALS. M. Balkonski. Project 7116(801), Contract AF 61(052)-130; ARL, SSB.

The fundamental transport mechanisms in cadmium sulfide are being investigated in this effort. This involves an understanding of the energy level schemes in cadmium sulfide. Theoretical calculations of the band structure of cadmium sulfide have been made. The theory has been verified in part by optical experiments. Apparatus for measuring cyclotron resonance has been constructed. This will help in experimentally determining the band structure. Measurements of photoconductivity are being used in an effort to determine whether energy is transported by exciton diffusion.

*Purdue Univ., Lafayette, Ind.
OXIDATION AND STOICHIOMETRY OF PRASEODYMIUM AND CERIUM OXIDES. A. F. Clifford. Project 9760 (802), Contract AF 18(603)-45; AFOSR, DSS.

This research is concerned with: (1) a careful correlation between the thermoelectric power and the stoichiometry of praseodymium and cerium oxides; (2) determination of magnetic susceptibility of these oxides as a function of composition to obtain information concerning the electronic properties in non-stoichiometric crystals; (3) determination of dielectric loss in the above compounds to complement studies (1) and (2); and (4) a study of the solid state kinetics by which some of the rare earth nitrates convert to rare earth oxides upon heating.

*Harshaw Chemical Co., Cleveland, Ohio.
PHOTOVOLTAIC CELLS. F. Shirland. Project 7116 (801), Contract AF 33(616)-6548; ARL, SSB.

This contract is aimed at high efficiency cells by use of graded functions and multi-gap materials. Preliminary tests have been made on some combination cadmium sulfide-zinc telluride and cadmium sulfide-zinc selenide cells. Open circuit voltage as high as 0.7V have been observed. However, the efficiencies have been low due to the high resistance of the material. Attempts will be made to produce high efficiency cells by use of graded functions and multi-functions of different materials.

*Wayne Univ., Detroit, Mich.
PHOTOVOLTAIC EFFECT IN COPPER OXIDE. D. Trivich. Project 9761(802), Contract AF 18(600)-481; AFOSR; DCS.

The project has the objective of investigating means of preparing front-wall cuprous oxide cells and of studying the properties of such cells. To this end the properties of cuprous oxide itself and the effect of impurities, of orientation, and of surface states will be studied.

*New York Univ., N. Y.
PHOTOVOLTAIC EFFECTS. H. Kallman. Project 6692(750), Contract AF 19(604)-5495; GRD, PL.

Research is being conducted on the large photovoltages reported for crystals and evaporated layers of the zinc-group VI type. Work is also being performed on the growth, purification and characterization of organic crystals.

*Stanford Research Inst., Menlo Park, Calif.
SOLID STATE ELECTROLYTES. W. J. Fredericks. Project 5620(802), Contract AF 19(604)-7231; ERD, EML.

A study will be made of all phenomena in solids which involve interactions between chemical imperfections and between chemical and physical imperfections to which the concept of solid state electrolytes can be applied. An over-all program will be undertaken to provide new data pertaining to ion-pairing reactions and their chemical and electrical consequences; to the interactions of imperfections to form donor and/or acceptor complexes and their stability and chemical identity; and to the kinetics and mechanism of solute precipitation. The method for study will include electronic and ionic conductivity, diffusion, electrolysis, optical absorption spectroscopy, magnetic susceptibility, luminescence emission, and photoconductivity. These data will be interpreted through application of the principles of solid state electrolytic theory.

*Armour Research Foundation, Chicago, Ill.
SOLID STATE PHENOMENA IN THIN FILMS FOR COMPOSITE MOLECULAR ELECTRONIC MATERIALS. F. Schossberger. Project 4150(803), Contract AF 33 (616)-6445; WADD, ETL.

Thin films of electronic materials are being deposited by pyrolytic, vacuum evaporation and electrochemical methods and their electronic properties evaluated. These films include both inorganic and organic polymers incorporating such elements as silicon, boron, aluminum, phosphorous, sulfur, carbon, oxygen, hydrogen, and nitrogen. Research is also being initiated on the formation of single crystal films by the topochemical process.

*Electronic Tech. Lab., WADD, Dayton, Ohio.
SOLID STATE PHYSICS STUDY OF THIN FILMS. C. R. Barnes. Project 4150(802), Internal.

New techniques for depositing thin films of electronic materials are being developed and evaluated.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

The methods of deposition being employed include pyrolysis, vacuum evaportaion, sputtering and electrochemical formation. Emphasis is being placed upon those techniques which will deposit highly oriented single crystal films on substrates whose crystallographic structure has been previously selected to match that of the film under study. Particular attention will be given to the possibility of the formation of new compounds, for example, evidence has been shown to support the formation of a new compound, silicon phosphide, by high temperature pyrolysis. Efforts to deposit single crystal film compounds of the III-V Groups and of the IV Groups will be included. Interfacial effects of forming superimposed single crystal films will be investigated. Careful correlation between contractually produced films and internally produced films may lead to new concepts concerning the mechanisms of multiple thin film combinations.

*Revere, Althea, Vineyard Haven, Mass.
SOLID STATE RESEARCH AND PROPERTIES OF MATTER. A. Revere. Project 7021(802), Contract AF 33(616)-6604; WADD, MC.

The research on detailed replication techniques of surfaces and cross sectional planes of whiskers, thin filaments, and etch pits with the electron microscope will be continued. This may involve the adaptation of well known techniques or the development of new ones for the special problems arising with whiskers, etc. Emphasis will be on studies of the replication of voids, all surface imperfections, and screw or layer-type growth at the tips of whiskers grown under a variety of conditions. Whiskers of varying cross sectional shape will be sectioned and investigated at intervals along their length. The investigation will include the study of replication techniques for the study of tips of whiskers and cross sections close to the nucleation point.

*Massachusetts Inst. of Tech., Cambridge.
THEORETICAL AND EXPERIMENTAL RESEARCH IN THERMOELECTRICITY. D. C. White. Project 5621(802), Contract AF 19(604)-4153; ERD, EML.

This research is to conduct theoretical and experimental research in thermoelectricity which will lead to an accumulation of basic solid state knowledge which can then be drawn upon to scientifically select and prepare thermoelectric materials for specific application.

*New York Univ., N. Y.
THERMOELECTRIC MATERIALS FOR USE IN THERMOELECTRIC GENERATORS. I. Cadoff. Project 6692 (750), Contract AF 19(604)-3902; GRD, PL.

Electrical properties and thermal conductivities of alloys and semiconductors are being investigated to determine their applicability as thermoelectric materials.

*Electronic Material Sciences Lab., ERD, AFCRL, Bedford, Mass.
THERMOELECTRIC PHENOMENA. A. D. Johnson. Project 5621(802), Internal.

This effort includes an investigation of basic theory and experiments on thermoelectric phenomena. Preliminary measurements have been made on single crystal bismuth telluride, and improved equipment has been constructed for precision measurements of thermoelectric properties. These measurements are to be extended to low temperatures, and the effects of magnetic fields on transport properties are to be investigated.

*New York Univ., N. Y.
THERMOELECTRIC PROPERTIES OF INTERMETALLIC COMPOUNDS OF HIGH MEAN ATOMIC WEIGHT. I Cadoff. Project 7021(802), Contract AF 33(616)-3883; ARL, MCB.

The resistivity, thermoelectric power and Hall coefficient are being studied as a function of temperature. The energy gap and the electron and hole mobilities are also being measured. The data is being analyzed to obtain information about the intrinsic properties of the intermetallic compounds under investigation (barium telluride and lead telluride at present) and about the ionization energies of the impurities. The temperature range used is that between the melting point of the alloy and the boiling point of liquid air. The nature of the fall-off of the thermoelectric power with composition on both sides of the stoichiometric composition is being studied. Single crystals of these and other compounds will be used for these measurements if suitable ones can be produced.

*Pennsylvania State Univ., University Park.
UTILIZATION OF SOLID STATE PHYSICAL PHENOMENA. C. Volz. Project 4150(803), Contract AF 33(616)- 6029; WADD, ETL.

The current objectives are: (1) to continue the survey of conventional electronic circuits to determine what basic circuit operations exist; (2) to analyze the above electronic circuit functions and formulate workable mathematical expressions to describe them; (3) to categorize the specific operations into groups based upon similarities of the mathematical operations being performed and present this data in the form of a "handbook" of electronic circuit operations; (4) to continue research on mathematical techniques leading toward a systematic procedure of synthesizing molecular circuits; (5) to attempt to envision some specific, representative molecular electronic circuits and make some detailed calculations on the required material parameters, fabrication techniques, and possible performance to be expected; and (6) to construct models of these molecular circuits for experimental analysis of their characteristics.

*Georgia Inst. of Tech., Atlanta.
VACUUM EVAPORATED AND CATHODE SPUTTERED THIN FILMS. R. B. Belser. Project 4150(803), Contract AF 33(616)-6379; WADD, ETL.

The objective of the task is to perform basic research on the properties of thin metal or semiconductor films deposited by vacuum evaporation or cathode sputtering. Feasibility studies on the growth of single crystal films or highly oriented films shall be made. Methods of producing single crystal films during the deposition of the films shall be investigated as well as methods of inducing crystal growth by the recrystallization of films subsequent to deposition. Epitaxial growth on prepared single crystal planes used as substrates shall be studied. Methods similar to zone-melting and zone-refining methods shall be devised to induce recrystallization. Studying the parameters involved in the formation of thin films which possess superior electrical and mechanical properties is another objective. Finally the alloying of refractory metal films deposited by sputtering and of alloys of films of metal Group III/V Shall be studied under this task.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research. Fiscal Year 1960.

*Georgia Inst. of Tech., Atlanta.
VARIOUS PHENOMENA FOR THE PERFORMANCE OF CIRCUIT FUNCTIONS. E. J. Scheibner. Project 4150(803), Contract AF 33(616)-6028; WADD, ETL.

The scope of this effort is a program of basic research to catalog and investigate all solid state phenomena which might be utilized for the performance of electronic circuit operations in composite molecular electronic circuits and systems. The current objectives are: (1) to provide a comprehensive, annotated bibliography of the literature pertinent to composite molecular circuits; (2) to extend the survey of solid state, physical phenomena which might be utilized for the performance of electronic circuit operations; and (3) to perform a theoretical analysis and classification of these phenomena in various solid state materials based on energy band structure and the nature of the chemical bonding. This analysis will serve, furthermore, to focus attention on the possibilities of yet undiscovered phenomena in new materials.

Thermal Stresses

*Stanford Univ., Calif.
AERODYNAMIC HEATING OF AEROSTRUCTURES. N. J. Hoff. Project 9782(806), Contract AF 49(638)-223; AFOSR, DAS.

The work presently being conducted in the study of creep phenomena is directed toward explaining the phenomena of primary and secondary creep and includes consideration of creep recovery. Analytical techniques are being developed which show promise of ultimately explaining these phenomena in a more precise (and tractable) form than currently available. In the area of buckling of structural elements, detailed and rigorous mathematical theories for the calculation of the thermal buckling of shell type structures are being developed. The present work is limited to consideration of circular cylindrical shells and thin solid wedge-shaped wing sections. Consideration will be given to the thermal buckling of circular cones and spheres. In the area of heating equipment and testing techniques, the work is being directed toward establishing advanced structural test techniques to support theoretical accomplishments. Of particular interest are the techniques to study the onset of edge buckling of solid wedge-shaped wing sections.

*Technische Hochschule, Vienna, Austria.
AERODYNAMIC HEATING AND THERMAL STUDIES IN AIRCRAFT STRUCTURE. H. Parkus. Project 9783 (806), Contract AF 61(052)-214; AFOSR, DMS.

This research concerns the mathematical theory of thermal stresses in aircraft structures due to aerodynamic heating. The particular problems to be studied are elastic and visco-elastic thermal stresses in shallow conical shells and in triangular plates (Delta wings). Previous results of the investigator concern elastic thermal stresses in circular conical shells under axially symmetric temperature distribution. These are to be extended to shallow conical shells with arbitrary temperature distribution and of visco-elastic behavior. The equations of elastic conical shells of arbitrary cross section subject to temperature changes will be derived, as well as the equations of visco-elastic conical shells of arbitrary cross section using the "Maxwell body" model. The study of conical shells of zero curvature, i.e., triangular plate, will also be included.

*Technion Research and Development Foundation, Israel.
BUCKLING OF CONICAL SHELLS SUBJECTED TO THERMAL STRESS. J. Singer. Project 9782(806), Contract AF 61(052)-123; AFOSR, DAS.

This research covers theoretical investigation of the buckling of thin conical shells subjected to (a) uniform hydrostatic pressure and (b) circumferentially and axially varying temperature distributions resulting from aerodynamic heating. It is further intended to obtain solutions for the buckling of thin truncated conical shells using the differential equations derived by Seide which remove the restriction of small cone angles. An experimental study of the critical pressure of thin truncated shells under hydrostatic pressure will also be performed.

*Mechanics Research Branch, ARL, Dayton, Ohio.
INDUCTION HEATING OF STRUCTURES. R. B. Baird. Project 7063(806), Internal.

A 200 KW induction heater facility has been installed for the simulation of aerodynamic heating. This facility is capable of supplying high rates of heat input, in the order of 1000 BTU/sq. ft./sec., to small samples. An automatic temperature-time programming controller has been ordered. Preliminary tests have been conducted to study the characteristics of the heater, and to assist in the design of suitable work coils. Initial research studies will concern experimental verification of the buckling theories for thin and thick shells. Later studies will investigate the behavior of insulated structures subjected to ablative heating conditions. The facility may also be used for gas bearing research at elevated temperatures, and for vibration studies under steady or transient temperature conditions.

*Bell Aircraft Corp., Buffalo, N. Y.
MULTIWEB STRUCTURES AT ELEVATED TEMPERATURES. J. Padlog. Project 7063(806), Contract AF 33 (616)-6448; ARL, MRB.

Research is to be performed on the behavior of structures subjected to cyclic thermal stresses arising from aerodynamic heating. An analytical method will be derived for predicting structural behavior under many cycles of load and temperature. Theories will be derived for determining the elastic and inelastic strain history under specific heating and loading conditions. These theories will then be expanded further to include the cases of generalized load and temperature histories.

*Johns Hopkins Univ., Baltimore, Md.
PLASTIC WAVE PROPAGATION IN METALS AT ELEVATED TEMPERATURES. J. F. Bell. Project 9763(802), Contract AF 49(638)-423; AFOSR, DSS.

A new technique has been developed by this investigator for the study of plastic wave propagation in metals. It involves the cutting of a diffraction grating on the metal to be studied. Reflections from this grating are recorded as the piece is subjected to impact or to static deformation. It can also be applied to samples in high speed motion. The present investigation at elevated temperatures will use this technique to study the transient mechanical behavior of metals subject to impact. The characteristics of the plastic wave propagation will give information on the high

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

temperature mechanics of metals in terms of their fundamental properties.

*Polytechnic Inst. of Brooklyn, N. Y.
PLATES AND SHELLS UNDER EXTERNAL LOADING AND ELEVATED TEMPERATURES. J. Kempner. Project 9782(802), Contract AF 49(638)-302; AFOSR, DAS.

The principal purpose of this research program is to develop methods of analysis for stresses in and deformations of structural components of aircraft and missiles which are subjected to external loads and elevated temperatures. More specifically, the objective is to determine fundamental relations appropriate for the accurate analysis of plates, shells and bars subjected to the mechanical and thermal environments produced during high-speed flight, and to apply these relations to specific problems of basic interest. In the solution of such problems, new methods of analysis e.g., refinements in numerical methods, are also being sought. A considerable part of this work includes the determination of techniques for investigation of the heat-conduction problems which must be solved before stresses and deformations can be determined. In view of the fact that material properties are seriously influenced by high temperatures, studies of the effects of variable thermal and mechanical properties as well as the influence of plasticity and creep are included in the overall program.

*Florida Univ., Gainesville.
RESPONSE OF STRUCTURES TO RANDOM NOISE. W. A. Nash. Project 9782(806), Contract AF 49(638)-328; AFOSR, DAS.

The purpose of this investigation is to study theoretically the response of structural elements, curved panels in particular, when there is random acoustic excitation, atmospheric turbulence, and other sources of "noise" in addition to the normal aerodynamic loading. Several treatments of the response of certain structural elements to random loads are to be found in the literature. These studies are based upon the techniques of generalized harmonic analysis. Unfortunately, the pressure cross-correlations employed in the existing work are not entirely realistic. The present study will replace the unrealistic cross-correlations with a more realistic one corresponding to the situation existing in random acoustic excitation, atmospheric turbulence, and many other phenomena of interest in aeronautical design. The study also takes account of boundary conditions other than those already treated and even more important would treat, analytically, the response of curved panels to random noise. Also, the study treats, analytically, the effect of reinforcements on structural components subject to random noise and investigates the optimization of such reinforcement. Lastly, the investigation provides basic information regarding the characteristics of certain sound fields. This information is necessary to evaluate the reality of existing analyses. A small-scale experimental study of the statistical properties of the acoustic field near a siren or air jet is being made. It is expected that more detailed jet noise spectra than currently available will be obtained.

*Technische Hochschule, Hanover, Germany.
STABILITY AND ULTIMATE STRENGTH OF THIN WALLED COLUMNS. A. Pfluger. Project 9782(806), Contract AF 61(052)-365; AFOSR, DAS.

The research objective is to extend the original area of interest to include establishing the influence of preliminary local buckling on the failure of a column by Euler buckling. Moreover, the effect of temperature on column stability will also be investigated.

*Convair, San Diego, Calif.
THERMAL STRESSES IN PERFORATED PLATES AND BODIES OF REVOLUTION. R. D. Sutherland. Project 9782(806), Contract AF 49(638)-592; AFOSR, DAS.

The research being accomplished under this contract can be generally divided into two separate investigations: (a) the extension of the work begun on thermal stresses in singly perforated plates and experimental verification of analytical results, including an investigation of the visco-elastic case for simple configurations under thermal loading; and (b) the development and solution of the thermo-elastic equations derived for thick-wall, low drag shells of revolution subjected to hypothetical temperature gradients intended to approximate aerodynamically induced thermal loads. This last was evolved from the development of an ogive coordinate system through which configurations from a circular cylinder to a sphere can be expressed by variation of a single parameter.

*Pennsylvania State Univ., University Park.
TRANSIENT THERMAL STRESSES IN SOLIDS. W. Jaunzemis. Project 9782(806), Contract AF 49(638)-705; AFOSR, DAS.

The research objectives are to theoretically investigate transient thermo-elastic stresses such that the effects of thermal inertia of the structure can be taken into account. The statement of work also includes investigation of thermal stresses in elasto-plastic bodies and visco-elastic bodies.

*Massachusetts Inst. of Tech., Cambridge.
UNSTEADY AERODYNAMIC AND NONLINEAR STRUCTURAL PROBLEMS. H. Ashley. Project 9782(806), Contract AF 49(638)-160; AFOSR, DAS.

Theoretical research will be performed on the aerodynamic loading generated by unsteady supersonic flow over two and three dimensional wings, and further application will be made of variational principles to develop approximate solutions for finite deflections of non-orthogonally stiffened shallow shells with temperature-induced stresses and temperature-modified material properties.

Transitions & Dislocation Arrangement

*General Electric Co., Schenectady, N. Y.
BEHAVIOR OF NEARLY PERFECT CRYSTALS. P. D. Gorsuch. Project 7021(802), Contract AF 33(616)-6181; ARL, MCB.

A theoretical and experimental investigation is to be made of the dislocation structure of metal whiskers. The beginning studies shall be made on iron whiskers. The density and distribution of dislocations in "as-grown" whiskers shall be determined and correlated with the size and orientation of the whiskers. Etching techniques shall be developed which are capable of producing etch pits at the intersection of dislocation lines with the surfaces. Work

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

shall be done to establish the importance of dislocation introduced during growth upon the multiplication of dislocations and formation of glide bands under applied stress. Mechanical tests shall be made to establish the stress levels at which dislocations nucleate, grow and multiply in the whisker. X-ray studies shall be made as needed to determine the orientation of the individual whiskers and to reveal individual dislocations (Berg-Barrett technique).

*Alloyd Research Corp., Cambridge, Mass. DEPENDENCE OF BRITTLE-DUCTILE TRANSITION ON DISLOCATION ARRANGEMENT AND ITS INTERACTION WITH IMPURITY ATOMS. M. F. Connerford. Project 7021(802), Contract AF 33(616)-6454; WADD, MC.

Study of the dependence of the brittle-ductile transition on dislocation arrangement and its interaction with impurity atoms conducted on single crystals of vacuum, melted, zone refined, high purity iron. Measurement of dislocation array utilizing etch pit and X-ray techniques. Resistance measurements during tension tests shall be made to detect the first appearance of microcracks.

*Johns Hopkins Univ. Baltimore, Md. DESLIP IN ALUMINUM SINGLE AND BICRYSTALLINE SPECIMENS. R. B. Pond. Project 9761(802), Contract AF 49(638)-509; AFOSR, DSS.

Techniques have been previously developed for investigating the plastic behavior of metals during deformation by utilizing optical interferometric principles. These techniques dramatically illustrate the velocities of propagation of edge dislocations as a function of strain rate and crystal orientation in high purity metal crystals. The current experiments are intended to determine the role that time could play in the deslipping of a crystal. It seems probable that reversing the stress field imposed on single crystals during the time interval before strain hardening may set in would require deslip as the easiest mode of deformation. Since the interferometric cinemicrographic techniques lend themselves to an investigation of this type, these experiments are being used to determine accurately whether or not there is any directional hardening developed on the active slip band of the single crystal or whether there is a delay in developing such hardening. This investigation will supply some of the much needed information to support and further the dislocation theories of plastic deformation of metals.

*Manufacturing Labs., Inc., Boston, Mass. DISLOCATION AND PLASTIC BEHAVIOR OF IRON SINGLE CRYSTALS. P. Fopiano. Project 7021(802), Contract AF 33(616)-6348; WADD, MC.

It is the purpose of this research program to determine the vector geometry of dislocations in iron single crystals and to study the effect of this variable on observed plastic and strength properties. To date single bi and tri crystals of high purity iron have been grown and the quantitative specification of substructural parameters in the as-grown crystals by etch-pit and x-ray techniques have been initiated.

*Alloyd Research Corp., Cambridge, Mass. DISLOCATION AND SURFACE ENERGY EFFECTS ON THE MECHANICAL PROPERTIES OF COMPOSITE MATERIALS. R. A. Covert. Project 7023(802), Contract AF 33(616)-7091; WADD, MC.

The study of the formation and evaluation of composite materials, composed of alternate layers of extreme thinness of different metals, will be continued. The foils so formed may offer a material of greater strength since advantage may be taken of the combined dislocation and surface energy effects. Layer-thicknesses of 10^{-6} and 10^{-5} cm. theoretically represent a range in which interfacial surface energies of the order of magnitude of the elastic volume energy could develop and in which dislocations could be prevented from moving in the normal manner, respectively. The foils, constituted of silver and chromium and built up to a total thickness of several millimeters, currently are formed by the simultaneous vapor phase deposition of the two metals on a rotating copper disk. A foil of silver and iron will also be investigated. Perfection of the foil-fabricating techniques will permit specimens of composite materials, in the form of flat tensile strips, to be thoroughly examined in order to determine the mechanical and physical behavior pattern. The parameters of interest (from the experimental stress-strain curves) are Young's modulus, yield stress, the ultimate tensile strength, and the corresponding strains.

*Franklin Inst., Philadelphia, Pa. DISLOCATIONS IN METALS BY TRANSMISSION ELECTRON MICROSCOPY. H. G. F. Wilsdorf. Project 7024 (802), Contract AF 33(616)-6996; ARL, MCB.

A basic study of the motions and interactions of dislocations under unidirectional and reversed stress is being made using transmission electron microscopy techniques. Both tensile and fatigue specimens will be examined before and after deformation for dislocation movements and groups, patterns, cross-slip, vacancy production, and interactions of these with themselves or the parent structure. These studies will encompass a wide range of stresses, strains, and strain rates and are directed toward increasing our general understanding of the mechanisms of deformation under alternating stress.

*General Electric Co., Schenectady, N. Y. DISPERSION HARDENING IN ALLOY SYSTEMS. R. W. Guard. Project 7021(802), Contract AF 33(616)-6406; ARL, MCB.

A systematic study of dispersion hardening in two phase alloy systems will be conducted in order to determine the effects of dispersed particles on the deformation behavior and mechanical properties of metals and alloys. Etch pit techniques combined with high resolution electron microscopy and reflection X-ray microscopy techniques will be used in an effort to make direct observations of the interactions of dislocations with dispersed particles. The temperature dependence of particle hardening in the range of -195°C to 300°C will be investigated by means of the Stokes-Cottrell technique, strain hardening measurements, and by observations of slip-particle interactions as a function of temperature.

*Armour Research Foundation, Chicago, Ill. EFFECT OF A DISPERSE PHASE ON THE DUCTILE BRITTLE TRANSITION OF BODY CENTERED CUBIC METALS. R. H. Read. Project 7024(802), Contract AF 33(616)-6506; ARL, MCB.

The effect of a disperse phase on the ductile-brittle transition temperature of body centered cubic metals will be determined. A further theoretical treatment of the

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

equations proposed by Cottrell and others has indicated some simple relationships might exist for solid solutions containing dispersions. The existence of optimum values of grain size and dispersion of a hard second phase is indicated by this theoretical treatment and will be studied experimentally.

*Carnegie Inst. of Tech., Pittsburgh, Pa.
EXPERIMENTAL STUDY OF TRANSPORT PHENOMENA IN METALS. E. M. Pugh. Project 9760(802), Contract AF 49(638)-257; AFOSR, DSS.

The purpose of this work is to conduct an active experimental program designed to provide current and refined data which will check the predictions of existing theories of electron transport in metals and which will aid in reformulating these theories to give more accurate mathematical descriptions. The following types of experiments are being carried on over wide temperature ranges: (1) the measurement of the ordinary and extraordinary Hall effects; (2) the measurement of resistivities and magnetoresistance; (3) the measurement of magnetic susceptibilities; and (4) the measurement of thermoelectric powers. Metals being studied include high purity copper, ordered and disordered nickel-manganese alloys, nickel containing palladium, and pseudonickel.

*Illinois Univ. Urbana.
INTERACTION OF DISSOLVED INTERSTITIAL ATOMS WITH DISLOCATIONS. C. A. Wert. Project 7024 (802), Contract AF 33(616)-6176; ARL, MCB.

The work under this contract is an exploratory research program to determine if amplitude independent relaxation effects are present in body centered cubic metals containing interstitial solute atoms. Further, the effort will determine the possible effects of various interstitial solutes on such factors as the deformation peaks in Fe and the normal interstitial peaks in other body centered cubic metals.

*Illinois Univ. Urbana.
PHASE TRANSFORMATIONS AND DISLOCATIONS IN SOLIDS. T. A. Read. Project 9760(802), Contract AF 49(638)-420; AFOSR, DSS.

This investigation is concerned with experimental and theoretical research in the crystallographic features of diffusionless phase changes in metals. The objectives are to account for the diversity of habit planes and orientation relationships observed in martensite formation; in particular, the role played by crystal imperfections in the formation of martensite plates is being studied. As a phase of this study of imperfections, the transformation characteristics of iron whiskers has been undertaken. It is contemplated that the results of these investigations will contribute to a better understanding of the mechanism of the austenite-martensite transformation and the factors which affect it. Research is also to be conducted on a combined theoretical and experimental investigation of single dislocations in solids under both static and dynamic conditions. The experimental work is intended to elucidate the electrical properties of a dislocation, the process by which climb of a dislocation occurs, and the details of how a single dislocation moves under a force. The theoretical effort is intended to balance and complement the experimental work by treating problems such as the details of the "field" aspects of the dislocations and the details of the crystal or core effects and their consequences on the dislocation motion.

*Cambridge Univ. Great Britain.
STRUCTURAL INVESTIGATION BY X-RAY DIFFRACTION METHODS OF TRANSITION METAL ALLOYS. W. H. Taylor, P. J. Black. Project 9760(802), Contract AF 61(052)-50; AFOSR, DSS.

This research is providing information about the electron distribution in the alloys of the transition metals--iron, cobalt, nickel, copper, chromium, and manganese. Recent investigations of these metals have suggested that electrons which would normally be imbedded within the constituent atoms on the d-shell are really present in the "electron gas" between the atoms. Determination of the actual electronic structure of these metals will provide a basis for understanding their magnetic and mechanical properties. The approach in this research consists of measurement of the absolute intensity of x-ray diffraction patterns from selected alloy structures. Since the scattering of x-rays is due to the electrons, the precise determination of this scattering in diffraction affords a means for determining the electronic structure of the alloys.

*Illinois Univ. Urbana.
TRANSITION METAL ALLOYING BEHAVIOR OF THE TRANSITION METALS. P. Beck. Project 7021(802), Contract AF 33(616)-6770; ARL, MCB.

The work of Hume-Rothery, Raynor, and others has contributed substantially to the understanding of the electronic structure and atomic size of transition elements. In a parallel research program, this research is oriented towards the gaining of knowledge in the behavior of transition elements in alloys with the expressed purpose of developing theories of alloying behavior of the transition elements. This knowledge will be obtained through the measurement of physical parameters of transition element alloys. Specifically, temperature dependence of the specific heat of various solid solution alloys of body and face centered cubic types through the range of 1-5°K shall be investigated. X-ray diffraction studies will be used to ascertain the effects of composition on lattice parameters. The effects of ferromagnetic to anti-ferromagnetic changes on the electrical resistivity of several alloy systems based Cr, Mn, V, and Fe. Information obtained in these studies, together with data obtained by the Argonne National Laboratory on the low temperature bulk magnetic and atomic moment measurements, will, if possible, be used to arrive at conclusions as to the density of electron states in these materials and to any electron transfer that may occur.

*Franklin Inst. Philadelphia, Pa.
SUBSTRUCTURE AND DISLOCATION NETWORKS IN METALLIC CRYSTALS. M. Herman. Project 9760 (802), Contract AF 49(638)-821; AFOSR, DSS.

The verticle floating zone refining techniques for preparing high purity metal specimens developed under a previous contract will be used for the purification of iron, zinc and other structurally analogous metals such as W, Mo, V, Cr, Nb, Ta, etc. High purity metals will be studied from the viewpoint of interpreting the relationships between measured stress-strain properties and observed dislocation substructure networks. The specific problems deal with: (1) an attempt to treat body-centered

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960

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cubic transition metals as a class in an effort to better describe and predict their mechanical behavior; and (2) an attempt to quantitatively describe these transition metals in terms of current theory of imperfections in crystals. Investigations will be conducted of (a) dislocation networks in metallic single crystals, (b) generation of motion of dislocations under applied stress, (c) mechanical properties of metallic single crystals in terms of the observed networks of dislocations.

*Illinois Univ. Urbana

TIME AND TEMPERATURE DEPENDENCE OF THE DUCTILE-BRITTLE TRANSITION IN METALS. G. M. Sinclair. Project 7024(802), Contract AF 33(616)-5153; ARL, MCB.

A theory for crack propagation on a microscopic basis has been derived using dimensional analysis. Experimental verification is presently being obtained. The kinetics and energetics of the return of the yield point in molybdenum are being determined. These data will provide additional insight into another program wherein the mechanism of the fatigue limit in molybdenum is being studied. A study of the energy associated with the upper yield point in iron is presently being conducted. The equipment has been modified to allow tests to be conducted down to the temperature of liquid helium so as to investigate the anomalous brittle fracture strength of molybdenum.

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

PART IV

PROBLEMS IN MATERIALS TESTING AND APPLICATION

Adhesives

United States Rubber Company.
TR 5896, Part 1.
HIGH TEMPERATURE METAL-TO-METAL ADHESIVES. P. M. Elliott and W. C. Imholz. W33-038-ac-20810. July 1949.

Forest Products Laboratory. TR 5928.
GLUING TESTS WITH ROOM-TEMPERATURE-SETTING ADHESIVES TO FABRIC-BASE PLASTIC LAMINATES. Herbert W. Eickner. P. O. (33-038)-47-359, P. O. (33-038)-47-2902-E, P. O. (33-038)-48-11, P. O. (33-038)-49-180. August 1949.

United States Rubber Company.
TR 5896, Part 2.
HIGH-TEMPERATURE METAL-TO-METAL ADHESIVES. A. E. Smith, W. C. Imholz and P. M. Elliott. W33-(038)-ac-20810 (20050). July 1951.

Bloomingdale Rubber Company. TR 52-156.
IMPROVED STRUCTURAL ADHESIVES FOR BONDING METALS. H. C. Engel. AF 33(038)-21669. December 1952. PB 126 55. LC Mi $3.00, Ph $6.30.

Work under USAF Contract No. AF 33(138)-21669 has been directed toward the development of an improved structural adhesive for bonding metals. An adhesive designated as PA-101, developed and tested under this contract, meets most of the research objectives and so far as is now known conforms to the requirements of Specification 14164. The PA-101 formulation has been evaluated as a two-part, liquid adhesive.

Shell Development Company. TR 53-126.
ELEVATED TEMPERATURE-RESISTANT MODIFIED EPOXIDE RESIN ADHESIVES FOR METALS. M. Naps. AF 33(600)-6514. September 1953.

A metal-to-metal adhesive which is useful at temperatures up to 500°F has been developed. The adhesive, designated as Formulation No. 422, is a one-package system composed of EPON 1001 resin, a liquid phenolic resin, Plyophen 5023, and dicyandiamide as the curing agent. Aluminum dust is used as the reinforcing filler. The adhesive is cured at contact pressure and at elevated temperature (300°F).

Bonds to aluminum from adhesive Formulation No. 422 have a shear strength of 2100 psi at room temperature and 1400 psi at 500°F. After 200 hours aging at 500°F adhesive shear strength is mediocre (ca 200 psi). Bond strength is, however, 1000 psi after approximately 70 hours aging at 500°F. Aging the adhesive bonds for 200 hours at 400°F reduces the shear strength (at 400°F) from 1750 psi to 1340 psi. Cycling the adhesive bonds between room temperature and elevated temperatures (up to 500°F) for fifty times has had no apparent effect upon the bond strength.

Adhesive Formulation No. 422 is used as a pliable tape, preferably supported on a glass fabric carrier. The adhesive must be stored under refrigeration; storage life at 40°F is about one month.

Systematic variation of the concentration of the components of the adhesive led to the development of the following formula (parts by wt): 33 EPON 1001 + 67 Plyophen 5023 + 100 aluminum dust + 6 dicyandiamide. Higher EPON 1001 resin content decreased hot strength; higher phenolic content increased brittleness and decreased thermal resistance upon aging. Either lower amounts of filler or curing without dicyandiamide reduced adhesive shear strength, especially at room temperature.

The conclusions summarized above represent the status of the work at this writing, but since the work is continuing, these conclusions are only tentative. The adhesive 422 is considered an experimental product, and further work is indicated before the adhesive becomes a commercial product.

Battelle Memorial Institute. TR 54-88.
NONDESTRUCTIVE TESTING OF METAL-TO-METAL STRUCTURAL ADHESIVE BONDS. B. A. Kulp, M. E. Greenstreet and J. H. Cahn. AF 18(600)-124. April 1954. ASTIA Document No. AD 34417. PB 128 430. LC Mi $2.40, Ph $3.30.

A theoretical study of electrical characteristics of metal-to-metal adhesive bonded joints, with emphasis toward developing a satisfactory non-destructive test to determine quality of the bond, is presented.

To supplement the theoretical study, three types of experimental tests were conducted on adhesive bonded joints fabricated under close laboratory control. These tests were:

1. sonic - employing a "sonizon," a commercial type instrument.
2. capacitance - using an impedance bridge at 100 kilocycles.
3. d. c. resistance - using a standard ohmmeter.

The results indicate that more extensive research on electrical characteristics of adhesive bonded joints must be conducted to establish definite correlation to bond quality.

Bjorksten Research Laboratories, Inc.
WADC TR 53-294.
DEVELOPMENT OF ROOM-TEMPERATURE-CURING STRUCTURAL ADHESIVES FOR METALS. Johan Bjorksten, Risto P. Lappala, Luther L. Yaeger and Robert J. Roth. July 1954. PB 111 764 $2.25.

The work described herein has been directed toward the development of a room-temperature low-pressure curing metal-to-metal adhesive suitable for the fabrication and field repair of certain airframe structural parts where the use of heating and pressurizing equipment would be impracticable or impossible. An adhesive designated as P-262A, consisting basically of methacrylic acid and methyl methacrylate, has been developed which meets most of the research objectives and possesses properties comparable to those required by Military Specifications MIL-A-8331 (USAF). Its development, formulation, processing, and properties are presented. Further improvement lies basically in the lengthening of pot life and open assembly time toward which objectives further research and development work is contemplated.

Bloomingdale Rubber Company. WADC TR 52-156. IMPROVED STRUCTURAL ADHESIVES FOR BONDING METALS. H. C. Engel. AF 33(600)-23194. July 1954. ASTIA Document No. AD-35125. PB 126 5510. LC Mi $3.30, Ph $7.80.

The work of improving the structural metal-to-metal adhesive formulation that was previously developed under Contract AF 33(038)-21669 is presented. Shear strength test data of aluminum joints bonded with experimental modifications of the adhesive formulation and with experimental curing cycles and assembly procedures are listed. The strength data is the basis on which the conclusions are made as to the best formulation, its degree stability, and the optimum conditions for bonding which result in the best strength properties.

Shell Development Company. WADC TR 53-126. ELEVATED TEMPERATURE-RESISTANT MODIFIED EPOXIDE RESIN ADHESIVES FOR METALS, Pt. 2. M. Naps. AF 33(600)-6514. August 1954.

A metal-to-metal adhesive which is useful at temperatures from -70°F to 500°F has been developed. The adhesive, designated as Formulation 422, is one package system based on a combination of one part of a solid poly-epoxide, EPON 1001, and two parts of a liquid phenolic resin, Plyophen 5023. The resins are co-cured with dicyandiamide at contact pressure and at elevated temperature (330°F). Aluminum dust is the reinforcing filler and a small amount of a copper chelate compound of 8-quinolinolate or diethylene-triamine is used to improve the thermal stability of the adhesive.

Bonds to aluminum from Formulation 422 have a shear strength retention of about 75% at 500°F; tensile shear strength is 2150-2500 psi at room temperature and 1645 to 1850 psi at 500°F. This shear strength is about 1000 psi after aging the unstressed bond for 70 to 90 hours at 500°F and 600 to 800 psi after 200 hours. The long time load strength at 500°F is about 800 psi. Shear strength retention at 500°F after cycling the unstressed bond between room temperature and 500°F is approximately 65% after 100 cycles.

Adhesive Formulation 422 is used as a pliable tape preferably with a glass fabric carrier. The storage life of the film is three to five weeks at room temperature and eight to twelve months under refrigeration (30° to 40°F).

The adhesive is compounded by a holt melt process. Limited studies of a solvent process indicated that this technique may be more adaptable to large-scale work, but offers no improvement in the room temperature storage stability and bond performance of the adhesive.

Formulation studies included evaluation of a large number of phenolic resins, reinforcing fillers, and heat stabilizers as well as a few curing agents other than dicyandiamide. Maximum adhesive strength was obtained, however, with the adhesive components given above. Two phenolic resins, Lebec 102210 and Conolon 1007, may be substituted for the Plyophen 5023 with some sacrifice (about 10 to 20%) in shear strength. Asbestos is the most promising reinforcing filler, other than aluminum powder. No other adequate curing agent for the adhesive was found.

Connecticut Hard Rubber Company. WADC TR 54-98. INVESTIGATION AND DEVELOPMENT OF HIGH-TEMPERATURE STRUCTURAL ADHESIVES. H. N. Homeyer, Jr., J. H. Preston and K. L. McHugh. AF 33 (616)-427. August 1954. PB 111768. $3.00.

This report describes work on the investigation and development of structural adhesives from silicon materials for high temperature service conditions such as are encountered in certain aircraft operations. The results of lap-shear tests on aluminum-to-aluminum specimens bonded with a large number of silicone resins, rubbers, copolymers, and experimentally compounded mixtures are reported. The data presented shows that adhesives with considerable strength can be obtained and that the target objectives, although not yet reached, are not beyond the realm of possibility. It was found that shear strengths averaging over 2000 psi at 70°F, 700 to 900 psi at 300°F, and 400 psi at 500°F, can be obtained with unmodified resins.

Wyandotte Chemicals Corporation. WADC TR 53-477. SURFACE TREATMENT OF METALS FOR ADHESIVE BONDING. Robert S. Shane, Theodore L. Eriksson, Alexander Korczak and Dwight B. Conklin. AF 33(616)-2055. September 1954.

Preliminary studies have been made on the preparation of aluminum, stainless steel, and magnesium surfaces for adhesive bonding. The lap joint shear test at room temperature was used as the criterion. Tests on samples exposed to 95% relative humidity for 14 days and salt spray for 30 days are also included. A Dillon Universal Tester, as modified to provide motorization, pacing, and improved grips, is detailed. Bloomingdale FM-47 adhesive was used as the screening adhesive; other adhesives evaluated were Scotchweld No. 585 Tape, Shell EPON VIII adhesive, and Metlbond MN 3C tape. Eight other adhesives were examined in making a choice of screening adhesive. Approximately twenty different etchants were tried for stainless steel; a surface treatment is recommended as giving bond strengths substantially the same as those produced on aluminum using a modified Forest Products Laboratory surface treatment. In preliminary work on magnesium surfaces, eighteen etchant formulations were examined. Surface treatments AD and AE show mean shear strengths in excess of 2300 psi at room temperature without zinc chromate lacquer primer. A method for distinguishing between adhesive and cohesive failure is described.

WADC TR 54-153. EFFECT OF RATE OF LOADING ON SHEAR STRENGTH OF ADHESIVE-BONDED LAP JOINTS. R. E. Wittman. December 1954.

The effect of various rates of loading on the shear strengths of several metal-to-metal adhesives is presented. The loading rate ranged up to values as high as 10^8 psi per minute.

The shear strength for the more rigid adhesive types, vinylphenolic and epoxide, remained fairly constant at the increased rates of loading.

The shear strength of the less rigid types of

adhesives, nitrile rubber-phenolic and nylon-neoprene rubber-phenolic, increased appreciably with increases in loading rate and at the highest rate (0.002 second to failure) the strengths were more than doubled, the maximum increase being about 230 percent.

Strengthwise, the more rigid adhesives had higher values when stressed statically (600-700 psi per minute) than did the less rigid types. As a group, these values averaged 4110 psi and 2960 psi respectively.

Forest Products Laboratory. WADC TR 54-447. WEATHERING OF ADHESIVE-BONDED LAP JOINTS OF CLAD ALUMINUM ALLOY, Part 1. H. W. Eickner. PO 33(038)-51-4326 E. February 1955.

Lap-joint panels of clad 24S-T3 aluminum bonded with 5 metal-bonding adhesives were exposed to weathering at the Panama Canal Zone; Fairbanks, Alaska; Miami, Fla.; State College, N. M.; and Madison, Wis., with panels being removed for testing after 3 months and 1 year of exposure. Panels were exposed when stressed in bending and in the unstressed condition. Test panels were also exposed to several laboratory-controlled exposure conditions in addition to the weathering exposure.

The exterior exposure of panels for 1 year at the Panama Canal Zone has caused an appreciable deterioration in the quality of bonds with 2 of the 5 adhesives, and these same 2 adhesives have also shown some deterioration during 1 year of exposure at Miami, Fla. There was no deterioration in the bonds exposed for 1 year at the other exposure sites.

The adhesives showing deterioration were of the phenolic-neoprene-nylon and epoxy-resin types, with the deterioration of the phenolic-neoprene-nylon being the more drastic. The phenolic-neoprene-nylon bonds were deteriorated in both the stressed and unstressed condition while the epoxy-resin bonds were not seriously affected, except in the stressed condition.

Laboratory tests consisting of continuous exposure at 120°F and 97 percent relative humidity or a cyclic exposure involving the same temperature and humidity provided results that showed good correlation with the results obtained after exterior exposure in the Panama Canal Zone.

Bjorksten Research Laboratories, Inc.
WADC TR 53-294 (Sup 1).
DEVELOPMENT OF ROOM TEMPERATURE CURING STRUCTURAL ADHESIVES FOR METALS. Risto P. Lappala. AF 33(616)-165. April 1955. PB 111764s. $1.25.

A previous investigation on room temperature curing metal-to-metal adhesives (WADC TR 53-294) resulted in the development of an acrylic adhesive which met the specified requirements except for its short pot life and open assembly time. This report describes a supplemental investigation which was made in an attempt to improve these properties.

A large number of formulations were prepared using acrylic monomers and carboxylic acids which had not been investigated previously. The best adhesive, on the basis of shear strength screening tests of aluminum-to-aluminum bonds, contained MPL monomer, methacrylic acid, Acryloid B-82, and glass fibers sized with vinyl trichlorosilane. This adhesive did not completely fulfill the requirements, but used alone or preferably in combination with the best adhesive of the previous investigation, it allowed 1/2 hour or more of open assembly time. The pot life could be adjusted by varying the proportions of catalysts.

A two-part formulation prepared late in the program was the only adhesive to meet the low temperature shear strength requirement. Time did not permit full evaluation of this formulation.

The Connecticut Hard Rubber Company.
WADC TR 54-98, Part 2.
INVESTIGATION AND DEVELOPMENT OF HIGH TEMPERATURE STRUCTURAL ADHESIVES. Kenneth L. McHugh. AF 33(616)-2448. January 1956.

An epoxy modified silicone resin composition for use as a metal-to-metal adhesive has been developed which displays shear strength values slightly in excess of the target requirement of 1000 psi at 500°F, and shear strength values at room temperature of about 1100 psi. The highest shear strength value obtained at 500°F in an evaluation of seventy-seven commercial silicone and organo-modified silicone resins was 560 psi. A commercial organo-modified silicone resin produced a much higher shear strength at room temperature (average, 2300 psi), but a low shear strength at 500°F (average, less than 100 psi).

Experimental silicone resins were prepared which displayed shear strength values appreciably higher than those of the best commercial silicone resins. The best high-temperature shear strength values were found in resins having an R/Si ratio from 1.10 to 1.30 and a methyl content from 30 to 60 percent.

Epoxy-modified silicone resins were the most promising of almost one hundred modifications prepared with epoxy, alkyd, phenolic, and polyamine resins. Silicone-isocyanate copolymers, while not yet practical, showed interesting results. The Karl Fischer reagent was found useful for the determination of silanol groups in silicone resins.

Asbestine X and titanium dioxide fillers reinforced the silicone and organo-modified silicone resins to some extent. Glass fabric was of little or no value as a reinforcement.

The Connecticut Hard Rubber Company.
WADC TR 55-289.
ROOM TEMPERATURE VULCANIZING SILICONE ADHESIVE. Aldo J. DeFrancesco. AF 33(616)-2542. January 1956. PB 121 209 $1.75.

All commercially available adhesives recommended for bonding silicone rubber to aluminum and to itself without the use of heat and pressure have been evaluated. Dow Corning A-4000, a two-component adhesive, fulfilled nearly all target requirements. Two catalysts, Dow Corning XY-22 and XY-27, were tested with A-4000; of the two, XY-27 is recommended. It is suggested that the 24-hour peel strength requirement be lowered from ten pounds per inch to eight pounds per inch, which would enable the A-4000:XY-27 combination to meet all the specifications of the contract.

A study of other room-temperature-curing systems with various modified siloxanes is reported. With the use of several curing systems which were developed during the course of the work, materials were prepared for bonding silicone rubber to aluminum and to itself. Poor bonds were obtained to aluminum. Good bonds were obtained to silicone rubber with the use of a cement compounded from Linde Y-1170, a hydrogenmethyl silicone fluid.

A literature survey of silicone adhesives and room-temperature curing systems is also included in this report.

Shell Development Company. WADC TR 53-126. ELEVATED TEMPERATURE RESISTANT MODIFIED EPOXIDE RESIN ADHESIVES FOR METALS. Marguerite Naps, Frank C. Hopper. AF 33(616)-2430. March 1956.

A heat-resistant adhesive, designated Adhesive 422, developed under a previous contract, was more thoroughly investigated. Adhesive 422 is based on a mixture of EPON 1001 and Plyophen 5023, filled with aluminum dust. Attempts to obtain improved performance by changing various ingredients or proportions in the basic formula, or by

the addition of modifiers, were substantially unsuccessful. The surface preparation of the metal adherent and the use of primers were investigated. The properties of bonds to numerous adherents were determined.

More promising in resistance to aging at 500°F were new adhesives based on EPON X-15100.

U. S. Forest Products Laboratory. WADC TR 56-24.
PRELIMINARY INVESTIGATION OF LAMINATING TECHNIQUES FOR ALUMINUM SHEET MATERIAL. Fred Werren, B. G. Heebink. DO (33-616)53-20, Amend. A2(55-295). March 1956. PB 121336 $0.75.

The bonding of relatively large sheets of aluminum alloy to one another presents certain fabrication problems not experienced in the bonding of small areas. In an exploratory study of fabrication techniques, it was found that by varying the details of fabrication to suit the method of application and the specific adhesive, blister-free bonds of uniform quality could be made with all of the six commercial adhesives evaluated.

This report presents the results of preliminary investigations on the mechanical properties of 36- by 40-inch laminated panels consisting of 8 plies of 0.032-inch aluminum alloy. Five different adhesive systems were used to make the panels. Short-column edgewise-compression tests and flexural tests over short and long spans were run on the laminated material. The results show that, if the adhesive system used is rigid enough, the mechanical properties of the laminate, calculated on the basis of net area or net moment of inertia of the metal, are about equal to those of the same metal in solid form. If the shear modulus of the adhesive is low, buckling may occur at low loads under compressive edge loading, and excessive shear deflections may occur in flexure.

Some theoretical considerations concerning the behavior of laminated metal are presented in Appendix I.

Quantum, Inc. WADC TR 55-271.
RESEARCH ON ELEVATED TEMPERATURE RESISTANT INORGANIC POLYMER STRUCTURAL ADHESIVES. Harold H. Levine. AF 33(616)-2555. March 1956. PB 131934 $1.50.

Attempts to prepare inorganic metal-to-metal adhesives stable at 1000°F are described. The S-triazine and phosphonitrilic compounds were selected as the best approach because of their inherent thermal stability and reactivity.

Six promising products resulted from the following reactions: (1) melamine-phosphorus pentachloride (2) melamine-phosphorus oxychloride (3) melamine-phosphorus pentoxide (4) ammeline-phosphorus pentachloride (5) cyanuric trihydrazide-phosphorus pentachloride (6) carbohydrazide-phosphorus pentachloride. These products are stable from 750°F to red heat, are generally insoluable, and they possess very reactive atoms which may be capable of modifications resulting in adhesive properties.

Attempts to incorporate metal atoms into an adhesive precursor was successful with titanium.

Epoxy resins can be cured, without external heat, by use of phosphorus pentoxide.

Rigorous examination of the above six reactions is recommended. The effect of reaction conditions and reactant ratios, the degree of poly-functionality in starting materials, and the chemistry of the reactive atoms should be investigated to attain a desirable inorganic adhesive.

The Franklin Institute Laboratories for Research and Development. WADC TR 55-87.
TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING. Samuel N. Muchnick. AF 33(616)-2347. April 1956.

A theoretical basis is presented for the adhesion of a metal to an organic bonding agent. The contact angle is shown to be an effective measure of the adequacy of a surface treatment for a metal surface. For example, when a water drop makes a low or a zero contact angle on an aluminum or a stainless steel surface immediately following a surface treatment, joint strength is generally at a maximum and most likely to be reproducible. A study has been made of the importance of the film absorbed on the metal surface and of the mechanical properties of the metal as they are related to nominal joint strength. Four different adhesives have been used to evaluate the surface treatments which result in the highest joint strengths. In addition, those procedural variables are investigated which might affect surface properties and joint strength.

Stanford Research Institute. WADC TR 54-231, Part 4
DEVELOPMENT OF NON-DESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS. J. S. Arnold. AF 33 (616)-2035. June 1956. PB 121495 $1.25.

An ultrasonic technique for the evaluation of structural adhesive bonds (The STUB-meter) is being developed. The behavior of a ferro-electric transducer, when mechanically coupled to a test speciman, is affected by the structural properties of the test speciman. The design and construction of a portable laboratory model of the STUB-meter are described, and a plausibility argument for the existence of a relationship between bond strength and the STUB-meter behavior is presented. An evaluation program that will be carried out in cooperation with organizations in the aircraft industry is described.

The Connecticut Hard Rubber Company. WADC TR 54-98.
INVESTIGATION AND DEVELOPMENT OF HIGH-TEMPERATURE STRUCTURAL ADHESIVES. Alfred S. Kidwell, Kenneth L. McHugh. AF 33(616)-2448. September 1956. ASTIA Document No. AD 97287. PB 121657.

Wide variations in the composition of the CHR-M-60 epoxy-modified DC-2103 silicone resine with Asbestine X filler, which has shown shear strength values as a metal-to-metal adhesive in excess of 1000 psi at 500°F, have been made and the results plotted to indicate composition areas yielding maximum shear strength values. Extended high-temperature aging tests have shown DC-803 silicone resin ard epoxy-modified DC-803 to have better aging resistance than DC-2103 and epoxy-modified DC-2103, respectively. The DC-803 showed little loss in shear strength at 500°F after aging 1200 hours at 500°F on both aluminum panels and stainless steel panels (residual shear strength about 300 psi at 500°F). This resin also withstood 300 hours at 600°F on stainless steel and 200 hours on aluminum. The 200 percent epoxy-modified DC-803 lasted 800 hours at 500°F and 100 hours at 600°F (residual strength in each case about 350 psi at room temperature and 150 psi at 500°F).

A number of silicone-epoxy-phenolic resin blends were prepared, which showed shear strength values and high-temperature aging resistance slightly better than those for the epoxy-modified silicone resins.

Up to 20 percent of epoxy resin in epoxy-modified DC-2103 silicone resin was found to have relatively little effect on the change in shear strength values when tested over a temperature range from -70°F to 900°F; the epoxy resin mainly improved the strength over the lower half of

the temperature range. Shear strength at 900°F was about 100 psi.

Manuscript released by author August 1957 for publication as a WADC Technical Report.

University of Illinois. WADC TR 55-491. RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES. Richard M. Spriggs, Henry G. Lefort and Dwight G. Bennett. AF 33 (616)-2556. September 1956. ASTIA Document No. AD 97316. PB 121 659.

Twenty-three ceramic adhesives of the ceramic-oxide, glassy-bond type, including six commercially available enamel frit compositions, were evaluated during the contract period. Evaluation was based, primarily, on the results of room and elevated temperature shear strength tests. Best results were obtained with ceramic adhesive UI 117-50. When used with 28 mesh stainless steel screen as a carrier, this adhesive gave shear strengths of up to 3680 psi when tested at 800°F. Twelve ceramic adhesives of the cermet type were tested in shear at room and elevated temperatures. Of these, only the adhesive which contained 30% metallic cobalt incorporated in a lead borosilicate glass matrix appeared to offer promise. Thirteen ceramic adhesives of the air-setting temperature-resistant type were also evaluated. Adhesive A-1 was the only air-setting adhesive which approached the target property of 1000 psi at 1000°F, giving shear strengths of up to 1400 psi at 1000°F after a selected curing treatment to 600°F. Its resistance to moisture, however, was quite low. The oxychloride and oxysulfide adhesives investigated developed nominal shear strengths of 200 to 250 psi at room temperature and maintained these strengths to an upper temperature limit of 600°F.

Forest Products Laboratory. WADC TR 56-239. TENSILE STRENGTH OF ADHESIVE BONDS IN SANDWICH WITH ALUMINUM FACINGS AND ALUMINUM HONEYCOMB CORES. V. C. Setterholm, H. W. Eickner, E. W. Kuenzi. AF 33(616)-56-9. September 1956. ASTIA Document No. AD 97292.

Results of tensile tests of eight adhesive systems for bonding aluminum alloy facings to aluminum honeycomb cores of various cell sizes and foil thicknesses are presented. Analysis of these results shows that the tensile strength of bonds to honeycomb cores with any one adhesive system was found to be mainly dependent on the available fillet length.

Seven of the adhesive systems were adequate for sandwich with cores of 0.001-inch 3003H19 aluminum alloy foil, and 2 adhesives were adequate for sandwich with cores of 0.002-inch foil. None of the adhesives were adequate for producing maximum facing stresses in sandwich with cores of foil thicker than 0.002 inch. Five of the adhesives were strong enough to cause 0.001-inch foil to fail in tension, but none were strong enough to cause failure in thicker foils. Good bonds were not obtainable with one tape adhesive used on cores with cutting burrs on the cell wall ends unless an adequate liquid primer was applied to the core.

Quantum, Incorporated. WADC TR 55-271, Part II. RESEARCH ON ELEVATED TEMPERATURE RESISTANT INORGANIC POLYMER STRUCTURAL ADHESIVES. Harold H. Levine. AF 33(616)-2555. November 1956. ASTIA Document No. AD 110588. PB 121 908.

This report describes the progress achieved toward the development of an elevated temperature resistant inorganic polymer structural adhesive.

Increasingly severe thermal operating conditions have been engendered by high aircraft speeds. It is necessary to develop a structural adhesive that can satisfactorily withstand temperatures up to 1000°F.

An inorganic adhesive stable at 800°F, with a room temperature shear strength of 285 lb./sq. in. has been obtained from the ammeline-phosphorus pentoxide reaction product.

The use of titanium dioxide as a primer and dimethylformamide as an extractive solvent enabled the above shear strength to be obtained from an original value of 65 lb./sq. in.

Evidence was obtained to indicate that the adhesion was a result of chemical interaction between the adhesive and the stainless steel surface.

The success of the research to date indicates the necessity of further work to increase the adhesive and flexural strength by chemical methods. This should be done without sacrificing the present heat resistance and preferably with increasing the present thermostability.

University of Illinois. WADC TR 55-491. RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES. Henry G. Lefort, Richard M. Spriggs and Dwight G. Bennett. AF 33(616)-2556. January 1957. ASTIA Document No. AD 110736. PB 121 941.

The study of ceramic compositions as adhesives was activated due to the need for materials to act as adhesives for metals at elevated temperatures and to possess high shear strengths for extended periods of time at such elevated temperatures.

The object of the investigation was to develop high temperature resistant structural adhesives for type 301 and 302 stainless steel or other desired alloy metals, particularly 17-7 PH, from ceramic-oxide, glassy-bonded coatings, cermets with sintered metal bonds, air-setting, temperature-resistant silicates, aluminates, oxychlorides, oxysulfides, and ceramic-oxide, resin-bonded coatings.

Twenty-four ceramic adhesives of the ceramic-oxide, glassy bond type, including three commercially available porcelain enamel ceramic adhesives, were evaluated for use at room and elevated temperatures. Evaluation was based on shear strength tests using type 302 stainless steel. Several of these ceramic adhesives were used to bond small type 17-7 PH stainless steel honeycomb sandwiches. Type 17-7 PH stainless steel was evaluated for shear strength using ceramic adhesive UI 117-50. Various methods of specimen and ceramic adhesive preparation and fabrication were investigated for increase in shear strength including knurled overlap areas, mechanical scouring of overlap areas, chemical pickling and nickel plating of overlap areas, and the use of various foils and screens as carriers. Stainless steel screen of 325 mesh when used as a carrier gave an average shear strength of 1160 psi at 800°F. Copper screen of 60 mesh gave an average shear strength of 1180 psi at 800°F when used as a carrier. Porcelain enamel ceramic adhesives of thermal expansions approaching that of ingot iron shear specimens generally gave the highest shear strengths. Resorcinol-formaldehyde-Portland cement adhesives did not show great promise as adhesives, especially at elevated temperatures. Stress rupture determinations using ceramic adhesive UI 117-50 on type 302 stainless steel shear strength specimens yielded results of 700 psi for 171 hours with no failure, and 800 psi for 22 hours with failure at room temperature. At 600°F, the adhesive withstood 600 psi for 500 hours without failure.

Stanford Research Institute. WADC TR 54-231. DEVELOPMENT OF NON-DESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS, Pt. 5. J. S. Arnold. AF 33(616)-2035. February 1957. ASTIA Document No. AD 118-83. PB 131046. Order from Office of Technical Services. $1.75.

An ultrasonic technique for the evaluation of structural adhesive bonds (the STUB-meter) is being developed and tested. The operation of the instrument is based on the empirically observed fact that the behaviour of a ferro-electric transducer, when mechanically coupled to a test specimen, is affected by the structural properties of the test specimen. To define more closely the scope of the technique, an evaluation program is being carried out in cooperation with organizations in the aircraft industry. Initial data have already provided improved means of choosing optimum frequency ranges. Laboratory development of the STUB-meter has included work on probes for curved surfaces and for standard lap shear specimens; electrodes with improved wear resistance; visualization of vibration modes; effects of loading; and improved circuitry.

WADC TR 56-467
EFFECTS OF NUCLEAR RADIATION ON STRUCTURAL ADHESIVE BONDS. Robert S. Arlook, 1/Lt, Douglas G. Harvey, 1/Lt. February 1957. ASTIA Document No. AD 118063.

Nine commercially available metal-to-metal adhesives were investigated to determine the effect of gamma radiation upon their strength properties. Standard 2024-T3 alclad aluminum alloy lap joint speciments were prepared, and three series of exposures were made at the MTR gamma facility. The approximate dosages were 9×10^7, 3×10^8, and 9×10^8 roentgens. Tensile shear and bend tests were conducted at room temperature, at 180°F, and at 260°F. Two adhesives were also tested at 500°F. Fatigue tests were performed at room temperature.

Only one adhesive was essentially unaffected by gamma radiation at all testing temperatures. The remaining adhesives were affected to varying degrees.

Narmco, Incorporated. WADC TR 56-533 ELEVATED TEMPERATURES RESISTANT SILICONE STRUCTURAL ADHESIVES FOR METALS. Frank J. Riel, Jr., M. Bruce Smith. AF 33(616)-3007. March 1957. ASTIA Document No. AD 118153. PB 131 024.

During the period covered by this report, two possible methods of approach to the synthesis of a new silicon containing polymer suitable for a high temperature adhesive were investigated.

The first type of materials investigated, polymers made from polyhydroxy compounds and halosilanes, proved to be too susceptible to hydrolysis to be useful as adhesives.

The second line of approach involved the synthesis of polysiloxane and polysilane resins containing tolyl groups, followed by oxidation of the tolyl groups to polar carboxyphenyl groups. A number of such resins were prepared, identified, and evaluated.

Evaluation of the carboxyphenyl polysilioxanes showed them to be better high temperature resistant laminating resins than high temperature resistant metal-to-metal adhesives when compared with conventional commercially available silicones. Ultimate flexural strengths of fiber glass reinforced laminates obtained from the system as a whole, were 36,300 psi tested at room temperature and 16,700 psi tested at 500°F. Ultimate tensile shear values of aluminum-to-aluminum bonds, also obtained from the system as a whole, were 1,322 psi tested at room temperature and 632 psi tested at 500°F.

Heat degradation, of laminate flexural strengths and metal-to-metal bond strengths, was especially evident in tests at R. T. after exposure to 500°F, but not so evident in tests at 500°F after exposure to 500°F.

Little effort was expended in the study of carboxyphenyl polysilanes.

Forest Products Laboratory. WADC TR 56-650 DEVELOPMENT OF METAL-BONDING ADHESIVE WITH IMPROVED HEAT RESISTANCE. J. M. Black, R. F. Blomquist. DO 33(616)-56-9. April 1957. ASTIA Document No. AD 118193. PB 121 856.

Studies were conducted on an experimental phenol-epoxy resin tape adhesive, FPL-878, to determine the effects of glass and asbestos supporting mediums and of inorganic fillers on the resistance of bonded aluminum joints to aging at 500°F.

Aluminum-to-aluminum bonds with a tape adhesive employing a supporting medium of either glass cloth No. 112 or No. 120 and with either a heat cleaned or Volan A finish were superior in resistance to aging at elevated temperatures to all other fabrics evaluated. The most promising of the asbestos materials was a felt product, Pyrotex No. 9526-RBI. All lap-joint bonds made with tape adhesive formulations, however, were lower in resistance to thermal aging than bonds made from the liquid adhesive.

Tests on antimony trioxide used as an inorganic filler in tapes of both FPL-878 adhesive and a straight epoxy resin adhesive showed this material was the best of the filler materials tested for resisting thermal degradation and also contributed to increase joint strength.

Koppers Company Inc. WADC TR 56-410 RESEARCH ON ROOM TEMPERATURE CURING STRUCTURAL ADHESIVES FOR METALS. Roy H. Moult, William E. St. Clair. AF 33(616)-2862. April 1957. ASTIA Document No. AD 118203. PB 131059.

A study has been made on the formulation of room temperature, low-pressure cured metal-to-metal adhesives, based on the use of glycidyl ethers of resorcinol resins. Various formulations have been found which meet all of the primary strength requirements for tensile shear strength at various temperatures, bend strength, creep-rupture strength and fatigue resistance.

Resistance to salt spray and soaking in water has been tested at Wright Air Development Center on one formulation and found to be unsatisfactory for the former but excellent with the latter.

The preferred formulation resulting from this study have been those with pot lives of one to three hours as obtained by use of solid diamines as curing agents. Longer pot lives of four hours or more have been obtained, but only with the sacrifice of physical strength characteristics.

The Glenn L. Martin Company. WADC TR 56-320 RESEARCH ON STRUCTURAL ADHESIVE PROPERTIES OVER A WIDE TEMPERATURE RANGE. H. R. Merriman. AF 33(616)-2620. April 1957. ASTIA Document No. AD 118228.

The widespread increased use of adhesive bonded construction in military aircraft requires that the mechanical properties of adhesives be determined over a wide temperature range in order to determine the useful operating temperature range.

The mechanical properties of adhesive bonded aluminum joints were determined using standard test procedures over a range of temperatures from -100°F to 800°F.

The effect of exposure at test temperature for various periods of time was also determined. The effect on the properties of using a different adherend was determined by duplication of some of the test conditions on stainless steel lap joint specimens. Nine general purpose adhesives, AF-6, PA-101, Plastilock 608, Metlbond 4021, FM-47 Liquid, FM-47 Film, Redux E (Type R), Cycleweld 55-20 and EPON VIII; and two high temperature adhesives, Shell 422 and HT-20, were tested.

The program was confined to the testing of standard specimens, bonded in accordance with exact procedure specified at the beginning of the contract by the adhesive manufacturers to obtain the optimum properties over a wide temperature range. Tensile shear, creep rupture, tensile, impact, bend, cleavage, fatigue and peel tests were conducted.

Lap specimens of general purpose adhesive decreased in strength after 1/2 hour exposure at test temperature compared to test values obtained immediately after reaching test temperatures. Further exposure in general produced increased strength over the initial values obtained for the bonded joints.

Shell Development Company.
WADC TR 53-126 Part IV
ELEVATED TEMPERATURE RESISTANT MODIFIED EXPOXIDE RESIN ADHESIVE FOR METALS. Marguerite Naps. AF 33(616)-3347. July 1957. ASTIA Document No. AD 130884.

Metal-to-metal adhesives which are useful at temperatures up to 500°F, primarily on stainless steel, have been studied. The most promising formulations are based on a solid polyepoxide designated as Experimental Resin X-131. This material is used on organic solvents for the preparation of supported adhesives tapes with a glass fabric carrier. The adhesives are cured at elevated temperature (330°F) and usually at 100 psi. Bonds to type 301 steel are characterized by brittleness and mediocre reproducibility.

An adhesive (Formulation No. 1236) based on combination of Experimental Resin X-131, EPON[a]) 1009 and Formvar 15/95 E (polyvinyl formal) appears to have the best thermal stability on aging at 500°F of any of the formulations investigated. The adhesive contains aluminum dust as the reinforcing filler and is cured with diaminodiphenylsulfone. Shear strength at 500°F on 301 steel is 1200 psi after 200 hours exposure at 500°F and about 800 psi after 400 hours exposure at 500°F. Strength retention on rapid heating is mediocre, however. The shear strength decreased from about 4000 psi to 530 psi as the test temperature is increased from room temperature to 500°F. Postcuring the bonds at 400°F improves the hot strength slightly to about 800 psi.

Exploratory investigations of adhesives based on ten new experimental resins were unsuccessful for the development of a formulation which is superior in heat resistance to Formulation No. 1236. Several promising paste-type and hot melt adhesives comparable in bond performance to X-131 resin adhesive tapes have been evaluated briefly. The new experimental resins are similar to Experimental Resin X-131 in brittleness and are characterized by a slow rate of cure with conventional curing agent systems.

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Forest Products Laboratory.
WADC TR 54-447 Part II
WEATHERING OF ADHESIVE-BONDED LAP JOINTS OF CLAD ALUMINUM ALLOY. H. W. Eickner. DO 33(616)-56-9. July 1957. ASTIA Document No. AD 130879.

Lap-joint panels of clad 2024-T3 aluminum bonded with nine metal-bonding adhesives were weathered at Panama Canal Zone; Fairbanks, Alaska; Miami, Fla.; State College, N. Mex.; and Madison, Wis. Panels were exposed while stressed in bending and while unstressed. Test panels were also exposed to several laboratory conditions.

Three of the adhesives (2 epoxy resins and a phenolicneoprene-nylon) were deteriorated seriously at the Canal Zone and Florida. Exposure at the other 3 sites was much milder, and only some stressed epoxy-bonded panels failed at these sites. Through 1 year, conditions at the Canal Zone caused greater bond deterioration than those in Florida. After 3 years, however, the deterioration was generally worse in Florida, and this exposure caused 2 other adhesives (nitrile-rubber type) to deteriorate. Stressing the bonded panels over bending frames generally accelerated the deterioration of those adhesive bonds that were subject to deterioration.

Laboratory exposure to salt-water spray or exposure to continuous or cyclic conditions of 120°F and 97 percent relative humidity promoted deterioration in the same types of bonds that deteriorated from weather at the Canal Zone or Florida sites.

In general, the adhesives that performed well in these exposure tests were types that have qualified under the applicable Military specification, while those that showed appreciable deterioration have not.

The Franklin Institute.
WADC TR 55-87 Part III
TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING PART III. Contact Angle and Contact Resistance Devices for Quality Control. Richard H. Hollinger. AF 33(616)-2347. September 1957. ASTIA Document No. AD 131093.

Two instruments have been developed for quality control use to determine whether surfaces of metals have been adequately prepared for adhesive bonding. The contact resistance apparatus measures the break-through voltage of a surface film at a given voltage. The contact angle apparatus measures the angle of contact between a drop of water and the specimen surface.

WADC TR 57-513
STRUCTURAL ADHESIVES AND SANDWICH CONSTRUCTIONS WADC - UNIVERSITY OF DAYTON JOINT SYMPOSIUM. Theodore J. Martin. November 1957. ASTIA Document No. AD 142152.

This report is a compilation of papers presented at the Wright Air Development Center - University of Dayton joint symposium on Structural Adhesives and Sandwich Constructions, 12-13 June 1957, Dayton, Ohio. The papers, for the most part, reviewed work done on Materials Laboratory research and development contracts.

University of Illinois. WADC TR 55-491 Pt III. RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES. Ricahrd M. Sprigge, Charles N. Williams, Henry G. Lefort, Dwight G. Bennett. AF 33(616)-2556. Dec. 1957. ASTIA Document No. AD142226.

The object of the investigation was to develop high temperature resistant structural adhesives for Type 301 and 302 stainless steel or other desired alloy metals, particularly Type 17-7 PH, from ceramic-oxide glassy-bond coatings, cermets with sintered metal bonds, air setting, temperature-resistant silicates, aluminates, oxychlorides, oxysulfides, and ceramic-oxide resin bonded coatings.

Nine new ceramic glassy-bond adhesives developed to mature within the relatively low temperature range of 1300-1500° F, gave only nominal shear strengths of 530 psi at 600° F and 750 psi at 800° F.

Glassy-bond ceramic adhesive UI 117-50, when matured at 1750° F during the hardening treatment RH 950 for 17-7 PH stainless steel, gave average (two to four specimens) shear strengths ranging from 990 psi at room temperature to 980 psi at 1000° F. The curve was quite flat through four of five temperature points. It reached a high of 1750 psi at 900° F.

Glassy-bond ceramic adhesives UI 1067-3 and UI 1067-4, containing 10 and 20 per cent carbonyl iron, respectively, both developed more than 1500 psi at room temperature and at 1000° F, with higher values at 600 and 800° F. Their shear strength-temperature curves were quite flat with all values being within about 10 per cent of the average values which were slightly above 1700 psi.

The advantageous use of carbonyl iron (20%) and silicon powder (10%) is shown with ceramic adhesive UI 117-63 which gave an average shear value of 1455 psi at four temperature points (room to 1000° F). The highest value was 1505 psi at room temperature. The lowest was 1415 psi at 800° F. All of the values were within about three per cent of the average.

Adhesive bonded specimens subjected to the severe environment of 100 hours immersion in boiling water, in general, lost strength but one adhesive (UI 117-59) containing 10% carbonyl iron and used with a 28 mesh stainless steel carrier actually showed an increase in strength after immersion in boiling water.

Other specimens, both with and without additions ov carbonyl iron, were subjected to 143 hours of heating at 1000° F plus 25 thermal shock cycles between room temperature and 1000° F. Their shear strengths were improved rather than harmed by this treatment.

The great potential usefulness of metal powders in ceramic adhesives is shown by UI 117-64 which contains 30 per cent carbonyl iron. This adhesive, although it fell off sharply on either side, developed an average of more than 5600 psi at 800° F.

In the field of air-setting or low temperature cure adhesives it appears that those of the aluminum phosphate type offer some promise.

Tests conducted with air-setting adhesives indicate that their moisture sensitivity can be reduced through the use of a dense adhesive structure, or by the addition of a sealant such as tetraphenyltin.

The Martin Company. WADC TR 56-320 Pt II. RESEARCH ON STRUCTURAL ADHESIVE PROPERTIES OVER A WIDE TEMPERATURE RANGE. H. R. Merriman, H. L. Goplen. February 1958. ASTIA Document No. AD 150967 PB 131 711.

Time-deformation creep curves were obtained for a number of adhesives using specified test temperatures and stress levels which resulted in time-to-rupture life in the 100-to-200-hour range. The stress levels used in this project were determined from information obtained from work reported in WADC TR 56-320.

The adhesives tested were AF-6, PA-101, Plastilock 608, Metlbond 4021, FM-47 Liquid, FM-47 Film, Redux E (Type R), Cycleweld 55-20, Shell 422, and HT-424. Two metal alloys were used, 2024-T3 clad and bare aluminum alloy and Type 301, 1/2 hard stainless steel.

The tests were conducted on 1/2-inch lap coupons, at three different temperatures for each adhesive. Stress was applied by dead weight loading. The deformation of the glue line was measured by observing the displacement of three fine scribe lines across the vertical edges of the lap joint. A Gaertner 100-power microscope with a bilfilar micrometer eyepiece and 10X objective lens was used to measure deformation of the glue line.

The results obtained in these tests indicated that the method of measuring creep deformation by observing scribe lines through a microscope, properly controlled, provides a reproducible test procedure. It was noted that each specific adhesive had an approximately constant total deformation and creep rate for the three tests temperatures and corresponding stress levels.

The Franklin Institute. WADC TR 55-87 Pt II. TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING. Samuel N. Muchnick. AF 33(616)-2347. February 1958. ASTIA Document No. AD 150989.

A preliminary investigation of the effectiveness of mild acidic or basic solutions of wetting agents for treating metal surfaces prior to adhesive bonding shows that the strong acid solutions presently in use may possibly be replaced by these milder solutions. Joint strengths approximating the values obtained with strong acid solutions have been obtained with aluminum, stainless steel, and titanium. The pH, concentration of components, and ionic type of wetting agent are specific for each type of metal.

Additional correlative tests were developed for evaluating surface treatments. The interfacial contact angle between oil and water on the treated surface shows a greater sensitivity than the contact angle in air, and makes it possible to differentiate between two treatments that give zero contact angles against water. Contact resistance and hydrogen overvoltage measurements can be definitely correlated with the contact angle measurements. The contact resistance measurement which also evaluates the effect of oxide thicknessappears to be a useful instrument for industrial application.

A study of the role of the adsorbed film on adhesive behavior indicates that the more reactive metals, aluminum and magnesium, are more sensitive to differences in the chemical structure of the film. There is also an indication that these films may affect the adhesional characteristics of some type of bonding agent.

The temperature of the rinse water following treatment of aluminum is shown to be important in the adhesional characteristics of the metal. Reproducibility and specificity of attractive forces between adhesive and metal were investigated, and it was apparent that the material and procedural parameters must by studied along with specific affinities in an adhesive system.

The Franklin Institute. WADC TR 55-87 Pt IV. TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING. Harry G. Bickford. AF 33(616)-2347. February 1958. ASTIA Document No. AD 150993.

The scope includes the development and evaluation of mild surface treatments for clad aluminum alloy, stainless steel, and titanium alloy and investigations of the critical factors involved in the treatment of magnesium alloy. Methods of evaluating the degree to which a surface has been cleaned are discussed with regard to the ease of conducting the tests and with regard to their reliability. Effects of electrical potentials, strong alkali and acid

exposure of treated surfaces, and time delays between treatment and priming of the surfaces on bond strength are subjects of discussion. Results of radioactive-tracer work using isotopic chromiun are used to lay a basis for a theory of bonding.

The Franklin Institute. WADC TR 55-87 Pt V. TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING. Edmund Thelen, Richard Hollinger, Thomas I. Haigh, Jr., James B. Drew, Charles J. Varker, Wallace E. Frank. AF 33(616)-2347. February 1958. ASTIA Document No. AD 150998.

The scope includes analysis of strong and mild surface treatments for clad aluminum alloy, stainless steel, and titanium alloy and of the best treatment for magnesium alloy. Reaction of bonded surfaces to sand and vapor blast treatments is analyzed. The resistance of the treatments to salt spray are evaluated. Field-repair treatments are discussed and results of tests tabulated. Quality control instrumentation is outlined. Application of d-c potentials to bonded surfaces was determined to be deleterious. Radio-tracer studies using isotopic chromium provided valuable information regarding the role of chromiun ions in metal surface preparation. A means of providing continuous indication of bond strength during the life of the plane is suggested.

The Franklin Institute. WADC TR 55-87 Pt VI. TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING PART VI. Summary of Methods. Richard Lindsay, Jr. AF 33(616)-2347. February 1958. ASTIA Document No. AD 150999.

Methods for surface preparation or stainless steel, titanium, aluminum, and magnesium for adhesive bonding are contained in this summary. The treatments are those that were found to be most promising when used with certain adhesives.

Quantum, Incorporated. WADC TR 55-271 Pt III. RESEARCH ON ELEVATED TEMPERATURE RESISTANT INORGANIC POLYMER ADHESIVES. Harold H. Levine. AF 33(616)-3824. April 1958. ASTIA Document No. AD 151107.

Attempts to prepare heat stable materials for formulation with the ammeline-phosphorus pentoxide inorganic adhesive are described. The two approaches deemed as most promising were epoxysilicon and epoxy-s-triazine compounds.

The synthesis and preliminary evaluation of 2,4-bis(glycidyl)-6-methoxy-s-trisine is described. With certain formulations a tensile sheer value of about 2100 psi was obtained from room temperature up to and including 400° F.

An unexpected degree of reactivity was found in 2,4-bis(glycidyl)-6-methoxy-s-triazine and materials such as diphenylsilanediol and commercial silicone polymers could be used as true curing agents.

The inorganic adhesive prepared from ammeline and phosphorus pentoxide cures the 2,4-bis(glycidyl)-6-methoxy-s-trisine. Further work is necessary on this particular system.

Synthesis of trimethylsilylpropylene oxide, as a model compound, was shown by infra-red analysis but could not be isolated.

The Borden Company. WADC TR 57-696. ROOM TEMPERATURE CURING STRUCTURAL ADHESIVES FOR METALS. E. A. Blommers, E. W. Lane, B. D. Halpern. April 1958. ASTIA Document No. AD 151126.

Formulations containing novolac epoxy resins have been evaluated as room temperature, low pressure cured metal-to-metal adhesives. A number of combinations of novolac epoxy resins, Epon 562, Thiokol LP-3, fillers and mixtures of primary and tertiary amines have been found to give bonds with good strengths and salt spray resistance.

Attempts to use cyclohexene diepoxides in room temperature curing adhesive formulations did not prove successful. Copolymerization of methacrylatoethyl phosphates with methyl methacrylate in redox systems at room temperature yielded adhesives with mediocre strengths.

Narmco Industries, Inc. WADC TR 56-533 Pt II. ELEVATED TEMPERATURE RESISTANT SILICONE STRUCTURAL ADHESIVES FOR METALS. Beverly B. Stewart, Frank J. Riel, Samuel E. Susman. AF 33(616)-5141. August 1958. ASTIA Document No. AD 155806.

This report describes research and development under Contract AF 33(616)-5141, "Elevated Temperature Resistant Silicone Structural Adhesives for Metals" during the period 15 March 1957 to 15 May 1958.

The first objective of this program was the synthesis and evaluation of carboxyphenyl polysiloxanes. Several polymers, which contained varying percentages of carboxyphenyl groups, were prepared and evaluated as adhesives. It was observed that those resins containing carboxyphenyl groups had slightly lower bond strength compared to control resins. Failure of all polysiloxane bonds was primarily cohesive failure indicative of weak polymer structure.

The second program objective was the development of polymer structures other than simple polysiloxanes made from silane and silicone monomers. A bisphenol silane was synthesized, and phenolic and epoxy resins derived from it were evaluated as metal-to-metal adhesives. Synthesis of several intermediates required for hydroxphenyldisiloxane synthesis was accomplished; preparations of two hydroxyphenyldisiloxanes were carried out.

Three types of siloxane resins derived from Bisphenol-A and ethoxysiloxane monomers were examined. One type of siloxane-phenolic and a polysiloxane resin were found to have excellent heat resistance. Various combinations of these resins were compounded with conventional epoxy and phenolic resins, and evaluated as metal-to-metal adhesives.

Manuscript released by author July 1959 for publication as a WADC Technical Report.

Forest Products Laboratory. WADC TR 58-364. EVALUATION OF STRUCTURAL METAL BONDING ADHESIVES FOR BONDING GLASS-FABRIC LAMINATES TO METALS. H. W. Eickner. DO 33(616)-58-1. November 1958. ASTIA Document No. AD 204798.

An investigation was made by bonding lap-joint panels of glass-fabric laminates to aluminum alloy and to stainless steel with nine structural metal-bonding adhesives to determine if these adhesives were also suitable for this type of bonding. Test data were obtained on the aluminum-plastic bonds at -67° F, at 70° to 80° F, at 350° F, and after a 30-day exposure to salt water spray. The stainless steel-to-plastic bonds were tested at 70° to 80° F, at 350° F and at 500° F. Laminates prepared with four representative types of resins were included in this research.

Lap-joint strength values generally averaged from 2,000 to 2,500 pounds per square inch at -67° F at 70° to 80° F and after 30 days exposure to salt water spray. However, at 180° F some of the adhesives started to lose strength, and, in tests at

350° and 500° F, only the strength values of the more heat-resistant adhesives exceeded 650 pounds per square inch. Strengths at the higher temperatures were generally better on the plastic laminates prepared with heat-resistant resins. The results indicated that a number of adhesives, as presently formulated to qualify for structural bonding of metals, will adequately bond glass-fabric laminates to metals. No special requirement or specification is required, therefore, for adhesives for this type of bonding, other than the existing specifications on metal-bonding adhesives.

Narmco Industries, Inc. WADC TR 59-11. RESEARCH AND DEVELOPMENT ON ELEVATED TEMPERATURE RESISTANT STRUCTURAL METAL-TO-METAL ADHESIVES. Edward C. Janis, W. R. Boram, F. J. Riel, S. E. Susman. AF 33(616)-5488. May 1959. ASTIA Document No. AD. 214 698.

This report describes research and development under Contract No. AF 33(616)-5488, "Research and Development on Elevated Termperature Resistant Structural Metal-to-Metal Adhesives" during the period 1 February 1958 to 1 February 1959.

The main objectives of this program were the development of an adhesive system, for bonding 17-7 PH stainless steel, which would give 1000 psi tensile shear strength of 600°F after 200 hours exposure at 600°F, 130 pounds bend strength, 30 day salt spray resistance, and retention of useful structural strengths after various exposure times at temperatures from 500°F and to 1000° F.

An epoxy novolac modified silicone-phenolic adhesive, heat stabilized with arsenic pentoxide, gave 70% to 75% of the 200 hour, 600°F aged strength requirement, 98 pounds bend strength, and adequate 30 day salt spray resistance. Satisfactory strengths were obtained at temperatures ranging from 500°F to 975°F (10 minute exposure). A pyrophoric reaction at 1000°F resulted in unsatisfactory low strengths.

Test results of this adhesive, in general, did not meet the requirements of Military Specification MIL-A-8431 (USAF), Type III adhesives; however, structurally useable strengths were obtained at each test conditions.

Evaluations of edge sealant materials to exclude oxygen entrance into the glue lines of high temperature adhesives, to prevent oxidative degradation, resulted in the conclusion that no available materials accomplished this purpose.

Modifications of high temperature resistant epoxy, phenolic, and epoxy-phenolic adhesives exhibited poor 600°F aging properties.

A silicone-phenolic adhesive and an epoxy-silicone resin, each cured with arsenic pentoxide, showed promise of use as high temperature resistant adhesives.

Narmco Industries, Inc. WADC TR 59-82. INORGANIC HIGH TEMPERATURE ADHESIVES FOR METALS AND SANDWICH CONSTRUCTIONS. Roger A. Long, William Bassett. AF 33(616)-5776. June 1959. ASTIA Document No. AD-216 $4.52.

This program was divided into two parts. Part I consisted of an investigation of glassy phase adhesives and Part II was devoted to a study of metal-ceramic adhesive bonding of stainless steel by low temperature sintering technique.

PART I. Stainless Steel 17-7PH (RH 950) shear specimens bonded with the glass adhesives separately by both University of Illinois and Narmco gave equivalent shear strengths at all temperatures up to and including 1000°F. Minor material and processing procedure modifications by Narmco gave average shear strengths up to 4200 psi at 1000° F.

PART II. Excellent cohesive structure was developed by sintering a silver-copper alloy coated alumina in vacuo and argon. Lap shear strengths, on a 50% bonded area, up to 1200 psi at room temperature were obtained when bonding 302 stainless steel. Stainless Steel 17-7PH presented a more formidable problem but bonding was achieved by first copper plating the 17-7PH stainless surface prior to adhesive bonding.

Adhesive bonding by the sintering technique is sensitive to flatness of the stainless steel surfaces, the contact pressure, and to atmosphere contaminates which may prevent diffusion bonding.

University of Illinois. WADC TR 55-491. RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES. Gene H. Haertling, Kanaiyalal N. Parikh, Henry G. Lefort, Dwight G. Bennett. AF 33(616)-5468. June 1959.

The objective of the investigation was to develop high temperature resistant structural adhesives for desired alloy metals, particularly Type 17-7 PH Stainless Steel, from ceramic-oxide glassy-bonded coatings, from cermets with sintered metal bonds, and from air setting temperature and moisture resistant inorganic materials. In furthering this investigation, such adhesives were to be studied with appropriate optical and X-ray equipment in order to determine their basic structure and structural changes induced by variation in composition or thermal treatment. Such data, it was believed, might be usefully applied in improving existing adhesives and the methods and techniques of applying them.

Twelve new ceramic glassy bond adhesives were developed to mature at 1450°F or below. In general, they yielded shear strengths of about 1000 psi at room temperature and 600°F. At 800°F, values in excess of 2000 psi could be expected. However, at 1000°F, the shear values declined generally to the 300-1000 psi level.

Type PH 15-7 Mo stainless steel, bonded with UI 117-50, yielded shear strengths of about 1100 psi at room temperature and 600°F and more than 3000 psi at 800°F. Values at 1000°F, however, declined to about 300 psi.

Certain selected variations in the amount and ratio of powdered silicon and carbonyl iron (added to ceramic adhesives) yielded generally flat strength curves in excess of 1000 psi up through 800°F. At 1000°F, the shear values increased strikingly to between 2000 and 3000 psi. These unusual but very encouraging results suggested a possible "break-through" toward inorganic adhesives capable of performing effectively in a temperature range above 1000°F.

Air Setting or low heat cured adhesives, based on aluminum phosphate as the cementing agent, yielded shear strength values of up to 800 psi at room temperature. Efforts were made to minimize a bubble structure that developed during curing and thus increase shear strengths. The few variations tried were not appreciably successful.

Some modifications of glassy bond UI 1-67-1, by additions of ceramic oxides to the frit, gave visual indications of reducing the moisture sensitivity while still maintaining shear strength in excess of 1000 psi at temperatures up through 1000°F.

Several laboratory methods and practices were improved upon. A new stainless steel firing rack, which gave better alignment of shear specimens and allowed a larger number to be fired, was put in service. Continuous stirring of ceramic adhesive slips, during application to shear specimens, gave more uniform adhesive layers. Metal preparation for sandwich bonding was studied using aklaline cleaning and ferric chloride-hydrochloric acid etching. This method appeared to sufficiently roughen the honeycomb core and skin so that ceramic adhesive bonding was successful.

Microscopic examination of ceramic adhesive-

metal interface revealed either the presence of or an indication of (1) bubbles in the adhesive layer, (2) grain boundary effects in the adhesive, (3) some solution of metal in the adhesive layer, and (4) no noticeable change in either the adhesive layer or the metal with additional heat treatment of 145 hours at 1000°F.

Solutions of rare metals, when added to ceramic adhesives in small quantities (6-25 drops of 12-6% resinate solutions), did not, except for certain specific additions of gold and platinum, appreciably affect the shear strength of UI 117-50. However, additional thermal treatment, for nucleation and crystal growth about these rare metal ions in the adhesive layer, may possibly improve the strength and adhesiveness of such materials.

Forest Products Laboratory. WADC TR 54-447, Pt III. WEATHERING OF ADHESIVE-BONDED LAP JOINTS OF CLAD ALUMINUM ALLOY. H. W. Eickner. DO 33(616)-58-1. June 1959. ASTIA Document No. AD 227 441.

Lap-joint panels of clad 2024-T3 aluminum alloy bonded with nine commercial metal-bonding adhesives were exposed to the weather at the Panama Canal Zone; Fairbanks, Alaska; Miami, Fla.; State College, N. Mex.; and Madison, Wis. Panels were exposed while stressed in bending and while unstressed. Test panels were also exposed to several laboratory exposure conditions.

Results of these tests obtained after exposure periods of up to 3 years for 5 of the 9 adhesives were summarized in Part II of these reports. This Part III includes the results after completion of the 3-year tests on the other 4 adhesives included in this investigation.

Of the four adhesives included in this part of the study, the epoxy-phenolic adhesive (type intended to qualify under Type II of Military Specification MIL-A-8431) showed the best durability in weathering, with no appreciable loss of strength even in the test sites having the more severe exposure conditions. The nitrile rubber-phenolic and vinyl-phenolic adhesives performed well at the sites having the milder conditions, and also initially at the Panama and Florida sites. By the end of 3 years, however, panels bonded with these two adhesives and exposed at the Florida site showed low strength or complete failure. Strength values were also reduced in the Panama Canal Zone exposure.

The epoxy adhesive generally showed poor durability, with unstressed panels failing completely during the first year of exposure at Florida and Panama sites. The stressed epoxy-bonded panels also failed early in the exposure period at all test sites. The epoxy adhesive sample used in this study was one of the earlier batches of this adhesive. More recent batches, however, have shown improved durability under conditions of salt-water spray, high humidity, and climatic exposure, particularly when the bonds are protected with zinc-chromate-aluminized lacquer finish systems. Therefore, because of variability in the weathering resistance of the epoxy-resin bonds observed in these different studies, it is recommended that an appropriate finish system, such as zinc-chromate and aluminized lacquer, be applied to epoxy-resin bonded joints likely to be exposed to severe weathering conditions, to reduce the potential effects of this weathering on the quality of bond.

The exposure of unstressed bonded panels to conditions of 120°F and 97 percent relative humidity for 24 to 32 months reduced the strength of all four adhesives. The strength of the epoxy-bonded panels was only about 50 percent of their original strength after this exposure, as compared to 67 percent to 79 percent for the other three adhesives.

Aeronca Manufacturing Corporation. WADC TR 59-113, Part I. RESEARCH AND DEVELOPMENT OF INORGANIC HIGH TEMPERATURE ADHESIVES FOR METALS AND COMPOSITE CONSTRUCTIONS. Joseph Bayer, William A. Patterson. AF 33(616)-5538. July 1959. ASTIA Document No. AD-216 359.

An inorganic adhesive for 17-7 PH Stainless Steel metal bond was evaluated for resistance to various media and to various stresses. Factors were explored which affect the reproducibility of tests made on inorganic adhesive lap shear specimens.

An inorganic adhesive was developed for 17-7 PH Stainless Steel honey-comb sandwiches.

An inorganic adhesive bonding process was developed for fabricating 17-7 PH Stainless Steel honeycomb sandwiches.

Honeycomb sandwiches of 17-7 PH steel bonded with inorganic adhesives were evaluated in flexure and edgewise compression.

Forest Products Laboratory. WADC TR 59-152. STRENGTH PROPERTIES OF METAL-BONDING ADHESIVES AT TEMPERATURES FROM -100° TO +800°F. H. W. Eickner. DO 33(616)-58-1. July 1959.

Strength properties of metal joints bonded with vinyl-phenolic, acrylonitrile-phenolic, epoxy-elastomer phenolic, epoxy-phenolic, and epoxy type adhesives were determined over as much of the temperature range, -100°F to +800°F, as was practical, depending on the strength properties versus temperature obtained from the joints prepared from each adhesive. The adhesives selected are considered representative of good commercial materials for each chemical type included. Tensile-shear, long-time constant-stress, creep, fatigue and peel tests were conducted.

National Bureau of Standards. WADC TR 58-450. SURVEY OF ADHESION AND ADHESIVES. Frank W. Reinhart, Irma G. Callomon. AF 33(616)-53-9. July 1959. ASTIA Document No. AD 226 809.

The literature on the science of adhesion and the technology of adhesives published between 1945 and 1957 was reviewed. This survey is a continuation of the literature survey issued in 1945 by R. C. Rinker and G. M. Kline as N.A.C.A. Technical Note No. 989.

Special consideration is given to publications concerned with the basic aspects or science of adhesion. Reviews of some of the literature on surface science are included to show that there is sufficient knowledge concerning surface phenomena to indicate that studies in this field may be particularly helpful in further developments of the science of adhesion. The amount of technical data on adhesives published during the period covered by this survey is voluminous. Therefore, only data are presented that are considered typical or represent some unique or unusual aspect of adhesive technology.

Some additional references are given in Appendix A. Appendix B contains a selected list of articles published in 1958 after the text had been completed.

Narmco Industries, Inc. WADC TR 59-11, Supp. I. RESEARCH AND DEVELOPMENT ON ELEVATED TEMPERATURE RESISTANT STRUCTURAL METAL-TO-METAL ADHESIVES. Edward C. Janis, Frank J. Riel, William R. Boram, Samuel E. Susman. AF 33(616)-5488. October 1959. ASTIA Document No. AD 214 698.

The silicone-phenolic and epoxy-silicone-phenolic resins cured with arsenic pentoxide which were reported in WADC Technical Report 59-11 to possess the best

structural adhesive properties after exposures at 600°F, for the adhesive systems investigated, have been studied further with variations in curing cycles and post curing conditions.

Limited studies were also conducted on variations in the arsenic pentoxide content, substitution of a cyclic epoxy silopane for the epoxy novolac component, and use of sealants to retard oxidative degradation. The most promising adhesive formulations, when used with optimum processing procedures, show very little loss in strength when tested at 600°F after long time exposure to 600°F, and provide lap-shear strengths of about 900 psi.

Cryogenic Engineering Laboratory. WADC TR 59-260.
STRENGTHS OF STRUCTURAL ADHESIVES AT TEMPERATURES DOWN TO MINUS 424°F. William M. Frost. DO (33-616)-58-12. November 1959.

The tensile shear strengths of metal lap-joints bonded with ten commercially manufactured structural adhesives were determined at -107°F, -323°F, and -424°F. The effect of rapid cooling on bond strength was studied at -323°F; and results indicate that low temperature strength is not necessarily related to cooling rate. It was found that thin bonds may be less affected by thermal stresses than thick bonds. Most of the adhesives tested lost strength as temperature was decreased, but the amount of loss in strength was dependent upon the type of adhesive considered. Epoxy-phenolic adhesives, filled and supported on glass cloth, exhibited the best low temperature performance and showed little or no decrease from room temperature strengths. Filled epoxide adhesives demonstrated slightly less low temperature strength than the epoxy-pnenolics but better bonding techniques could improve the performance of these epoxides.

Forest Products Laboratory. WADC TR 59-564. Pt I. ENVIRONMENTAL EXPOSURE OF ADHESIVE-BONDED METAL LAP JOINTS. H.W. Eickner. DO 33(616)-58-1. February 1960. ASTIA Document No. AD 235 927.

An investigation was started of the relative durability of metal-bonding adhesives when exposed at two sites, Miami, Fla., and the Panama Canal Zone. Twelve sets of lap-joint panels of clad 2024-T3 aluminum alloy, respectively bonded with 12 different adhesives, and 1 set of 17-7PH corrosion-resisting steel bonded with another adhesive were prepared for exposures at these two sites and for laboratory-controlled exposure tests. Panels were exposed while stressed in bending and while unstressed. Partial sets of panels are to be removed and tested periodically through 3 years of exposure. This initial report summarizes the preparation of the test panels, data from the laboratory-controlled exposures and, for some of the adhesives, data after 1 year of natural environmental exposures.

Central Research Laboratory. WADD TR 59-15. DEVELOPMENT OF LOW PRESSURE AMBIENT TEMPERATURE CURING ADHESIVE FOR FIELD REPAIR OF METAL AIRFRAMES. Elizabeth A. Blommers, Earl W. Lane, Samuel Loshaek, Benjamin D. Halpern. AF 33(616)-5984. April 1960.

Two-part room-temperature-curing adhesive systems, based on novolac epoxy resin EPIPHEN 825, were developed which pass the bend strength, the salt spray exposure, and all the shear strength requirements, except at -67°F, of Military Specification MIL-A-5090B when the adhesives are bonded to Alclad aluminum (cleaned in WAC-1 solution) and are cured for either six days at 73°F or 30 minutes at 212°F. The exceptional salt spray resistance of these adhesives was obtained by the incorporation of zinc or lead chromate as part of the filler. Heat-cures of one to two hours at 212°F of these systems can raise shear strengths at -67°F to 2500 psi, from 2000 psi, but salt spray resistance can thereby be lowered as much as 50%. A special cleaning procedure for aluminum, or the use of a four component adhesive, can eliminate the sensitivity to corrosion after a heat-cure.

One particular two-part high-temperature-resistant adhesive, when cured at temperatures between 200°F and 220°F can pass Military Specification MIL-A-5090B and also exhibit strengths of 1000 psi at 300°F and 400 psi at 500°F. Other high-temperature-resistant adhesives give shear strengths of around 1800 psi at 300°F but their other properties are not always up to the requirements of Military Specification Mil-A-5090B.

University of Illinois. WADC TR 55-491. Pt. V. RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES. Gene H. Haertling, Kanaiyalal N. Parikh, H. Richard Thornton, Henry G. Lefort, Julian H. Lauchner, Dwight G. Bennett. AF 33 (616)-6192. June 1960.

Glassy bond and air setting adhesives were developed and studied. High temperature (to 2000°F) strength, ductility and moisture resistance were targets.

Methods of attack included metal and metal oxide powder additions, crystallization of gold nucleated bonding glasses, study of metal surface roughness function and evaluation of stress-strain relations in metal-adhesive composites.

In a study of moisture resistance, additions of about 33% of Fe_3O_4 were made to selected air setting or low temperature curing adhesives. Tensile shear values of the order of 1350 psi at room temperature and 1150 psi at 800°F were obtained but the adhesives disintegrated after a 3 1/2 hour boiling water treatment. When duplicate specimens were given a preliminary heat treatment of one hour at 1000°F shear strengths were reduced by about 50%, but they were essentially unchanged by the 3 1/2 hour boiling water treatment.

In glassy bond adhesives, stainless steel and phosphatized carbonyl iron powder additions gave improved strength, still increasing at 1000°F. Crystallized bonds gave higher strengths at the highest test temperatures. An optimum metal surface roughness value was noted. High order residual stresses (desirable to remove) were found in some metal-glassy bond composites.

Aeronca Manufacturing Corporation. WADC TR 59-113. Part II.
RESEARCH AND DEVELOPMENT ON INORGANIC HIGH TEMPERATURE ADHESIVES FOR METALS AND COMPOSITE CONSTRUCTIONS. Joseph Bayer, Ova E. Johnston, William A. Patterson. AF 33(616)-5538. July 1960. ASTIA Document No. AD-247 410.

Modifications of the previously recommended inorganic adhesive were attempted to increase the bend resistance of the bonded joint. The modifications, incorporated singularly or in combination, include (1) substitution of various metal oxides and powders in the adhesive formula, (2) special surface preparation of the metal powders, (3) variation of the particle size and weight percentage of the powders, and (4) the introduction of porosity in the adhesive. Increased bend resistance was noted with the addition of phosphatised carbonyl iron and the addition of aluminum oxide. The aluminum oxide apparently introduced porosity into the adhesive. The processes which increased the bend resistance require further development to assure

reproducibility of results.

Methods for producing inorganic adhesive bonded honeycomb sandwich panels were further investigated. Preliminary experiments indicate that the quartz lamp-quench die method may alleviate some of the problems encountered in furnace bonding.

The development of new adhesives and processing techniques for bonding super alloys has been initiated.

University of Minnesota. WADD TR 60-687. AN INVESTIGATION OF LONGITUDINAL SHEAR DISTRIBUTION AND DAMPING IN A VISCOELASTIC ADHESIVE LAP JOINT. Carlos P. Avery. AF 33(616)-6828. November 1960. PB 171 556. Order from OTS $1.00.

An analysis of the damping in a viscoelastic adhesive of a simple lap joint is presented. Expresions are developed for shear distribution and damping in the adhesive when the lap is sheared by a low frequency sinusoidal force. Calculations are made illustrating the variation in damping and shear distribution as a function of the dimensions and material properties of the lap components.

Experiments were performed which confirm the theory. Limitations, extensions, and possible refinements of the theory are presented.

Application Studies

University of Alaska. WADC TR 57-173. METEOROLIGICAL AND SOLAR RADIATION DATA COLLEGE, ALASKA. Wm. S. Wilson. AF 18(600)-40. May 1958. ASTIA Document No. AD 151190.

The daily observations of maximum and minimum temperatures, maximum, minimum, and mean of the relative humidity with the approximate time (Alaskan Standard Time) of the occurrence of each maximum and minimum, maximum intensity of solar radiation and total radiation received on a horizontal surface and a surface slanted 45° to the south are tabulated along with the monthly averages and totals of each of these factors as obtained in connection with the project on the "Exposure of USAF Materials near College, Alaska." The temperature data in each year are represented on a composite graph. The monthly values of all data are tabulated in separate tables. The period covered is from 1 September 1955 through 9 March 1957.

New Mexico College of Agriculture and Mechanic Arts. WADC TR 56-600. SOLAR RADIATION AND WEATHER DATA SOUTH CENTRAL NEW MEXICO. A. M. Lukens, A. E. Prince. AF 33(616)-3275. January 1959. ASTIA Document No. AD 208141.

The daily recordings of maximum and minimum temperatures, relative humidities, and the intensity of solar radiation on a surface slanted 45° facing to the south are tabulated with the monthly averages and totals of each factor. These data have been collected, compiled, and recorded in connection with work concerning the effects of actinic and biological agents of deterioration on Air Force materials. Charts are included showing a summary of temperature and humidity data from August 1947 through September 1956 and solar radiation data from June 1951 through October 1956.

Forest Products Laboratory, Forest Service, U.S. Dept. of Agriculture. WADC TR 58-455, Part I. PRESSURE-SENSITIVE TAPE SUSPENSION SYSTEM FOR AIRCRAFT PARTS IN SHIPPING CONTAINERS PART I. Laboratory Tests of Suspension Systems. Arnold W. Voss. PO (33-600) 53-4023. June 1959. ASTIA Document No. AD 216533. PB 151945. Order from OTS $1.75.

A suspension system utilizing pressure-sensitive tape in lieu of blocking and bracing or other means of supporting aircraft parts in a shipping container was found adequate to pass the rough-handling tests prescribed for military packaging. Methods of attaching the tapes to the container and necessary precautions were determined. The relationship between the weight of the item and the strength required for glass filament-reinforced tape was determined and a basis established for the design of tape suspension systems.

WADC TR 59-297. DEVELOPMENT OF LIGHTWEIGHT ALUMINUM CRATES FOR BULKY LOW DENSITY ITEMS. Warren D. Hypes. September 1959. PB 161 364. Order from OTS $1.00.

Formed aluminum channels were used in assembling crates for lightweight, bulky airframe components. The crates passed the Level A rough handling requirements of Specification MIL-P-7936. In addition to possessing the required strength, the channels are reusable, thus a modular system of crate components could be developed. Use of the aluminum crates will provide a means for considerable reduction in tare weight when used as shipping containers for airframe components. The use of the aluminum channels also appears to be economical.

Bolt Beranek and Newman, Inc. WADC TR 58-667. THE EFFECTS OF SIMULATED SPACE ENVIRONMENTS ON CUSHIONING MATERIALS. David N. Keast, Jordan J. Baruch. AF 33(616)-5887. September 1959. ASTIA Document No. AD 229 932.

Measurements of some of the properties of several types of package cushioning materials at temperatures of +75°F, -65°F, and -200°F and pressures of 760 and 10^{-4} mm Hg have been performed. Static and dynamic stress-strain curves and vibration transmissibility characteristics have been obtained for glass fiber, latex bound hair, polyurethane foam, silicone rubber foam, and crushable paper honeycomb materials. The data are presented and briefly discussed, and the instrumentation necessary for the performance of the measurement is described.

The dynamic stiffness and damping of the cushioning materials tested generally decreases in vacuum; the change being greatest for the polyurethane and silicone foams and least for the paper honeycomb and latex bound hair materials. Similarly, reduced temperature increases the stiffness of the materials; the effect again being greatest for the polyurethane foam, and least for the glass fiber and paper honeycomb materials.

Forest Products Laboratory, Forest Service, U.S. Dept. of Agriculture, WADC TR 58-455, Pt. II. PRESSURE-SENSITIVE TAPE SUSPENSION SYSTEMS FOR AIRCRAFT PARTS IN SHIPPING CONTAINERS, PART II. Effects of Accelerated Exposure on Tape Properties. Arnold W. Voss. PO (33-600) 53-4023. October 1959. PB 161362. Order from OTS $1.00.

Black pressure-sensitive tape backed with polyester film and reinforced with glass filaments was subjected to dynamic tests at room temperature and at -20°F to determine its strength and toughness in tension and the shear strength and toughness of its bond to aluminum. Tests were made before exposure and after 4, 8, 12, 16, 20, and 32 cycles of accelerated exposure. Each cycle

included 18 hours of ultraviolet light with alternate wetting and drying in a weatherometer, and 4 hours of freezing at below zero Fahrenheit. The initial effect of exposure was an improvement in these properties, but continued exposure beyond 12 cycles deleteriously affected the tensile strength and toughness of the tape. Principal conclusions are that suspension systems utilizing this tape have adequate weather resistance to withstand extended periods of outdoor exposure, and that the serviceability of the system after extended exposure may be determined by visual inspection.

WADD TR 60-4.
MEASURING FIELD HANDLING AND TRANSPORTATION CONDITIONS. Capt. Kenneth W. Bull, Materials Central, Dr. Carl F. Kossack, Purdue University. February 1960. PB 161 918. Order from OTS $1.00.

This study involves concealing shock recording instruments inside ordinary shipping containers and shipping the instrumented packages through normal supply channels to determine the actual heights from which packages are dropped.

Based on the data given in this report, a test drop of 21 inches is considered adequate for specifications. Further clarification is given in the report.

The recorders, calibration procedure, collected data, analysis, accuracy and associated problems are discussed. Recommendations for future studies of this sort are also given.

Materials Central. WADD TR 60-58.
SYMPOSIUM ON PROCESSING MATERIALS FOR RE-ENTRY STRUCTURES. May, 1960.

The theme of the symposium, "Processing" is carried throughout the papers which cover general requirements and problem areas, materials applications, methods used to form and fabricate items from high strength moderate temperature materials, high temperature metallics and high temperature non-metallics. Specific papers concern themselves with methods of processing materials such as: refractory ceramics, graphite and ablation materials, refractory metals, super alloys, forging methods, explosive forming, welding and plasma and metallizing. There is a limited amount of published information for most of these topics.

Chicago Midway Laboratories. WADC TR 59-366, Part II.
THERMAL PROTECTION OF STRUCTURAL, PROPULSION, AND TEMPERATURE-SENSITIVE MATERIALS FOR HYPERSONIC AND SPACE FLIGHT . William P. Manos, Donald E. Taylor, Anthony J. Tuzzolino. AF 33(616)-6006. July 1960. PB 171 147. Order, OTS $3.50.

This report presents the results of an investigation intended to examine various phenomena applicable to the protection of materials subjected to severe thermal environments and evaluate their effectiveness on the survival of materials now available. The heating experienced by materials in selected severe thermal environments is reviewed. The various phenomena that might be employed for protection are studied and evaluated analytically. Experimental tests were performed to aid in evaluation of certain phenomena, using the plasma discharge of a 1000-KW air-stabilized arc to provide a severe thermal environment. These results are reported, along with a correlation of the observed behavior with theory and previous tests.

McGraw-Hill Book Company. WADD TR 60-584.
HANDBOOK OF FIBROUS MATERIALS. Harry Mileaf. AF 33(616)-5726. Oct. 1960. PB 171 494. Order, CTS $6.00.

This report contains the summarized and consolidated information extracted from WADD Technical Reports covering several phases of fibrous materials research. The information is arranged to make the results of these reports more readily available and useful to decelerator designers and others interested in the fibrous materials phase of Air Force research.

The report is divided into sections covering the pertinent facets of fibrous material information. Fairly complete information is supplied on various phases of basic design data, sewability, friction, abrasion and weather resistance, porosity and air permeability, temperature properties and chemical resistance of different yarns, cords, webbings and fabrics. Some information is also supplied on sunlight and weather resistance, tear resistance, radiation, aging and electrostatic properties, stiffness, flexibility, elongation, elastic recovery and energy absorption.

Southern Research Institute. WADC TR 57-649, Part III. OTS Release.
DETERMINATION OF THE MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL MATERIALS AT VERY HIGH TEMPERATURES AFTER RAPID HEATING - PART III. The Effect of Simultaneous Heating and Loading on the Tensile Properties of Typical Structural Alloys. A. Clyde Willhelm, J. Robert Kattus. AF 33(616)-3494. Nov. 1960. PD 251 811. PB 171 985. Order, OTS $1.25.

Three series of experiments were conducted to determine the effects of simultaneous heating and loading on the stress-strain characteristics of some typical structural sheet metals, and to correlate the experimental data with predicted effects based on the concept of a Mechanical Equation of State. The first series of experiments was performed on Inconel X nickel-chromium age-hardening alloy and on 17-7 PH/TH 1050 stainless steel. The 17-7 PH and full-hard Type 301 stainless steel were used for the second and third series respectively. Improvements in the experimental method and equipment were sought in each succeeding series.

The results of the experiments indicate that although practical application of the concept of a Mechanical Equation of State to design problems appears promising on a qualitative basis, improvements in prediction and experimental methods will be required to obtain quantitative confirmation.

Syracuse University Research Institute. WADD TR 60-446.
INFORMATION REQUIREMENTS FOR APPLICATION OF NONMETALLIC STRUCTURAL MATERIALS TO AEROSPACE VEHICLES. C. S. Grove, Jr., R. Ford Pray, III. AF 18(600)-1876. November 1960.

The use of non-metallic materials in aerospace vehicles has increased phenomenally in recent years. However, data and information have not been completely or appropriately disseminated in a manner which would be most useful to the designer of such vehicles. The purpose of this project was to survey the aerospace industry and agencies concerned with aerospace problems. In particular, this study was directed toward determining which materials, properties, and problem areas in non-metallics exist now or will exist in the future and which methods can be used to place evaluated data and information into the hands of the designer. The following report presents the results of this survey. It was determined that a Design Handbook or Manual on non-metallic materials will greatly benefit the aerospace

field as a whole. Specific recommendations for such a handbook are presented in the report.

Belfour Engineering Company. WADD TR 60-867.
CONSIDERATIONS AND RECOMMENDATIONS FOR DEVELOPING A MATERIALS INFORMATION PROCESSING CAPABILITY. Albert J. Belfour. AF 33(616)-7064. February 1961. PD 258 643. Order from OTS $0.50.

A survey is summarized in conjunction with recommendations for the development of a Materials Information Processing Capability at the WADD Materials Central. The discussion covers some basic concepts of technical information, user requirements, and existing information "centers". A consistency in pattern of information processing techniques is examined and discussed. Recommendations are given for the development of a Materials Information Processing Capability.

McGraw-Hill Book Company, Inc. WADD TN 60-246.
MATERIALS INFORMATION CENTERS. K. A. Winter, Irving Lopatin. AF 33(616)-6288. February 1961. AD 259 318. Order from OTS $2.00.

This technical note contains a listing of all known organizations or agencies which may be designated as a materials information center on the basis of their activities concerned with the collection and dissemination, in some manner, of information on a class, group, or type of material, or materials.

The purpose of this technical note is to provide interested personnel of the Department of Defense, its contractors, and materials suppliers with a means of locating and obtaining information pertinent to their specific area of interest.

Coatings

TR 4617.
DEVELOPMENT OF GLOSS SPECIFICATION FOR CAMOUFLAGE FINISHES. Harry Schecter. March 1941.

TR 5645.
STUDY OF THE METALLURGICAL CHARACTERISTICS OF THREE INDUCTION-HARDENED STEELS HEATED AT VARIOUS RATES. James W. Poynter. October 1947.

TR 5657.
THE EFFECT OF OVERSTRESS IN FATIGUE ON THE ENDURANCE LIFE OF SAE 4340 STEEL. Ture T. Oberg. January 1948.

Battelle Memorial Inst. TR 5692.
AN INVESTIGATION OF ELECTRODEPOSITED ALLOYS AND PURE METALS AS SUBSTITUTES FOR ZINC AND CADMIUM FOR PROTECTIVE FINISHES FOR STEEL PARTS OF AIRCRAFT. C. A. Snavely, A. B. Tripler, and C. L. Faust. W33-038-ac-15723(16940). April 1948.

TR 5700.
EVALUATION OF THE CONDITION OF DOPED FABRIC SURFACE BY THE MULLEN BURST STRENGTH TEST AND OTHER METHODS. Elmer E. Jukkola. May 1948.

TR 5700, Supplement 1.
EVALUATION OF THE CONDITION OF DOPED FABRIC SURFACE BY THE MULLEN BURST STRENGTH TEST AND OTHER METHODS. Elmer E. Jukkola. July 1949.

Battelle Memorial Inst. TR 5692, Supp. I.
INVESTIGATION OF ELECTRODEPOSITED ALLOYS AND PURE METALS AS SUBSTITUTES FOR ZINC AND CADMIUM FOR PROTECTIVE FINISHES FOR STEEL PARTS OF AIRCRAFT. A. B. Tripler and C. L. Faust. W33-038-ac-21107(20145). September 1949.

TR 5125.
FATIGUE LIMIT OF CHROMIUM PLATED STEEL. Louis Mehr, 1/Lt., Ture T. Oberg, and J. Teres, Captain. July 1944.

TR 5700, Supp. 2.
EVALUATION OF THE CONDITION OF DOPED FABRIC SURFACE BY THE MULLEN BURST STRENGTH TEST AND OTHER METHODS. Elmer E. Jukkola. July 1950.

Balco Research Laboratories. TR 52-36.
DEVELOPMENT OF TRANSPARENT MATERIALS WHICH REDUCE EFFECTS OF PRECIPITATION STATIC IN AIRCRAFT. M. U. Cohen, PhD and G. A. Dalin, PhD. AF 33(038)-12240. March 1952. PB 118 281. LC mi $5.70 ph $16.80.

Methods of laying a transparent electrically conductive film on plastic airplane canopies are discussed. The purpose of such films is to conduct to the aircraft frame the static charges developed on the canopies by friction with air, dust, snow, etc., during flight. Unless these charges are properly dissipated, precipitation static results, producing serious interference with radio communication.

The types of film studied include metal oxides, metals, phosphors, polyelectrolytes, electrolytes dispersed in waxes, and electrolytes dispersed in non-polar polymers.

Special techniques employed in the research include film-forming by withdrawing slides from solution at a controlled rate, vacuum-evaporation of metals, metallic oxides and phosphors, a convenient method of measuring surface resistance, and the deposition from solution of adherent metal-oxide films on acrylic sheet.

Preliminary studies indicate that the monobutyl ester of orthophosphoric acid in polymethyl methacrylate and analogous systems merit further detailed investigation.

Battelle Memorial Inst. TR 5692, Supp. 2.
AN INVESTIGATION OF ELECTRODEPOSITED ALLOYS FOR PROTECTION OF STEEL AIRCRAFT PARTS. A. B. Tripler, Jr., J. E. Bride, J. A. Gurklis, and C. L. Faust. AF 33(038)-8750. November, 1952.

Work done in this report covers the preparation and evaluation of eleven alloy deposits. Methods and solutions used for the preparation of both co-deposits and diffused coatings are listed. Results of "Wet-Dry" exposure tests, X-ray diffraction, potential - time data of various coatings have been tabulated. A graphical system for the better interpretation and rating of the "Wet-Dry" test was developed. Manganese-zinc alloy diffusion type coatings of 50% manganese and 50% zinc composition exhibited the most promising corrosion protection properties

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of the various coatings investigated. Seventy-two panels were prepared and are being tested in outdoor exposure in northern Florida in order to evaluate the corrosion protection afforded by manganese and manganese-zinc coatings as compared to zinc, zinc chromated, and cadmium coatings. It is believed that a co-deposited zinc-silver coating of 25% silver merits further investigation. Also, further studies of corrosion products should yield information enabling the design of an alloy coating of maximum protection.

Battelle Memorial Inst. TR 5692, Supp. 3.
AN INVESTIGATION OF ELECTRODEPOSITED ALLOYS FOR PROTECTION OF STEEL AIRCRAFT PARTS. C. L. Faust, A. B. Trippler, Jr., C. R. Konecny, and W. C. Schickner. AF 33(038)-8750. November 1952.

Previous work reported under this AFTR No. 5692 indicated that binary metallic systems, having manganese as one of the elements, might afford good cathodic protection to steel and be a superior coating to that of zinc or cadmium. Following a literature search, methods of electrodepositing various alloys of manganese were investigated. Manganese-zinc and manganese-tin coatings were prepared and tested under exposure conditions of alternate condensation and drying. Certain compositions of the two alloys protected steel longer than pure zinc coatings, however, they were inferior to pure cadmium coatings. The plating processes were not completely developed, and are not ready for practical application. Methods for plating manganese-nickel, manganese-chromium, manganese-iron, and manganese-molybdenum were also studied but with less success.

Northrop Aircraft, Inc. TR 52-99.
RESEARCH INVESTIGATION OF PROTECTIVE COATINGS FOR MAGNESIUM. H. B. Childers, K. B. Niles, and A. G. Valles. AF 33(038)-23273. November 1952.

Numerous proprietary and developmental organic coatings are screened for relative corrosion protection of magnesium sheet alloy, with respect to a current Government specification system. Superior primers and systems are more fully evaluated for mechanical properties as well as corrosion protection. It was found that air-dry vinyl systems offer optimum protection, considering the systems tested. Developmental data and discussions of magnesium corrosion inhibitors and galvanic cell test methods are included in the report.

Bjorksten Research Laboratories, Inc. TR 52-48.
DEVELOPMENT OF ELECTRICALLY CONDUCTIVE TRANSPARENT COATINGS FOR ACRYLIC PLASTIC. Johan Bjorksten, Harry L. Hamilton, and Evelyn E. Smith. AF 33(038)-23319. December 1952. PB 118034. LC mi $4.50 ph $12.30.

In order to dissipate precipitation electrostatic charges built up on transparent plastic aircraft canopies during flight an electrically conductive transparent coating, easily applied to formed aircraft canopies made from acrylic sheet conforming to Specification MIL-P-5425, has been developed. The coating is applied by abrading or scratching the acrylic surface slightly with a suede brush, rubbing in finely divided graphite, and spraying the surface with a protective film of 1:5 methacrylic acid-methyl methacrylate copolymer resin.

The composite material, i. e., the acrylic and applied coating, retains essentially all the original mechanical properties, optical clarity and light transmittance characteristics of the base material. Its surface resistance is in the range 1-10 megohms per square and is relatively insensitive to the wide variation in relative humidity, temperature, and altitude conditions that might be expected in aircraft operation. The coated acrylic is transparent to radio frequency energy as well as light, its dielectric loss factor being only slightly higher than the acrylic alone. It has sufficient permanence to the effects of sunlight, temperature variations, crazing, rain, abrasion, wind erosion, and oil absorption associated with the normal operation and maintenance of aircraft.

Three F-86 aircraft canopies, two new and one removed from service, were coated with the developed coating. The new canopies were tested in flight at Wright Air Development Center and performed satisfactorily. Thus, the coating can be applied to newly produced canopies with the least disruption in current manufacturing practices. The used canopy crazed severly when the protective coating was applied. This was caused by the strains present in the surface. Work with small methacrylate samples similarly strained revealed that the crazing could be avoided by annealing prior to spraying. Thus, it seems likely that the developed coating can be applied to canopies removed from service after an annealing pretreatment.

TR 52-232.
ELECTRODEPOSITION OF TITANIUM FROM AQUEOUS SYSTEMS. Ernest J. Breton, 1st Lt., USAF. RDO No. 611-11. December 1952.

United Chromium, Inc. TR 53-271.
DEVELOPMENT OF A SUBSTITUTE OR IMPROVEMENT OF CHROMIUM ELECTRODEPOSITS, Jesse E. Stareck, Edgar J. Seyb, Jr. and Angelo C. Tulumello. AF 33(616)-234. July, 1953. PB 122098. LC Mi.$6.00 Ph $19.80.

The fatigue limit of heat-treated steel was studied with chromium deposits from various solutions with varying plating conditions. Baseline information was obtained from the conventional sulfate bath and the newer high speed CR-110 bath. The fatigue limit was shown to increase with increased fineness of crack pattern in the chromium deposit. By adjusting the plating conditions to give more filled-in cracks the fatigue limit was materially increased. Deposits with very few crack-lines showed extremely low fatigue limits.

Baking chromium deposits to eliminate hydrogen embrittlement drastically lowered the fatigue limit in most cases. Certain types of deposits possessing oxide inclusions gave higher rather than lower results after baking. A copper strike between chromium and steel was shown to largely prevent embrittlement without changing the fatigue properties.

The data is taken to show that residual tensile stress in electrolytic chromium is the primary mechanism for the lowering of the fatigue limit in chromium plated steel.

Battelle Memorial Inst. TR 5692, Supp. 4.
AN INVESTIGATION OF ELECTRODEPOSITED ALLOYS FOR PROTECTION OF STEEL AIRCRAFT PARTS. Arch B. Tripler, Jr., Glen Fuller and Dr. Charles L. Faust. AF 33(038)-8750. July 1953.

Pure-manganese, manganese-zinc-alloy, and zinc-tin-alloy coatings on steel were exposed for 21 months at the Battelle North Florida Research Station. Panels coated with zinc, cadmium, and chromated zinc were exposed concurrently as standards.

The pure-manganese and manganese-zinc coatings were inferior to the zinc and cadmium standards.

The zinc-tin-alloy coatings of 0.3 mil thickness and greater protected the underlying steel as well as the zinc and cadmium standards.

Missouri School of Mines and Metallurgy. TR 53-162, Part 1.

ELECTRODEPOSITION OF TITANIUM. Albert W. Schlechten, Martin E. Straumanis and C. Burroughs Gill. AF 33(616)-75. September 1953. PB 111797. OTS $1.50.

The high resistance of titanium to corrosion, particularly by sea water or nitric acid, makes it very desirable to plate other metals with a protective coating of titanium. Many attempts by many workers have been made to electrodeposit titanium either to form a coating or as a means of producing the metal but little success has been reported. This report describes a large number of experiments using aqueous and fused salt baths. It is doubtful if any true electrodeposits of titanium were obtained, but a procedure is described which will yield a thin but coherent and corrosion resistant titanium coating. Extensive data are also reported on the hydrogen overvoltage on titanium in aqueous electrolytes.

Battelle Memorial Inst. TR 53-218.

DEVELOPMENT OF A NONDESTRUCTIVE TEST FOR EVALUATION OF ADHESION OF ELECTRODEPOSITS ON STEEL AS IN SILVER-PLATED AIRCRAFT BEARINGS. Arch L. Walters and Samuel A. Wenk. AF 18(600)-124. November 1953. PB 131226. OTS $1.75.

This report surveys the nondestructive test methods currently being used for the inspection of silver-plated aircraft bearings. Details of the two principal test methods are given.

The selection of the test method depends on whether a copper or nickel strike is applied to the bearing shell before silver plating. The shot peening test, wherein the surface of the silver plate is lightly shot peened under controlled conditions is principally used on bearings having a copper strike. The nickel strike bearings are tested by heating the bearing to 950°F, followed by rough boring, or machining to size and then X-raying the bearing surface.

Shot peening will produce wrinkles in poorly bonded areas. The heating method produces blisters, which are readily revealed by X-ray examination following machining.

In addition, the report contains "Proposed Amendments to Technical Order No. 24-5-1 on Electrodeposition of Silver Plate". It suggests changes in the Technical Order for the purpose of improving the procedure for silver plating and reducing the occurence of poorly adherent silver plate at Air Force depots overhauling aircraft engines.

U. S. Bureau of Mines. TR 53-317.

ELECTRODEPOSITION OF TITANIUM AND ZIRCONIUM. Robert M. Creamer, David H. Chambers and Charles E. White. AF 33(038)-50-1085. December 1953. PB 111525. OTS $0.75.

Aqueous, non-aqueous, and fused electrolytes have been investigated in an attempt to electrodeposit titanium and zirconium. Aqueous and non-aqueous electrolytes have been found capable only of coplating small percentages of titanium along with another more easily plated metal such as tin, cadmium, or zinc. Fused alkali borate baths were found to give non-adherent, spotty cathode deposits of a titanium boride. Fused halide electrolytes gave powdered metal deposits and a potassium chloride-lithium chloride-titanium (III) chloride electrolyte gave weighable plates on iron, nickel, and copper cathodes. Notes on some of the chemical aspects of the investigation are included.

WADC TR 53-73.

FIRE-RETARDANT COATINGS. Sam Collis. August 1954. PB 131002. OTS $1.75.

This project was undertaken in an effort to develop an insulative fire retardant coating suitable for use on aircraft. The desired coating should be a paint-like material, and when applied at a rate of not more than 12 pounds per 100 square feet on metal, should be able to provide a temperature differential of at least 1650°F. for a period of 30 minutes when subjected to a 2000°F flame. In addition, the coating should have the usual properties of a good paint film.

This report covers the initial screening of a number of materials to determine their performance under some of the conditions to which the coating might be subjected. A number of commercially available coatings were evaluated. No entirely satisfactory coating was developed or evaluated. Types or classes of materials deemed worthy of further investigation are noted.

Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they were subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

WADC TR 53-466 (Part 1).

EVALUATION OF DRY-FILM LUBRICANT COATINGS. William C. Hart, Bernard Rubin. September 1954. PB 121922. OTS $0.75.

The endurance and load-carrying capacity of commercial and experimental dry-friction reducing films have been investigated at low surface rubbing speeds in the Falex Lubricant Tester. This investigation indicates that the most successful types of dry film lubricants have excellent extreme pressure properties and good anti-wear properties for plain bearing applications at low speeds.

Baked resin-bonded films have superior endurance life than airdrying spray and dip coatings. The endurance life and load-carrying ability of dry film lubricants is dependents upon the resin-bonding agent and pretreatment of the metal surface prior to application of the film. Maximum endurance life is obtained if both surfaces are dry film coated.

WADC TR 54-392.

MATERIALS REPORT ON THE LOCKHEED ATC-1 TRANSPARENT ELECTRICALLY-CONDUCTING COATING FOR ACRYLIC PLASTIC. L. Harold Bullis. September 1954.

Accelerated weathering tests, air and rain erosion tests, and flight tests were conducted on the Lockheed ATC-1 transparent, electrically conducting coating to determine its suitability for use on canopies of military aircraft for the reduction of precipitation static. The coating successfully retained the desired values of electrical conductivity, transmittance, and haze throughout most of the tests. On this basis it is recommended from the materials standpoint as a coating meeting the physical requirements established by the Communication and Navigation Laboratory, WADC.

WADC TR 54-240.

A STUDY OF CADMIUM-TIN AND ZINC-TIN ALLOY ELECTRODEPOSITS. Bennie Cohen. September 1954. ASTIA Document No. AD 51052. PB 121808. OTS $1.25.

A study has been made of cadmium-tin and zinc-tin alloy electrodeposits in the continuing efforts of the Air Force to find superior corrosion resistant coatings. The alloy systems evaluated were electrodeposited from

fluoborate solutions. Cadmium was used as a basis of comparison throughout. The cadmium-tin alloy coating was found to be superior to cadmium coatings in the majority of tests performed. The cadmium-tin alloy coating was found to have excellent resistance to salt spray, jet fuels, high temperature synthetic oils, organic acid vapors, and to have very little embrittling effect on hardened steel.

Sherwin-Williams Co. WADC TR 54-373. PROTECTIVE COATINGS FOR MAGNESIUM. T. Kirk Hay, Gerald F. Bechtle, Garmond G. Schurr, Maurice Van Loo. AF 33(616)-35. October 1954. PB 111886. OTS $5.50.

The purpose of this investigational work, which was to strive to develop improved coatings for the protection of magnesium, has been accomplished. The recommended finishing systems based on this work use a vinyl toluene-ether ester vehicle in both the primer and the topcoat. The primers are pigmented at 35% pigment volume concentration with zinc chromate and silicon dioxide. The solvent resistance of these systems has been variable. The two types used in the samples submitted for evaluation show bad softening but fair recovery upon solvent evaporation.

Time consuming difficulties were encountered in establishing a satisfactory test surface. Pretreatment of the magnesium according to MIL-M-3171, Type III, by a preferred commercial processor and the use of replicate test panels was the solution. Wash primers as pretreatments for magnesium or as an addition to dichromate pretreatments show promise but their performance is too variable to warrant inclusion in the recommended system at this time. Improvements in wash primers included the use of strontium chromate and silicon dioxide as the pigmentation and the reduction of acid activator content to 25% of normal with simultaneous control of water content.

Several items were investigated briefly and showed enough promise to warrant further study. These include polysulfide resins, furan resins, a pigment prepared from calcium sulfide and ammonium vanadate, pigmentation changes in wash primers, and a method for determining the permeability of films to chloride ions.

This work was done by the Paint Research Department of The Sherwin Williams Company under contract AF 33(616)-35 from 29 March, 1952 to 1 June, 1954.

Goodyear Tire & Rubber Co. WADC TR 53-511. DEVELOPMENT OF A HEAT-RESISTANT RAIN-EROSION-RESISTANT COATING. F. A. Jeffries. AF 18(600)-110. November 1954.

High speed flight through rain causes erosion damage to exposed parts of aircraft. Neoprene coatings have been used to protect parts against this damage.

Skin friction due to the high speeds of aircraft and the practice of thermal de-icing, have made it necessary to investigate erosion resistant coatings which will withstand temperatures up to 500°F.

All available elastomers and plastics giving promise of withstanding such temperatures have been examined. Of these, only acrylic ester rubbers, Teflon, and silicone rubbers meet the temperature requirement. Teflon is poor in erosion resistance.

The silicone and acrylic ester rubbers, although they do not have as high a degree of erosion resistance as Neoprene in the present stage of development, afford a fair degree of protection even after exposure to 500° F. Inconsistent adhesion has prevented the ultimate in erosion resistance from being realized.

Missouri School of Mines and Metallurgy. WADC TR 53-162,Part 2. ELECTRODEPOSITION OF TITANIUM. Albert W. Schlechten, Martin E. Straumanis, C. Burroughs Gill. AF 33(616)-75. November 1954. PB 111798. OTS $1.50.

A process has been developed for obtaining a di-diffusion coating of titanium on iron, mild steel, copper, and other metals. This coating is very high in titanium content and displays good corrosion resistance. Many of the variables in the process have been investigated and are reported. Data are also presented on the corrosion of titanium in fused salts, the resulting products, and the probable mechanism.

Sam Tour & Company, Inc. WADC TR 54-209. SURFACE TREATMENTS OF LOW ALLOY STEELS. Sam Tour. AF 33(616)-406. November 1954. ASTIA Document No. AD57463. PB 121507. OTS $3.00.

This report is to be used as a guide to Air Force Contractors in the selection of surface treatments of low alloy steels for corrosion and oxidation resistance at a minimum expense in critical materials. Forty metallic and nineteen paint types of heat resistant coatings applicable to plain carbon and low alloy steels are described and evaluated. Relative temperature, corrosion and abrasion resistances are given. Effects on base metal, formability of coated steel, weldability, joining characteristics and costs per square foot of surface coated are shown. A brief description is given of the processes used for applying and the nature of each of the coatings. Available information and sources of supply are given.

The scope of the protective treatments field is too broad to include all coatings; however, as many coatings were covered as possible. Further, because of the specialized applications involving combinations of elevated temperatures and corrosive environments mentioned in this report, many treatments were evaluated in applications for which they were not intended. Therefore it is not to be assumed that the results tabulated herein are equally valid for other applications or conditions of test.

Battelle Memorial Institute. WADC TR 54-325. RESEARCH FOR LOW-APPLICATION-TEMPERATURE, ELECTRICALLY CONDUCTING, TRANSPARENT COATINGS FOR AIRCRAFT WINDSHIELDS AND RELATED COMPONENTS. E.R. Olson, P. Schall, Jr., E.H. Layer, E.H. Lougher, R.E. Heiks. AF 33(616)-342. January 1955.

A survey of various methods for producing transparent conducting films at temperatures below 250°F and development studies of one method were conducted in a search for both low- and high-conductivity films. These films are needed for dissipation of static electrical charges and fog, frost, and ice from safety-glass aircraft enclosures.

Of approximately 12 methods investigated, the one chosen for more extensive study was the thermal conversion of metal films to transparent oxides. Evaporated indium films were successfully converted to transparent oxide films at temperatures below the bubbling temperature of the plastic laminate. Films with resistances below 500 ohms per square, measured in vacuum, were produced, but exposure of the films to air at atmospheric pressure caused the resistances to increase by at least two orders of magnitude.

National Bureau of Standards. WADC TR 54-485, Pt I. ELECTRODEPOSITION OF TITANIUM. Walter E. Reid, Jr., Joseph M. Bish, Abner Brenner. AF 33(616)-53-11. February, 1955. ASTIA Document No. AD 66658.

PB 126613. LC Mi $3.30, Ph $7.80.

Numerous non aqueous solutions were investigated in an attempt to electrodeposit titanium. Some work was also done with zirconium. Ether solutions containing halides, hydrides, borohydrides, and organo-metallic compounds of titanium were the most promising solutions investigated. A mixed type of bath containing both hydrides and borohydrides yielded titanium-aluminum alloys containing about six percent titanium. Similar baths containing zirconium, instead of titanium, were studied. The zirconium baths gave alloy deposits containing up to 45 percent zirconium.

New methods of preparation of titanium and zirconium borohydrides were developed.

United Chromium, Inc. WADC TR 53-271, Pt. 2. DEVELOPMENT OF A SUBSTITUTE ON IMPROVEMENT OF CHROMIUM ELECTRODEPOSITS. Jesse E. Stareck, Angelo C. Tulumello, Edgar J. Seyb, Jr. AF 33(616)-234. March 1955. PB 121264. OTS $4.25.

The effect of a chromium deposit on the fatigue limit of chromium plated, heat-treated steel is correlated with the stress possessed by the chromium deposit. High tensile stress results in severe lowering of the fatigue strength of steel and conversely low stress gives much less fatigue strength lowering.

Numerous stress measurements with both the "spiral contractometer" and "rigid flat strip" methods showed that the stress as measured by the two methods is different for the same deposits, the "rigid flat strip" method giving a lower stress value. Stress changes rapidly with chromium thickness and is correlated with the formation of a crack pattern in a deposit. Deposits possessing fine crack patterns showed compressive stress at thicknesses beyond a few thousandths of an inch.

The baking of a chromium deposit, as commonly practiced to relieve hydrogen embrittlement of plated steel severely lowers the fatigue strength for the usual electrodeposited chromium. This fatigue strength lowering is again correlated with an increase in stress which is due to a shrinkage of the deposit during baking.

This fundamental knowledge about the mechanism of fatigue in chromium plated steels led to the development of baths and plating conditions which give greatly improved fatigue properties for the unbaked plate and also other deposits giving equally good fatigue properties after baking.

National Bureau of Standards. WADC TR 54-485, Pt 2. ELECTRODEPOSITION OF TITANIUM. Walter E. Reid, Jr., C. Agnes Gaudette, Abner Brenner. AF 33(616)-53-11. April 1955. PB 111876. OTS $0.50.

The study of titanium-aluminum alloy baths as described in WADC TR 54-485 Pt. 1 was continued.

The preparation of concentrated titanium-aluminum and zirconium-aluminum alloy baths by other means than use of the difficulty obtainable borohydrides of titanium and zirconium was investigated. This was done by reacting in an ether solution boron trichloride, lithium aluminum hydride, aluminum chloride, and titanium or zirconium tetrachloride. Under the experimental conditions used, this method was not successful.

An investigation was made of the possibility of using alcohols as complexing agents in solutions of titanium halides. No metallic deposits were obtained from this type of bath.

The unavailability of lithium borohydride (used to prepare titanium and zirconium borohydrides) from commercial sources made necessary an examination of methods for preparing this compound and a quantity was prepared.

WADC TR 54-452.
TRANSPARENT ELECTRICALLY CONDUCTING COATINGS FOR THE REDUCTION OF PRECIPITATION STATIC IN AIRCRAFT. L. Harold Bullis. April 1955. ASTIA Document No. AD 66444. PB 122864. LC Mi $2.70, Ph $4.80.

The Wright Air Development Center program for the development of transparent electrically conducting coatings for the reduction of precipitation static in military aircraft is summarized and evaluated. Work carried out by Air Force contractors and other organizations is reviewed and discussed. Although a coating meeting all of the desired requirements for aircraft application has not been developed, some coatings, such as the Lockheed ATC-1 showed considerable promise. This coating was flight tested and was able to resist the forces of rain erosion for a considerable period of time without undergoing much change. However, additional flight tests have shown that the degree of static elimination which can be expected by the use of conductive coatings on windshields and canopies is small and does not provide a worthwhile operational improvement. In view of this fact, work on this program has been discontinued.

M. W. Kellogg Company. WADC TR 54-526. DEVELOPMENT OF AN ORGANIC LACQUER RESISTANT TO FUMING NITRIC ACID. Francis J. Honn, Robert E. Martin, David R. Wolf. AF 33(038)-21253. January 1956. PB 121205. OTS $1.25.

The objective of this contract was the development of a protective coating and sealant for the metal surfaces in the acid tank and ATO compartments of the B-47 aircraft. The evaluation of available coatings indicated polychlorotrifluoroethylene (KEL-F) was the only organic coating material with adequate resistance to fuming nitric acid. However its use was deemed impractical because of the difficulty of application on aluminum aircraft structures.

Research on the modification of KEL-F through copolymerization with vinylidene fluoride ultimately led to the development of Kellogg X-200 resin. Laboratory tests indicate that coating systems of X-200 filleting material and X-200 lacquer are adequate for the intended application. Coatings formed from the X-200 lacquer are far superior, in fuming nitric acid resistance, to any other lacquer coating evaluated.

United States Stoneware Company. WADC TR 54-527.
THE DEVELOPMENT OF A PROTECTIVE COATING RESISTANT TO NITRIC ACID AND HYDROCARBONS. D. F. Siddall, H. L. Cahn, E. Hillier, M. Gunther. AF 33(616)-150. February 1956. PB 121217. OTS $2.75.

Numerous film-forming raw materials from solvent systems and nonvolatile filleting compounds have been screened for their resistance to white fuming nitric acid. Results show Fluorocarbon Resin X-200 from M. W. Kellogg Company is the only available resin meeting this contract requirement for coating and filleting compounds. Air dry, force dry or low temperature bake coatings have been tentatively formulated to provide the required protection. Studies of physical and chemical properties in conjunction with the compounding of this resin indicates the need for additional work to provide a more practical drying cycle and work is being continued concerning this phase.

Filleting compounds have been developed and tested to meet basic requirements. Further studies are being made with regard to equipment and manufacturing methods. Work on all phases, including the refinement of coating, production procedures on filleting compounds, and final cycling tests are to be carried out.

Missouri School of Mines and Metallurgy. WADC TR 53-162, Part III.

ELECTRODEPOSITION OF TITANIUM. Albert W. Schlechten, Martin E. Straumanis, Sheng Tai Shih. AF 33(616)-75. March 1956. PB 111794 OTS $1.50.

Processes are described by which diffusion coatings of titanium can be obtained on iron and steel objects. A fused chloride bath containing a titanium-oxygen alloy powder operated under helium was particularly effective. It was shown that the coating mechanism of this process was only partially due to the formation of titanium lower chlorides. Coatings were also obtained by deposition from the vapor state, using iodide or chloride vapors. The physical properties of the coatings are described.

Battelle Memorial Institute. WADC TR 54-325, Pt II.

RESEARCH FOR LOW APPLICATION TEMPERATURE, ELECTRICALLY CONDUCTING, TRANSPARENT COATINGS FOR AIRCRAFT WINDSHIELDS AND RELATED COMPONENTS. Paul Schall, Jr., Edwin H. Layer, Leonard H. Mauk, Robert E. Barrett, Edwin H. Lougher, Earl R. Olson, Harvey L. Goering. AF 33(616)-2467. March 1956.

Transparent, conducting films of indium oxide on glass were prepared by thermal oxidation (at 250 to 400° F) of evaporated and sputtered indium films. Addition of tin to the films decreased their electrical resistance. Tin-doped films were prepared with resistance as low as 50 ohms per square and with optical transmittance of 75 to 80 percent. The films are photoconductive. However, for the low resistance films (less than 100 ohms per square) the resistance changes only a few percent when the films are light-dark cycled.

The temperature coefficient of resistance of the films is less than +0.04 per cent per degree (F).

Stability tests were conducted on the films under load (up to 1500 watts per square foot) and without load over a wide range of temperatures. Film resistance changes are less than 20 per cent in 150 hours.

United States Stoneware Company. WADC TR 54-527 Suppl 1.

THE DEVELOPMENT OF A PROTECTIVE COATING RESISTANT TO NITRIC ACID AND HYDROCARBONS. D. F. Siddall, R. Garling, E. Hillier, M. Gunther. AF 33(616)-150. June 1956. ASTIA Document No. AD 106911. PB 121 2175S.

X-200 (Kel-F 800) resin, a new fluorinated fuming nitric acid resistant material was formulated as a lacquer and a filleting putty. Application procedures were developed, and resistance of the lacquer coating, the filleting putty, and the complete system in FNA and JP-4 fuel were determined.

Results indicate that the X-200 system is suitable for application on aircraft metals. When force dried at 300° F, FNA resistance approaches that of fused Kel-F. Resistance of the system when air dried is considerably lower but improves slowly over a long period of time as residual solvent evaporates. The coating system softens in JP-4 fuel after 3 days at 125° F.

National Bureau of Standards. WADC TR 54-485 Pt. III.

ELECTRODEPOSITION OF TITANIUM. Walter E. Reid, Jr., Jean H. Connor, Abner Brenner. AF 33(616)-53-11. September 1956. ASTIA Document No. AD 110402. PB 121721.

The study of titanium-aluminum alloy baths as described in previous reports (1) was extended. The possibility of using sodium or potassium borohydride instead of lithium borohydride to prepare the titanium and zirconium borohydrides was examined. Among the new compounds prepared for use in the titanium-aluminum alloy bath were chloroborohydride etherates of titanium. Some work was done on the use of organometallic aluminum compounds in a titanium alloy bath rather than the hydride aluminum bath previously used. Alloys of titanium or zirconium with magnesium were obtained from a hydride type of bath. Codeposition of some metals similar to titanium, namely hafnium and thorium, with aluminum from the borohydride bath was studied for purposes of comparison with the titanium-aluminum alloys.

A number of reactions between reactive organic compounds and titanium salts were performed in the search for a suitable titanium plating bath. This included an unsuccessful attempt to prepare ethyltitanium and a tetraphenyl borate derivative of titanium.

WADC TR 56-385.

SERVICE TESTS OF CHEMICAL RAIN REPELLENTS. A. M. Savio, S/Sgt. D. S. Nantz, 2/Lt. September 1956. ASTIA Document No. AD 110405.

The rain removal systems on many of the current Air Force aircraft are unsatisfactory. Three methods of rain removal are presently being used; jet blast (compressor bleed) method, windshield wipers, and chemical rain repellents respectively. This report is primarily concerned with chemical rain repellents.

Three types of chemical rain repellents were suggested as a low cost, low weight method of providing visibility through rain. These materials were subjected to laboratory and service tests to determine their suitability for current Air Force requirements The three materials tested were equally satisfactory as rain repellents. However, one material was unsatisfactory for field use due to its toxic and corrosive nature.

Two equivalent materials were recommended for Air Force use, the Snell 1728S developed under Navy contract and Type II developed by the Canadian Research Council, respectively. These materials are easily applied and have suitable durability. They have been procured and are in Air Force stock.

Marquardt Aircraft Company. WADC TR 56-250.

SUMMARY OF DEVELOPMENT AND EVALUATION OF INSULATING TYPE REFRACTORY COATINGS. S. Sklarew, C. A. Hauck, A. V. Levy. AF 33(616)-2957. October 1956. ASTIA Document No. AD 110410. PB 121 759.

Initial development of metal reinforced refractory coatings to provide thermal insulation to aircraft structural members operating in the temperature range 2000° to 3000° F is reported. Reinforced refractories 0.080 to 0.15-inch thick, providing thermal drops of 5° to 10° F per 0.001 inch of thickness have been successfully tested in small scale. These reinforced coatings averaged half the density of steel.

WADC TR 55-212 Suppl. 1

RESISTANCE OF ORGANIC FINISHES TO ROCKET BLAST. Alvin M. Savio, S/Sgt. February 1957. ASTIA Document No. AD 118081.

Twenty-five different finishing systems were tested to determine their relative resistance to the corrosive and erosive effects of rocket exhaust gases. The results of these tests indicate that two distinct problem areas are associated with the rocket exhaust effects. These two problems are corrosion and erosion. Standard lacquer and enamel finish systems satisfactorily protect aircraft materials from the corrosive effects of the exhaust gases as long as the coatings are not damaged by abrasion or

heat. None of the coating systems tested satisfactorily protect aircraft materials from erosion by rocket exhaust debris and heat.

Enthone, Inc. WADC TR 56-591.
ICE ADHESION TESTS ON FILMS OF ORGANIC POLAR MATERIALS. W. R. Meyer, E. F. Foley, Jr. AF 33(616)-3444. March 1957. ASTIA Document No. AD 118099.

Those compounds which are known to produce superior water displacing films on metals were tested to determine their efficiency in lowering the adhesion of ice to metal surfaces. These tests were made to determine the feasibility of developing ice repellent materials using these compounds or their homologues. The results of ice adhesion measurements on aluminum, steel and cement surfaces coated with these compounds are presented. Most of the compounds tested reduced the force necessary to remove ice from the surfaces tested, but none of them reduced the adhesion below 18 lb/sq. in. It is concluded that none of the materials reduced ice adhesion sufficiently to indicate that practical ice repellent materials could be developed with these compounds as the major active ingredients.

Midland Industrial Finishes Company. WADC TR 56-622.
HIGH TEMPERATURE PROTECTIVE COATINGS FOR MAGNESIUM. Clyde R. Fitzgibbon, Edward H. Miller, Milton A. Glaser. AF 33(616)-2890. April 1957. ASTIA Document No. AD 118254. PB 131073.

The objective of the work discussed in this report was to develop an air-drying coating system for magnesium that would have film properties equal to or better than present air-drying finishing systems for this metal -- and which would also withstand temperatures up to 500°F. The major testing requirement for the desired coating system was the ability to maintain film integrity for relatively long periods of time when subjected to a cyclic high humidity-high temperature test.

Three coating systems were developed in this work. They are the "preferred" system and two alternate systems. The alternates, while inferior to the "preferred" system in high humidity-high temperature resistance, are "one-package" materials with better color retention than the "preferred" system. The major drawbacks of the "preferred" system are the lack of color retention at high temperatures and the fact that it would have to be supplied as a multi-package material. The "preferred" system is based on an epoxy-polyamide vehicle. It consists of a calcium chromate pigmented primer, containing 35% pigment, and an aluminized topcoat. The vehicles on which the alternates are based are:

1. A styrenated acrylonitrile modified alkyd-silicone copolymer resin
2. A mixture of an epoxy ester and a silicone resin.

This work was conducted under USAF Contract No. AF 33(616)-2890, and was performed by the laboratory of the Midland Industrial Finishes Company at Waukegan, Illinois.

Battelle Memorial Institute. WADC TR 54-325 Pt 3.
RESEARCH FOR LOW-APPLICATION-TEMPERATURE ELECTRICALLY CONDUCTING TRANSPARENT COATINGS FOR AIRCRAFT WINDSHIELDS AND RELATED COMPONENTS. Leonard H. Mauk, Robert E. Barrett, Edwin H. Lougher, Earl R. Olson, Harvey L. Goering. AF 33(616)-2467. April 1957. ASTIA Document No. AD 118184.

This report describes a continuation of the studies described in WADC Technical Report 54-325, Part 2.

Transparent, conducting films on glass were prepared by simultaneous evaporation of indium and tin and subsequent thermal oxidation of the metal films in air at 150 to 200°C. Films with resistance as low as 17 ohms/square and transmittance as high as 88 percent were prepared. Representative specimens had values of 50 to 100 ohms/square and 80 percent transmittance. Greater reproducibility of film properties was attained as a result of the use of improved techniques and equipment and studies of the effect of varying the process parameters.

Large panels (9 x 9 inches) were filmed, using a rotating substrate and a small source located off the axis of rotation. The transmittance and resistance varied only slightly over the surface. The variation can be reduced by relocation of the source.

A study of magnesium fluoride and silicon oxide overcoatings was made in an attempt to increase the transmittance of the conducting films and to confer mechanical protection. Transmittance increases as high as 13 percent were obtained. The hardness of the overcoatings approached but did not exceed that of the conducting films.

The Goodyear Tire and Rubber Company. WADC TR 53-511 Pt II.
DEVELOPMENT OF A HEAT RESISTANT COATING FOR PROTECTION AGAINST RAIN EROSION. Frank A. Jeffries. AF 33(616)-2231. June 1957. ASTIA Document No. AD 130780.

High speed flight through rain causes damage to exposed parts of aircraft. Neoprene coatings have been used to protect parts against this damage.

Skin friction due to the high speeds of aircraft and the practice of thermal de-icing, have made it necessary to investigate erosion resistant coatings which will withstand temperatures up to 500°F.

All available elastomers and plastics giving promise of withstanding such temperatures have been examined. Of these, only acrylic ester rubbers, Teflon, and silicone rubbers meet the temperature requirement. Teflon is poor in erosion resistance.

The silicone and acrylic ester rubbers, although they do not have as high a degree of erosion resistance as Neoprene in the present stage of development, afford a fair degree of protection even after exposure to 500°F. Inconsistent adhesion has prevented the ultimate in erosion resistance from being realized.

Hypalon S-2 will not withstand 500°F, but does afford a fair degree of erosion resistance where temperatures remain below 400°. Here again, adhesion is a problem.

A Goodyear experimental rubber shows promise of being useful as a primer for high temperature coating systems.

WADC TN 57-178.
EFFECT OF SHOT-PEENING PRIOR TO CHROMIUM PLATING ON THE FATIGUE STRENGTH OF HIGH STRENGTH STEEL. Bennie Cohen. June 1957. ASTIA Document No. AD 130821.

The effect of shot-peening prior to chromium plating on the fatigue strength of high strength steel has been studied in an effort to minimize the drastic drop in fatigue strength, approximately 50%, which occurs as a result of chromium plating. Shot-peened, chromium plated steel, 220,000 psi UTS, had an endurance limit 12% higher than the bare unshot-peened steel. Shot-peened, chromium plated steel, 220,000 psi UTS, which was baked after plating, had an endurance limit 18% higher than the bare unshot-peened steel. Shot-peened, chromium plated steel,

280,000 psi UTS, showed only a slight decrease in fatigue strength in both the baked and unbaked conditions when compared to the fatigue strength of the unshot-peened bare steel.

WADC TR 57-185.
CONDUCTIVITY TESTING OF ZINC DUST PIGMENTED COATINGS. Lt. D. S. Nantz. September 1957. ASTIA Document No. AD 131054. PB 131435. Order from OTS $0.75.

Zinc rich coatings have been in use for over 100 years as a protective pigment for steel. Since the effectiveness of these coatings are dependent upon the electrical conductivity from zinc particle to zinc particle and from zinc particle to the steel surface, it is important to have a good conductivity measurement test to determine the protective properties of any zinc rich coating system.

Several procedures were tried before a good conductivity test was developed. These conductivity tests can be then used as a scientific basis for formulation of zinc rich coatings which will provide good galvanic protection to steel.

It was concluded that the conductivity test developed can be used to determine optimum zinc concentration, film thickness, and drying time for any given vehicle pigmented with zinc.

Battelle Memorial Institute. WADC TR 57-317. THE DEVELOPMENT OF TESTING PROCEDURES AND THE EVALUATION OF COATINGS FOR TANTALUM, GRAPHITE, NICKEL AND COPPER. W. A. Spraker, A. E. Weller, R. E. Hess, J. L. Harp, F. L. Bagby. AF 33(616)-3227. September 1957. ASTIA Document No. AD 131070.

Recent developments in the field of aeronautics have focused attention on the behavior of materials under conditions of high heat flux and high temperature. A number of materials, whose physical properties are of interest, must be protected from erosion and oxidation when exposed to a flow of high temperature air. The resistance of these materials and their coatings to thermal shock is also important.

This report summarizes the results of the first year of a program to evaluate the resistance of a number of coatings for copper, nickel, tantalum, and graphite to erosion, oxidation, and thermal shock under high temperature service conditions. Specimens were tested in the throat of a hydrogen-oxygen rocket motor which could produce a maximum heat flux of approximately 3000 Btu/sec sq ft, and a maximum temperature of approximately 5500F.

The results of the research program indicate that several coatings have properties of promise. These coatings include Rokide A, chromium, and chromium-nickel coatings for copper; Rokide A and chromium coatings for nickel; and a Rokide C coating for graphite. In general, flame sprayed and electrodeposited types of coatings appeared to be the most successful. None of the coatings for tantalum that were tested were successful. The failure of coatings on tantalum appears to be the result of oxidation of the tantalum through the coatings; a successful tantalum coating, if developed, should have a low porosity. Tests of coatings applied by vapor deposition indicated that these coatings have relatively poor adhesion and are subject to erosion.

Sherwin-Williams Company. WADC TR 56-80. RESISTANCE OF PROTECTIVE COATINGS TO SYNTHETIC LUBRICATING AND HYDRAULIC FLUIDS. Robert W. Damm, George F. Salathe, William A. McIntyre. AF 33(616)-2919. September 1957. ASTIA Document No. AD 131056. PB 131428.

This report consists principally of a detailed evaluation of the resistance of 56 coatings or coating systems to five synthetic lubricating and hydraulic fluids in conjunction with supplementary tests designed to expose these coatings to the maximum limits encountered by high speed aircraft under service conditions. Test work was conducted by The Sherwin-Williams Company in the Chicago Lacquer Laboratories' Aircraft Finishes Group. Results show the following candidate coatings to have the most promise in resisting maximum detrimental effects of synthetic fluids and accompanying environmental conditions:

Skin Coatings

Candidate Coating No. C-1 - Andrew Brown, Brolite Synflex A-136 Black Enamel.

Candidate Coating No. C-6 - Sherwin-Williams, Experimental White System.

Candidate Coating No. C-55 - Sherwin-Williams, M49 W C4 System.

Engine Coatings

Candidate Coating No. C-31 - Pratt & Lambert, Heat Resistant Aluminum Enamel # 7897.

Sherwin-Williams Company. WADC TR 56-80 Sup 1. DEVELOPMENT OF PROTECTIVE COATINGS RESISTANT TO SYNTHETIC LUBRICATING AND HYDRAULIC FLUIDS. Robert W. Damm, George F. Salathe, William A. McIntyre. AF 33(616)-2919. October 1957. ASTIA Document No. AD 142030. PB 1314285.

This report is a presentation of the work performed in developing both aircraft skin and engine coatings that are resistant to the detrimental effects of synthetic fluids and the accompanying environmental conditions encountered by modern high speed aircraft. As a result of this work the contractor evaluated a skin coating system and an engine coating and developed two engine coatings. The skin coating system is as follows: Primer, Sherwin-Williams Modified Wash Primer, P40 G C1; Topcoat, Sherwin-Williams White Modified Acrylic Enamel, M49 W C5. The three engine coatings are: Pratt & Lambert Aluminum Engine Coating #7897; Sherwin-Williams Aluminum Silicone Jet Engine Coating, M49 S C2; and Sherwin-Williams Aluminum Silicone Jet Engine Coating, M49 S C3. All these systems are capable of closely approaching complete resistance to the severe conditions involved.

Gates Engineering Company. WADC TR 57-158 Pt I. DEVELOPMENT OF WHITE THERMALLY REFLECTIVE RAIN EROSION RESISTANT COATINGS. George K. Vogelsang. AF 33(616)-3027. October 1957. ASTIA Document No. AD 142033. PB 131598.

Radomes and other coverings for housing radar antennae exposed to rain during high speed flight require maximum protection against rain erosion. This study is for the development of improved coatings for exterior plastic components, which will be rain erosion resistant, thermally reflective and have satisfactory weatherability.

White coatings formulated from polyacrylic rubber or Kel-F elastomer yield the best overall diffuse reflectances, while Neoprene has the best resistance to rain erosion.

A white Neoprene coating system has been developed which satisfies the major requirements of this program.

Studies are under way to improve the above coating system and to render it anti-static.

WADC TR 57-342.
EFFECTIVE THICKNESS OF CHROMIUM PLATE ON THE SENSITIVITY OF MAGNETIC PARTICLE INSPECTION. W. Steindorf, B. Cohen. October 1957. ASTIA Document No. AD 142080. PB 131608.

A survey was conducted on small cracked rods to determine the size crack which can be detected by magnetic particle inspection. Chromium plating 2.2, 4.4, and 6.6 mils thick were successively plated to these rods and the rods were examined after each plating. Scotch tape transfers were taken and studied in conjunction with the subsequently measured cracks. The summation of results indicate that 4.5 mils is a practical limitation of the effective depth of chromium plate for magnetic particle inspection of the plated body.

Cornell Aeronautical Laboratory, Inc. WADC TR 57-435.
STUDY AND DESIGN OF SUPERSONIC ROTATING ARM RAIN EROSION TEST APPARATUS. John L. Beal, Norman E. Wahl. AF 33(616)3267. October 1957. ASTIA Document No. AD 142025.

The feasibility of building a 2500 mile per hour rotating arm, rain erosion, test apparatus based upon economic aspects and engineering problems was investigated. It was found that such a rain erosion test facility was practicable and one was designed which consisted of a large, refrigerated chamber evacuated to 0.01 atmosphere pressure in which the test blade, 56 feet in diameter, would rotate.

The problems and calculations leading to the design, as well as the cost of the equipment of a rotating arm test apparatus for three speeds (i.e. 2200, 2270 and 2500 mph) are reviewed.

WADC TR 57-186.
THE EFFECTS OF NUCLEAR RADIATION ON MILITARY SPECIFICATION PAINTS. 1/Lt. Lloyd A. Horrocks. November 1957. ASTIA Document No. AD 142143. PB 131599.

This report is an evaluation of the effects of gamma radiation and heat on several available types of specification and non-specification paints. Reflectance, adhesion, abrasion, humidity, and film condition data are presented on the effects of gamma radiation and heat on silicone-alkyd, fluorocarbon, alkyd, nitrocellulose, phenolic, and epoxy resin-based paints. Silicone-alkyd, alkyd, and phenolic resin-based paints appear to be satisfactory for use after exposure to 1×10^9 roentgens.

Armour Research Foundation. WADC TR 57-195. TEST METHODS FOR MAGNESIUM SURFACE TREATMENTS. Frank W. Pfohl, Howard T. Francis. AF 33(616)-2855. November 1957. ASTIA Document No. AD 142146. PB 131600.

A study was made of test methods whereby the quality of magnesium surface treatments could be determined. Methods which measured the corrosion-inhibiting power of the coating were found unsatisfactory. A method based on measurement of paint adherence after exposure to a corrosive environment, however, was found quite promising. The latter approach evolved the "Gauze-Peel Test," in which a gauze strip is embedded in the organic coating. After exposure to accelerated corrosion, the gauze is peeled from the surface. Both quantitative (peel strength) and qualitative (visual examination of the stripped area) evaluations are easily made. An important observation made during preparation of experimental specimens was that contact between magnesium and steel during Dow 7 treatment leads to rapidly deteriorating painted surfaces.

Gates Engineering Company. WADC TR 57-158 Pt II. DEVELOPMENT OF WHITE HEAT RESISTANT AND WHITE ANTI-STATIC RAIN EROSION RESISTANT COATINGS. George K. Vogelsang. AF 33(616)-3207. December 1957. ASTIA Document No. AD 142185.

Numerous elastomeric coatings have been developed and tested for retention of thermal reflectance and resistance to rain erosion after 100 hours aging at 400°F. Although Neoprene affords the best resistance to rain erosion, it has limited heat resistance, while Kel-F elastomer is the exact opposite.

A method has been developed to utilize ionically conductive white coatings in conjunction with electron conductive coatings for the development of white anti-static rain erosion resistant coatings. Preliminary results have been very encouraging, but considerable additional work is required before white anti-static coating systems are developed to a point where they will consistently conform to the requirements set forth in Specification MIL-C-7439B.

Arctic Research Inc. WADC TR 56-338 Pt I. BIBLIOGRAPHY OF ICE AND FROST CONTROL. Thomas H. McConica, III. AF 33(616)-3156. January 1958. ASTIA Document No. AD 142317. PB 131712.

This report reviews all unclassified publications relating to the control of ice and frost which could be obtained by this laboratory during the period July 1955 to June 1956. In preparing this bibliography an attempt was made to exclude publications not in the area of interest indicated by the title. Otherwise an unwieldy report would have resulted. Where abstracts or original reports could be reviewed a brief summary of the report is given. In addition, when known, the depository and call number of each referenced report is included. Depositories are coded as follows:

AMAU - Air University, Maxwell Air Force Base, Ala.
ASTIA - Armed Services Technical Information Agency, Dayton, O.
(Note: All "AD" and "ATI" numbers refer to ASTIA)
DAL - Army Library, Washington, D.C.
DLC - Library of Congress, Washington, D.C.
DWB - Weather Bureau, Washington, D.C.

All publications have been classified according to subject matter and coded using the arrangement given on page v.

A review of the patent literature is included in Part II of this report.

Arctic Research Inc. WADC TR 56-338 Pt II. BIBLIOGRAPHY OF ICE AND FROST CONTROL. Thomas H. McConica, III. AF 33(616)-3156. January 1958. ASTIA Document No. AD 142318. PB 131699.

This report constitutes an annotated bibliography of patents relating to the control of ice and frost on aircraft. All patents of interest in the U. S. Patent Office under classification list No. 244, subclass 134, up until July 1956, are included. Additional patents have been located through searching Chemical Abstracts, other bibliographies, etc. All patents have been classified according to subject matter and coding using the arrangement given on page v.

A review of unclassified publications is included in Part I of this report.

Stanford Research Institute. WADC TR 57-514. DEVELOPMENT OF ELECTROPLATING PROCESSES TO ELIMINATE HYDROGEN EMBRITTLEMENT IN HIGH-STRENGTH STEEL. J. E. Chilton. AF 33(616)-3429. January 1958. ASTIA Document No. AD 142316. PB 131721.

The embrittlement of cadmium plated high-strength steels e.g., (SAE 4340) by hydrogen is of serious concern to the Air Force. Unexpected failures of highly stressed components have been experienced. It has been found that cadmium plating from a sulfamate, a perchlorate, or a fluoborate bath with addition agents can be accomplished without embrittling the steel, but that the quality and adherence of the cadmium plate emerging from these baths, as compared with the cadmium plate from a cyanide bath, indicates the need for further evaluation before use.

In order to evaluate the effects of various degrees of hydrogen embrittlement on the steel, a sensitive mechanical test method was required. The important criteria were that the test coupon be of such geometry that it could be plated uniformly and that the test would evaluate only the effect of hydrogen embrittlement. Simple bend tests and notched bar tensile tests have been commonly used for studying the embrittling effect. In this program, the bend test, torsion test, and static sustained load beam test were investigated. A static sustained load beam test, using a 9- x 1/4- x 1/2-inch specimen with the grain transverse to the length was adopted. The SAE 4340 specimens were heat treated to 280,000 to 300,000 pounds per square inch tensile strength and the fracture time, under a given stress, was taken as a measure of the degree of hydrogen embrittlement.

It was found that replicate specimens show a large deviation in fracture times in spite of the care that was given to the selection, preparation, and testing techniques. These deviations are due to the normal defects and variables that are present in commercial, air melted, aircraft quality steel.

Gates Engineering Company. WADC TR 57-158 Pt III
DEVELOPMENT OF WHITE HEAT RESISTANT AND WHITE ANTI-STATIC RAIN EROSION RESISTANT COATINGS. George K. Vogelsang. AF 33(616)-3027. January 1958. ASTIA Document No. AD 142286.

Improved white rain erosion resistant coatings were obtained by simultaneously using combinations of Neoprenes, titanium dioxides, the inclusion of moderate quantities of acid accpetors and stabilizers, and the use of extra quantities of antioxidants. White Neoprene rain erosion resistant coatings can be firmly bonded to polyester or epoxy glass fiber laminates using Primers Ga_{co} KV-6006-N or KV-8510-E.

Improved electron conductive coatings for use in the "Series-Discharge" Anti-Static Coating System were prepared by blending white Neoprene non-conductive coating solutions with Neoprene conductive coatings, utilizing Vulcan XC-72, conductive carbon black. Several new white ionically conductive coatings were developed.

A number of "Series-Discharge" Anti-Static Coating Systems were found to possess and retain conductivities in the order of 0.5 and 15 megohms per square when measured after various conditions. Also, one hundred forty-five coating systems were found to possess and retain conductivities in the range of 10 to 100 megohms per square, when measured after similar conditions. The diffuse reflectances of these coatings are generally better than those of non-conductive white coatings, and their resistance to discoloration is substantially improved.

Boeing Airplane Company. WADC TR 58-493. DEVELOPMENT AND EVALUATION OF ROCKET BLAST AND RAIN EROSION RESISTANT COMPOSITE COATINGS PRODUCED BY FLAME SPRAY TECHNIQUES. J. R. Galli, G. I. Wheeler, R. B. Johnson, B. H. Clampitt, D. E. German. AF 33(616)-5284. February 1959. ASTIA Document No. 209913. PB 151 782. Order from OTS $3.50.

It was the purpose of this contract to determine the feasibility of utilizing multilayer coatings to protect metal surfaces from erosive and corrosive effects of rocket blast impingement. These coatings were to be comprised of two or more from the group: metal, ceramic, organic primer, and organic impregnant, with the metal and ceramic layers being applied by flame spray techniques. In the course of this study six ceramics, four powder spray metals, two wire spray metals, four substrate metals, and approximately sixty organic materials were combined into a large number of different coating systems. These coatings were subjected to a wide variety of tests culminating with actual exposure to rocket blast. A total of twenty-six different coating systems were obtained which successfully withstood four rocket blasts without significant deterioration, verifying the feasibility of this technique. The successful coatings were all of the spray material-impregnant type with the most resistant spray materials being the ceramics.

In addition to the work described above, a limited amount of study was directed toward a better understanding of the flame spray process utilizing the techniques of microphotography, high speed photography, and X-ray diffraction, along with certain theoretical derivations. It is felt that the results of this work, although not of extreme usefulness by themselves, represent a useful addition to the total knowledge about this complicated process.

Horizons Incorporated. WADC TR 58-545. RESEARCH FOR COATINGS FOR PROTECTION OF NIOBIUM AGAINST OXIDATION AT ELEVATED TEMPERATURES. Emanuel C. Hirakis. AF 33(616)-5356. February 1959. ASTIA Document No. AD 209914. PB 151 684. Order from OTS $2.00.

Niobium metal and its alloys should have a large number of applications at temperatures above 2000°F wherever superior strength and creep resistance are required. In order for such applications to be realized, a method must be found for protecting the metal from the corrosive and embrittling influences of oxygen bearing atmospheres. This work was originally directed toward the development of a niobium base alloy which would ultimately stand exposure up to 2500°F without undue chemical or erosive attack or loss in its physical or mechanical properties. Testing programs were to be carried out at 2000, 2300, and 2500°F with up to 100 hour exposures. Such alloys were to possess ductility and stand a reasonable amount of thermal cycling. Midway in the program, the testing temperature was restricted to 2500°F and the time, ductility, bonding and thermal cycle behavior requirements were relaxed in order to obtain more useful data for the program. The use of high temperature coating materials and methods was emphasized over alloy development. Coatings were applied primarily by flame spraying and by electroplating procedures. Preparation, testing procedures and results are described for both the alloy development and coating work. In the alloy work the oxidation rate of niobium at 2000°F was reduced by about a factor of 20 by additions of cerium, chromium, titanium, and aluminum. Flame spraying and electrodeposited coatings were developed affording protection to niobium for 4-6 hours at 2500°F.

National Bureau of Standards. WADC TR 58-5, Part II.

COATINGS OF NICKEL-ALUMINUM ALLOYS PREPARED BY ELECTROLYSIS. Dwight E. Couch, Joan H. Conner. AF 33(616)-57-10. May 1959. ASTIA Document No. AD 214-424.

Nickel-aluminum alloy coatings were produced by electrodeposition of aluminum over nickel. The aluminum was plated from baths operated at from 25° to 1000°C. Attempts to codeposit the two metals were not successful. The alloy was superior to nickel in salt spray tests, outdoor exposure tests, and in oxidation tests.

WADC TN 58-302.

TRANSPARENT CONDUCTING COATINGS OF GOLD ON GLASS. Richard E. Pawel, 1/Lt., USAF. May 1959. ASTIA Document No. AD 214386. PB 151 893. Order from OTS $0.50.

This work on transparent conductive coatings for glass was undertaken in order to determine if there were need for further consideration of metallic films for this purpose. Gold was deposited by vacuum evaporation onto glass slides using several subcoat materials in an effort to enhance the desired properties: good mechanical adhesion, high optical transmittance, and low electrical resistance. SiO, CuO, CaO, Sb_2O_3, PbO, CeO, Cr_2O_3, SnO_2, Cu-CuO, and MgF_2 were used as substrate materials, both the substrate and the gold being deposited without breaking vacuum. Of the oxides mentioned, CuO, and SnO_2 substrates materially increased the abrasive resistance of the films, however, still not to the desired level of adherence. Thin films of gold could be produced on these substrates in the range of 50 to 70+ per cent transmittance having resistances from about 10 to 150 ohms per square. In order to increase the transmittance values about 75 per cent, extremely high resistance values were encountered. This abnormally sharp change in the resistance characteristics is thought due to physical discontinuities in the film. Similarly, thin films of initially high transparency and resistance would not stabilize at elevated temperatures. Increasing values of transmittance were measured during heat treatment; however, for films of high transparency the effect was small.

In the range of application requiring greater than 75 per cent transmittance, the electrical stability of the subcoated gold films is poor compared with the properties of doped-oxide films. However, the thicker gold films appear more stable and may be applicable as conductors when very high transmittance is not desired.

Commonwealth Engineering Co. of Ohio. WADC TR 59-88, OTS Release.

DEVELOPMENT OF A METHOD TO ACCOMPLISH ALUMINUM DEPOSITION BY GAS PLATING. Malvern J. Hiler, William C. Jenkin. AF 33(616)-5594. June 1959. ASTIA Document No. AD 214 887. PB 151 923. Order from OTS $1.75.

The deposition of aluminum was accomplished by thermally decomposing vapors of tri-isobutyl aluminum on the surface of cleaned, heated copper test panels in a heated plating chamber.

The test panels were subjected to a preliminary deoxidation by induction heating to 800°F in a hydrogen atmosphere, purging the chamber with argon gas while cooling the sample to 500 degrees, then admitting a mixture of gases containing tri-isobutyl aluminum vapors for varying periods.

The plate produced is substantially pure aluminum, lustrous and ductile, of excellent conductivity, and in thickness up to 4.7 mils. Adhesion varied from poor to excellent.

University of Illinois. WADC TR 52-12, Part II.

HIGH TEMPERATURE ELECTRICAL INSULATING INORGANIC COATINGS ON WIRE. Clifton G. Bergeron, Paul F. Schwarzlose, Arthur L. Friedberg, Robert L. Hallse, David L. Wilcox. AF 33(616)-3943. June 1959.

The program on high temperature electrical insulation on wire, in its second year, covered the further work on processing of coating, coating continuity, multiple coating, formation of coating, oxidation of copper, high temperature electrical properties, and flexibility of coated wire.

It has been demonstrated that impervious ceramic coating of wire by the technique of flow-coating continuously moving wire through a porcelain enamel-type slip is a practicable manner of applying insulating coatings, and that multiple coating, that is, the successive application of coatings upon previously-coated wire, permits the elimination of pinholes and other such defects that would destroy insulation continuity.

It has been further demonstrated, based on the studies dealing with one type of coating, that the oxidation of copper and the associated solution of CuO into the coating is not in itself deleterious; in fact, CuO in the coating may be considered beneficial in stabilizing the coating with regard to changes in electrical properties with change in CuO content and for the development of better high temperature electrical properties.

The studies dealing with apparent flexibility of the coated wire indicated that the thickness of the coating, the ratio of coating thickness to wire diameter, and the degree of bond established between the coating and the wire are the principal variables affecting flexibility of insulation.

Coating formulation work has proceeded to the extent of establishing coatings that (a) have excellent bond on copper wire, and (b) have room temperature electrical properties that approach the target specifications.

WADC TR 59-415.

SUMMARY OF SECOND HIGH-TEMPERATURE INORGANIC REFRACTORY COATINGS WORKING GROUP MEETING. David Roller. June 1959. ASTIA Document No. AD 232 536.

This report summarizes the proceedings of the second High-Temperature Inorganic Refractory Coatings Working Group meeting held jointly by the WADC Materials Laboratory and the Georgia Institute of Technology Engineering Experiment Station on 26-28 May 1959 in Dayton, Ohio. Forty-three technical personnel representing thirty-nine organisations met to present informal technical discussions of their work on inorganic refractory coatings for use over approximately 2500°F and for an informal seminar on the subject. This report describes each organization's presentation, summarizes the seminar, and furnishes a bibliography of recent reports on refractory coatings and related subjects.

Georgia Institute of Technology. WADC TN 58-13, Part II.

HIGH TEMPERATURE INSULATION FOR WIRE. J. N. Harris, J. D. Walton. AF 33(616)-3944. July 1959. ASTIA Document No. AD 216 360. PB 151 944. Order from OTS $1.25.

Aluminum Coatings plated on substrates of copper,

chrome, iron and Inconel were successfully anodized but efforts to anodize aluminum coatings plated over barrier layer metals on copper were unsuccessful.

Since aluminum-clad copper wire has become commercially available, work on aluminum plating of copper wire with barrier layer metals has been stopped.

Two types of sealing coatings for anodized aluminum were considered: (1) frit-resin, (2) colloidal silica.

Proper curing of frit-resin coatings applied to substrates of iron eliminated all reaction with the substrate. Coatings were continuous to the 1300° to 1400°F range but a wetting problem was encountered above 1400°F.

Frit-silicone resin compositions were applied to aluminum wire in two separate coats to eliminate gelling problems encountered when frit and resin were milled together. Electrical properties of frit-silicone and frit-epoxy resin coatings applied to anodized wire were determined.

Colloidal silica was deposited electrophoroetically or anodized aluminum as a possible substitute for frit-resin coatings.

Work on a "one-time at temperature coating" showed possibilities of continuous operation to 900°F. Work was limited to 1100°F, due to the softening of aluminum in this range.

Marquardt Aircraft Co. WADC TR 59-102.
DEVELOPMENT AND EVALUATION OF INSULATING TYPE CERAMIC COATINGS. E. W. Blocker, S. Sklarew, C. A. Hauck, A. V. Levy. AF 33(616)-5441. November 1959. ASTIA Document No. AD 231 732. PB 149 888. Order from LC, Mi $4.50, Ph $12.30.

A metal reinforced ceramic coating system has been developed that will successfully withstand temperatures of 3500°F while providing insulative values for the supporting structure. This ceramic coating system utilizes a corrugated strip of stainless steel as the reinforcement media and phosphate bonded alumina as the refractory medium. The great advantages of a coating system of this nature, aside from high temperature resistance and thermal insulation, is the wide variety of applications wherein it can be utilized.

Attempts have been made to use zirconia as the refractory grain in phosphate bonded reinforced coatings. These attempts have not been wholly successful, due mainly to the poor thermal shock resistance of zirconia. It is anticipated that this drawback can be eliminated.

Several items of research equipment have been designed and built to facilitate study of the various ceramic materials used in this program. These include a differential thermal analysis setup, a high temperature modulus of rupture furnace, and a modification of the existing thermal drop apparatus.

North American Aviation, Inc. WADC TR 59-465.
ELECTRODEPOSITION OF ALUMINUM ON MAGNESIUM. E. W. Cooke, S. Kritzer. AF 33(616)-5854. November 1959. ASTIA Document No. AD 232 138. PB 161 892. Order from OTS $0.75.

This report describes detailed tests made to electrodeposit aluminum directly onto magnesium, using a modification of the non-aqueous bath developed by the National Bureau of Standards. The report shows that good adhesion is not obtained and a reaction occurs between the electroplating solution and magnesium. However, the report indicates that by using a zinc immersion intermediate coating, satisfactory adhesion is obtained. The purity of the aluminum deposited in this manner is greater than 99.8%, with low porosity. Good adhesion is also obtained on copper and steel. These coatings can be satisfactorily anodized.

American Cyanamid Co. WADC TR 59-354.
STUDIES ON THE PROTECTIVE ULTRAVIOLET ABSORBERS IN A HIGH VACUUM ENVIRONMENT. Richard G. Schmitt, Robert C. Hirt. AF 33(616)-5945. January 1960. ASTIA Document No. AD 233 454.

Compounds which are effective in protecting materials from terrestial ultraviolet radiation may, in a high vacuum environment, suffer loss both by evaporation and photochemical degradation from the shorter wavelength ultraviolet radiation. The rates of evaporation in a high vacuum of the ten commercially available protective ultraviolet absorbers have been measured as a function of temperature. Vapor pressures of the solid absorbers were calculated from the rate of evaporation data. The equilibrium vapor pressures as a function of temperature have been measured for the liquid absorbers by the direct spectrophotometric method. The rates of evaporation of three of the more non-volatile ultraviolet absorbers dispersed in cellulose acetate have been measured and compared with the pure absorbers. The volatility of the protective ultraviolet absorbers was found to be sufficiently high in a high vacuum environment to cause serious loss of the pure absorbers in a relatively short time. Absorbers containing more than one hydroxy group were considerably more stable than those containing only one hydroxy group. This high volatility could be reduced by a factor of 100,000 or more by incorporating the absorbers into plastic media such as cellulose acetate. The photochemical decomposition of three of the less volatile absorbers has been studied in air and in a vacuum in the 2000-4000Å region using an AH-6 high pressure mercury arc to simulate sunlight above the earth's atmosphere. The three most nonvolatile ultraviolet absorbers were found to be as photochemically stable under conditions which simulate sunlight above the earth's atmosphere as they are to terrestial sunlight. Radiation of wavelengths less than 3000 Angstroms was found to be more effective in causing photochemical degradation than the longer wavelengths. Oxygen and possibly other components of air were found to have a pronounced effect on the rate of photochemical decomposition, the rate being considerably lower in vacuum than in air.

Georgia Institute of Technology. WADC TN 58-13, Part III.
HIGH-TEMPERATURE INSULATION FOR WIRE. J. N. Harris, J. D. Walton, Jr. AF 33(616)-3944. March 1960. PB 161 788. Order from OTS $1.00.

Normal anodizing of commercial aluminum-clad copper wire did not result in complete anodization of the aluminum due to its thickness. Advantage was taken of the solvent action of sulfuric acid to remove a portion of the excess aluminum, however, complete anodization was not possible due to uneven removal of the aluminum causing exposure of the copper. Best results obtained were an anodized coating thickness of 0.8 to 1.0 mils and an unanodized aluminum layer 0.3 mil in thickness.

The use of electrophoretic deposition of colloidal silica for sealing the pores of anodized coatings was not successful. Better results were obtained by providing colloidal silica sealing by gelling hydrolyzed ethyl silicate solutions on phosphoric acid anodized wire. This resulted in a wire insulation with an average dielectric constant of 2.89 and a dissipation factor of 1.87 percent.

The best insulation system was provided by sealing sulfuric acid-magnesium chloride anodized wire with a frit-resin coating. This wire was capable of operating at 800°F.

Rutgers University. WADD TR 59-14. INVESTIGATION OF THE THERMAL STABILITY OF VARIOUS SLIGHTLY SOLUBLE SALTS AND THEIR PROPERTIES. Edward R. Allen, Julius Ciric. AF 33 (616)-6160. May 1960. ASTIA Document No. AD 210 479. PB 151717. Order from OTS $2.25.

To meet the ever increasing demand of high temperatures, corresponding inhibitive materials are needed. Of the array of materials suggesting themselves the chrome bearing zinc composition, zinc yellow, its cadmium counterpart, two basic chromates of zinc, the alkaline earth chromates, and the alkaline eartch chromito-chromates were studied. The temperature range was restricted to the 1000°C zone.

Zinc yellow showed some deterioration in the 200-250°C range and a substantially complete breakdown between 300-400°C. This was shown by marked color change, appreciable losses in weight and in chromium (VI). The basic chromates of zinc were slightly superior to zinc yellow. The cadmium counterpart of zinc yellow was stable to 550°C-600°C.

The alkaline earth chromates appeared to be more promising. Calcium chromate showed a gradually increasing change from just above 600°C and reached an essentially complete breakdown, accompanied by fusion at 950°C. Strontium chromate showed little change when heated for 12 hours at 1000°C.

Both calcium and strontium chromate are sensitive to impurities, being most sensitive to their respective oxides. Upon calcination of these chromates with their corresponding oxides even more promising products are obtained, the chromito-chromates. The calcium derivatives show an increase in thermal stability with an increase in percentage of the oxide. The optimum composition, however, remains to be determined. Both the calcium and the strontium derivatives are stable for 12 hours at 1000°C. The former show a decrease in solubility over the unmodified calcium chromate, while the one composition of strontium derivative showed an increase in solubility as compared with the unmodified strontium chromate.

Extensive solubility studies were made on many of the materials under investigation. The indications are that this property may be an index of thermal stability.

The conclusion at this time is that the alkaline earth chromates and chromito-chromates deserve further study.

Armour Research Foundation. WADD TR 60-157. ULTRASONICS AND CERAMIC COATINGS. R. R. Whymark, W. E. Lawrie. AF 33(616)-6396. May 1960. PB 171 057. Order from OTS $1.50.

This report describes investigations into the use of ultrasonic techniques to determine strength of ceramic-metal bonds and to locate defects in the bonds. The techniques evaluated have involved the use of both high frequencies (1-10 mc/s), low frequencies (14 kc/s) and the simultaneous use of high and low frequencies. Experiments at low signal frequencies indicate the average bond strength of flame-sprayed zirconium oxide to be about 1500 psi. Promising results have been obtained using high frequency transmitted energy and a Schlieren detection system. Apparatus is designed to generate surface acoustic waves. The application of surface waves to defect detection is considered. An experimental method has been developed to utilize intermodulation between high and low frequency sound waves to indicate bond continuity. Intermodulation techniques obviate the need for repeatable coupling of transducers which is required in direct high frequency transmission methods. Consideration is given to ultrasonic image converter systems.

Arthur D. Little, Inc. WADD TR 60-317. RESEARCH ON CERAMIC COATINGS WITH CONTROLLED REFLECTIVE AND EMISSIVE PROPERTIES. D. G. Burgess, J. R. Jasperse, L. Marcus, W. S. Martin, E. P. Flint. AF 33(616)-6371. July 1960. ASTIA Document No. AD 245 233.

Two types of ceramic coatings were formulated for application to Inconel in order to give: (a) the highest possible emissivity from room temperature to about 1200°C, and (b) the highest possible reflectivity to solar energy (0.4 to 2.0 microns) while having high emissivity in the infrared from 25° to 700°C or higher. Both types of coatings were formulated from a modified barium silicate frit. This frit was opacified with various black spinels to give the (a) type of enamels having total normal emittances of close to 0.8 at thicknesses of 5 mils or greater, with only minor changes over the specified temperature range. The (b) type enamels were made up with equal parts of stannic and ceric oxides as opacifiers. At thicknesses greater than 5 mils, these have hemispherical reflectances varying between 0.62 and 0.67 at wave lengths of 2.6 microns and 0.6 micron, respectively; together with total normal emittances of 0.75 at 400°C and 0.60 at 800°C.

WADD TR 60-126. THE VACUUM-THERMAL STABILITY OF ORGANIC COATING MATERIALS PART I. The Polyurethanes. James J. Mattice. August 1960. ASTIA Document AD 245 327.

This report is divided into two sections, Section 1 is a survey of the basic knowledge of polyurethane chemistry and of the research which has been conducted in studying the synthesis and degradation reactions of these materials. The application of this information in studying the adverse effects of the high vacuum of space and high temperature is emphasized.

Section 2 presents the results of the vacuum-thermal exposures of commercially available, unmodified polyurethane resins. The relationship between structure, cure, film thickness and weight losses of the polymers is discussed. The urethane bond appears to be the major labile species, leading to a characteristic degradation, regardless of structure which is complete at 500°F. The physical appearance and properties of degrading films is good and pigmentation of a film with titanium dioxide has different effects at differing temperature levels.

Battelle Memorial Institute. WADD TR 60-427. INVESTIGATION OF THE FATIGUE PROPERTIES OF MOLYBDENUM UNDER VARIOUS CONDITIONS OF TEMPERATURE, COATINGS, AND STRESS CONCENTRATION. Alexander A. Mittenbergs, Dean N. Williams, Robert I. Jaffee, Horace J. Grover. AF 33(616)-5915. October 1960. ASTIA Document No. AD 249 770. PB 171 617. Order from OTS $2.75.

Fatigue behavior of unalloyed arc-cast molybdenum was investigated in tension-tension fatigue loading on uncoated and coated, unnotched and notched sheet specimens at 1800°F, room temperature, and -40°F. Three oxidation-resistant coatings were evaluated under fatigue loading. Static tensile tests were also conducted on

specimens of the four surface conditions at the three temperatures. The fatigue strength of unprotected molybdenum at 1800°F was in the same range as the fatigue strength of some other high-temperature materials at this temperature. At room and low temperatures, the fatigue strength of unprotected specimens was relatively high. The notch sensitivity of unprotected molybdenum was low at 1800°F, but rather high at room and at low temperatures. All coatings lowered the fatigue strength of molybdenum at all three temperatures. The loss of fatigue strength was higher in the notched specimens. The coatings investigated did not prove to be completely reliable for high-temperature applications under continuous fatigue loading. For short-life high-temperature fatigue loading, however, utilization of coated molybdenum appeared to be possible with proper precautions. Further research is suggested on protective coatings and on molybdenum-base alloys.

B. F. Goodrich Company. WADC TR 55-58, Part VI.
NUCLEAR RADIATION RESISTANT POLYMERS AND POLYMERIC COMPOUNDS. John W. Born. AF 33 (616)-6442. October 1960.

This research includes the following: fundamental studies of radiation energy transfer and of the mechanisms of radiation damage in high polymers; the selection, design, and synthesis of special new monomers to produce new high polymers with outstanding inherent radiation resistance and heat stability; polymerization of said monomers, identification of the resulting polymers, and evaluation of their radiation and heat stabilities; applied studies of the effect of gamma irradiation in air at room temperature on the compression set properties of various rubber compounds, with and without potential antirads present; and static and semi-dynamic radiation testing of "O" rings with attempts to protect the military-approved rubber compounds against radiation damage with antirads.

The work which is reported includes fundamental, basic, and applied research. The results are stated briefly in the summary which follows.

WADD TR 60-386.
SPECTRALLY SELECTIVE COATINGS FOR TEMPERATURE CONTROL OF SPACE PROBES. Robert M. Van Vliet, James J. Mattice, Robert A. Cross. October 1960.

Two special purpose organic coatings were developed for evaluation on a short duration space probe. The coatings required very accurate tailoring of the reflective and emissive properties for temperature control of the space vehicle. The coatings were designed for solar absorptivities (α_s) of 0.53 and 0.94 with emittances of 0.95 and 0.8, respectively.

In order to make the first coating above, absorbing and reflecting pigments were used in combination. It was found that α_s varied with log of the absorbing pigment concentration when using a constant concentration of reflecting pigment. This derived relationship permitted exact control of the solar absorption. The emittance was controlled by selecting pigments that were absorbing in the infrared (even though they reflect short wavelength light).

The latter coating was required to be black in the visible and near infrared but gray in the far infrared. This was accomplished by using pigments that were highly absorbing at short wavelengths and partially transparent at long wavelengths. Aluminum flake was then added to this coating to give improved infrared reflectance (to form a gray) but without changing the short wavelength absorption.

Useful relationships were developed in carrying out these studies. Of greatest interest is the fact that the blending of black and white pigments appears to follow an exact mathematical relationship over a wide range of reflectivities. Also, the selective reduction of emittance of organic coatings with leafing aluminum pigment has been established even for highly pigmented enamels.

U. S. Naval Research Laboratory. WADD TR 60-703, Part I.
THE ULTRAVIOLET DEGRADATION OF ORGANIC COATINGS PART I. Degradation in Air. A. L. Alexander, F. M. Noonan, J. E. Cowling, R. E. Kagarise. MIPR 33(616)-59-21. November 1960.

A research program to develop suitable coatings for space vehicles, and for studying the effects of ultraviolet radiation on typical polymeric coatings in air is described.

The initial study under this program required irradiating a number of typical polymeric coatings with a near-ultraviolet source at ambient conditions for about 300 hours. It was observed that the relative stability of the polymeric materials which were irradiated roughly parallels their relative order of stability to gamma radiation. Infrared data indicate the relative stability, in decreasing order, to be as follows: polystyrene, melamine formaldehyde, urea formaldehyde, styrenated alkyd, soya oil alkyd, silicones, and polyvinyl chloride.

These initial experiments, qualitative and largely exploratory in nature, indicate that infrared spectroscopy affords a satisfactory means of qualitatively following the chemical changes induced by the ultraviolet.

U. S. Naval Research Laboratory. WADD TR 60-703, Part II.
THE ULTRAVIOLET DEGRADATION OF ORGANIC COATINGS PART II. Degradation in Vacuum. A. L. Alexander, F. M. Noonan, J. E. Cosling, Suzanne Stokes. November 1960. ASTIA Document No. AD 252 727.

This report is a continuation of that described in Part I of this series which was largely concerned with the effects of ultraviolet radiation on polymers in the presence of a normal atmosphere. This work now has been extended to include studies of the effects of very short wavelength radiation (i.e., between 1150 and 2000 Angstroms) on polymeric films in a high vacuum. A comparison of the effects of ultraviolet radiation on several polymeric films in air with those obtained in absence of air (high vacuum) point to the possibility that some organic coatings may be much better able to withstand a space environment than normal weathering at the earth's surface. It appears that the dominant effect of ultraviolet radiation in space may be to cause cross-linking, while in the normal atmosphere at the earth's surface numerous volatile scission products are formed, usually as a result of reactions with oxygen and moisture of the atmosphere.

Experimentation of the type described in this and the previous report are continuing.

American Cyanamid Co. WADD TR 60-704.
STUDIES ON THE PROTECTIVE ULTRAVIOLET ABSORBERS IN A HIGH VACUUM ENVIRONMENT. II Richard G. Schmitt, Robert C. Hirt. AF 33(616)-5945. February 1961. ASTIA Document No. AD 258 379.

Compounds which are effective in protecting materials from terrestrial ultraviolet radiation may, in

a space environment, suffer loss both by evaporation and by photochemical degradation from the shorter wavelength ultraviolet radiation. The loss of absorber by evaporation from plastics was found to be negligible for thermosetting resins such as melamine-formaldehyde and polyesters. Evaporation from thermoplastic resins such as cellulose acetate and methyl methacrylate was appreciable for the more volatile absorbers. The photochemical stability of the ultraviolet absorbers has been studied in air and in vacuum in the 2000-4000 Angstrom region using an AH-6 high pressure mercury arc to simulate sunlight above the earth's atmosphere. It was determined that the ultraviolet absorbers are considerably less stable in a space environment and that the loss in stability is due primarily to a higher quantum yield for the short wavelength ultraviolet radiation. A rapid decrease in the rate of decomposition was observed for the absorbers dispersed in polymeric substrates; this was attributed to a decrease in the rate of energy absorption due to the formation of decomposition products of both absorber and polymer. The stability of the organic absorbers can be improved by the use of certain stable ferrocene derivatives. The effectiveness of the ultraviolet absorbers in protecting polyester resins from photochemical degradation under terrestrial and extraterrestrial conditions was studied. The absorbers were found to be much less effective in a space environment due to the strong ultraviolet absorption of the polyester at the shorter wavelength which competes for the incident radiation. This latter effect, the lower effectiveness of the absorbers, is a more important factor than the decrease in photochemical stability for strongly absorbing polymers. At the present time, the use of more transparent polymers containing one or more absorbers is recommended for long exposures to a space environment.

Cornell Aeronautical Laboratory, Inc. WADC TR 58-4.
DEVELOPMENT AND FABRICATION OF RAINFALL SIMULATION EQUIPMENT FOR SUPERSONIC WHIRLING ARM TEST APPARATUS. Roy R. Lapp, Norman E. Wahl. AF 33(616)-5316. February 1958. ASTIA Document No. AD 142330.

The development and method of calibration of spray nozzles for rainfall simulator, operating in a vertical plane, are reviewed. The design of the spray ring and its fabrication are described.

University of Michigan. WADC TR 57-150 Pt I.
EFFECT OF PRIOR CREEP ON MECHANICAL PROPERTIES ON AIRCRAFT STRUCTURAL METALS. (2024-T86 Aluminum and 17-7 PH Stainless). Jeremy V. Gluck, Howard R. Voorhees, James W. Freeman. AF 33(616)-3368. ASTIA Document No. AD 150956. PB 131 716 Order from OTS $2.50.

Tests have been performed on two typical aircraft structural sheet alloys in an investigation to study changes in mechanical properties brought about by prior exposure to elevated-temperature creep conditions. Specimens of 2024-T86 aluminum alloy and 17-7PH (TH 1050) precipitation hardening stainless steel were exposed for times of 10, 50, and 100 hours at stresses giving up to 3% total deformation, using temperatures of from 350° to 500° F for the 2024-T86 and 600° to 900° F for the 17-7PH.

Following the exposures, short-time tensile, compression, or tension-impact tests were run at either room temperature, the temperature of exposure, or both. The results indicate that the short-time strength of structural materials may be either raised or lowered. The changes in properties may approach as much as 50 percent of the original value. The direction of the change depends on the material, test temperature, creep exposure conditions, and property being measured.

From the standpoint of the structure's designer the most important changes found to date are a large drop in strength for 2024-T86 after prior creep exposure for times of from 10 to 100 hours and an apparent decline in the room temperature ductility of 17-7PH (TH 1050 condition) after prior creep exposure for 100 hours at temperatures near 600° F.

National Cash Register Company. WADC TR 57-296.
MARKING MATERIALS FOR RUBBER, PLASTIC FILMS AND COATED FABRICS. Harry N. Vosmer. AF 33 (616)-3624. February 1958. ASTIA Document No. 150985.

A standard procedure for marking ink evaluation was developed based on the target properties of the contract and the laboratory techniques established by the contractor.

Six inks from commercial sources were found to be acceptable for use as marking inks on the majority of the coated fabrics and plastic films.

Four inks developed in the laboratories of NCR proved to be acceptable. Two of these are based on acrylic resins, and two on a polyvinyl butyral resin system.

None of the ten inks recommended are entirely satisfactory for use on polyethylene or Cotton-vinyl coated, Type II, Class C, Specification MIL-F-4143, since they fail the adhesion and immersion tests.

Titanium adhesion promoters were investigated but were found to contribute little toward the improvement of adhesion on the plastic films and coated fabrics.

Work with treated polyethylene indicated a slight increase of adhesion over the untreated material. Printing on polyethylen still remains a serious problem, since the inks developed and evaluated in this report have not proved satisfactory on this material.

Vitro Laboratories. WADC TR 58-11.
PREPARATION OF PROTECTIVE COATINGS BY ELECTROPHORETIC METHODS. Allen C. Werner, Robert J. Abelson. AF 33(616)-5002. February 1958. ASTIA Document No. AD 150970. PB 131 726.

An investigation was conducted of the preparation of oxidation-resistant coatings for molybdenum by electrophoretic methods. Multi-layer coatings of 80% nickel - 20% chromium and nickel-bonded columbium and titanium carbide provided good oxidation resistance and good erosion resistance. The ballistic impact resistance requirement was fulfilled by inclusion of a 50% dense, 80% nickel - 20% chromium layer. The effects of sintering and coating thickness on resistance to thermal shock were noted. Attempts to incorporate an intergranular diffusion barrier alloy layer for oxidation resistance at 2000° F proved unsuccessful, although a gold-chromium diffusion barrier layer proved resistant at 1800° F. The coating technique has been refined to permit application of a single layer in one operation instead of two.

National Bureau of Standards. WADC TR 58-5 Pt I.

COATINGS OF NICKEL-ALUMINUM ALLOYS PREPARED BY ELECTROLYSIS. Dwight E. Couch, Jean H. Connor. AF 33(616)57-10. March 1958. ASTIA Document No. AD 151078.

The electrodeposition of nickel-aluminum alloy coatings for the protection of Thermold J. Alloy steel was investigated because of certain properties of the alloy, namely, extreme hardness and oxidation resistance. The alloy was formed by plating aluminum upon a layer of nickel. When the fused salt baths operated above the melting point of aluminum, the alloy was formed directly. When the aluminum was deposited at lower temperatures, the samples were later heated to form the alloy by diffusion. Oxidation data and salt spray corrosion resistance of the alloy-plated steel are given. Optimum protection of the steel was afforded by the alloy formed at 700°C, consisting of 2 mils of nickel and 0.2 mil of aluminum.

Rutgers, The State University. WADC TR 57-752.

THE INVESTIGATION OF THERMOSTABILITY OF INHIBITIVE PIGMENTS. Edward R. Allen, Harold V. Lindstrom. AF 33(616)-5290. March 1958. ASTIA Document No. AD 151012.

This report is the result of a three months study of the heat stability of some corrosion inhibiting alkaline earth chromates. Certain chromates were collected, analyzed and compared as to behavior upon heating. As indicated in the literature, the presence of certain impurities, such as calcium oxide, tends to foster decomposition at high temperatures. The result is that the yellow chromate form is changed in part to a green chromic form with the loss of oxygen.

The behavior of a highly purified form of zinc phosphate was studied for its possible application as a corrosion inhibiting pigment.

Armour Research Foundation. WADC TR 58-203.

PROTECTIVE COATINGS FOR TANTALUM. C. Arne Arenberg. AF 33(616)-3983. July 1958. ASTIA Document No. AD 155717.

The results presented in this report cover the work period 1 March 1957 to 28 February 1958.

The program was established to determine the feasibility of protective ceramic coatings for tantalum under high temperature and high air velocity conditions. The method of coating that proved to be most practical was the "Flame Ceramics" process.

Since no single laboratory test could, at the time, be devised to duplicate the actual conditions three separate test facilities were used: (1) an oxy-acetylene cutting torch, (2) a water stabilized arc plasma, and (3) a liquid fuel rocket exhaust.

None of the tests were completely valid since the shape factor of the test specimen, the time-temperature profile, and gas velocities were not realistic. The tests established by the Chicago Midway Laboratories using the proper geometry and feeding the specimen into the plasma at a predetermined rate constitutes a more realistic approach and should be applied to any future testing programs.

However, the testing program as carried out does establish the feasibility of protective ceramic coatings for tantalu, and points up the importance of a heat sink, particularly at ultra-high temperatures. It particularly points to the need for additional research in this area.

Metcut Research Associates Inc. WADC TR 58-269

HIGH TEMPERATURE COATINGS FOR CHROMIUM HOT WORK TOOL STEELS. Elwood B. Norris. AF33(616)-3916. October 1958. ASTIA Document No. AD 203121. PB 151 423. Order from OTS $2.25.

Chromium hot work tool steels are normally useful to approximately 1000°F provided that they are protected from corrosive atmospheres. A typical 5% chromium tool steel, Thermold J, was used to study the protective qualities of various plated, dipped or sprayed coatings. In addition to investigating the oxidation and corrosion protection, selected coatings were checked for their effect on tensile, stress-rupture and fatigue properties of the base metal at room and elevated temperature. The relative resistance of the selected coatings to abrasion and thermal shock was also studied.

From a corrosion standpoint, the best of the coatings investigated were Nickel-zinc, Aluminizing, Alumicoat and Nickel. Of the selected coatings, Aluminizing and Alumicoat reduced the strength of the base metal because of the high temperatures encountered during coating. Aluminum-silicone paint was the only coating which did not significantly reduce the fatigue strength. Watts nickel had high thermal shock resistance. Electroless nickel had the best abrasion resistance.

WADC TR 58-481

A NEW LOOK AT THE HYDROGEN EMBRITTLEMENT OF CADMIUM COATED HIGH STRENGTH STEELS. Norman M. Geyer, G. William Lawless, Bennie Cohen. December 1958. ASTIA Document No. AD 206386.

A reliable and sensitive test procedure for ascertaining detrimental hydrogen embrittlement as a result of cadmium plating has been established. The test consists of a sustained loaded notched tensile specimen, loaded at 75% of the ultimate notched tensile strength for a minimum of 200 hours.

Detrimental hydrogen embrittlement is shown to be dependent upon the steel alloy, the current efficiency of the cadmium plating bath, and the physical structure of cadmium coating obtained. Current efficiency-current density curves have been obtained for the conventional cadmium cyanide bath, the high efficiency cadmium cyanide bath, the cadmium sulfamate bath, and the cadmium fluoborate bath.

Cadmium electroplating processes for coating high strength steel, 280,000 psi UTS, without detrimental hydrogen embrittlement are given. Vacuum cadmium metallized coatings are also shown to be non-embrittling to high strength steels.

WADC TR 58-199.

THE USE OF BASIC MAGNESIUM CHROMATE PIGMENT IN CORROSION INHIBITING PRIMERS. A. Stanley Dalton. January 1959. ASTIA Document No. AD 207524.

The objective of this work was to investigate the feasibility of using basic magnesium chromate as an inhibitive pigment in metal primers which would withstand temperatures of at least 550°F. A literature survey was conducted to ascertain the amount of work previously done with this pigment. As a result of the references found and the work performed as related herein, it is concluded that basic magnesium chromate does inhibit corrosion though not as efficiently as zinc chromate at normal temperatures. It is, however, more thermally stable in the temperature range 400° - 550°F.

Battelle Memorial Institute. TR 5690.
REPRODUCIBILITY OF HIMIDITY EXPOSURE TESTS. Lewis E. Michael. W33-038-ac-14160. April 1948.

Ohio State University Research Foundation. TR 6519, Part 1.
MATERIALS FOR HANDLING FUMING NITRIC ACID. Frank H. Beck, M. L. Holzworth, and Mars G. Fontana. AF 33(038)-10381. March 1952.

Welded drums of Type 347 stainless steel fail by "knife-line" attack if the heat effected zone (destabilized zone) is subjected to a sensitizing treatment (900° to 1500°F) after welding. Failure by knife-line attack occurs because the columbium carbide in a narrow zone adjacent to the weld goes into solution and remains in solution on subsequent cooling (this effect is called destabilization). Sensitizing treatments cause the precipitation of chromium carbide in the grain boundaries (as is observed in Type 304 stainless steel) and intergranular corrosion results. Failure by knife-line attack has not been observed in Extra Low Carbon Type 347 stainless steel. Type 347 stainless steel is not susceptible to stress corrosion by fuming nitric acid at 160°F.

The high corrosion rates which occur on Type 347 stainless steel in fuming nitric acid at 160°F can be reduced from 100-200 mils per year to approximately 13 mils per year by the addition of aluminum nitrate or aluminum metal to the acid solution in concentrations of approximately 0.1% aluminum or higher.

Titanium, Haynes alloy "25" (L-605) and zirconium show excellent resistance to fuming nitric acid in the temperature range, room temperature to 160°F.

Aluminum and some of its alloys show better corrosion resistance than the stainless steels at 160°F.

Investigation of the properties, such as decomposition characteristics, of fuming nitric acid was begun.

Armour Research Foundation of Illinois Institute of Technology. TR 6591.
CORROSION PREVENTIVE ADDITIVES. M. Feinleib and H. T. Francis. AF 33(038)-9202. July 1952.

A reproducible test for ball bearing corrosion in instrument oils has been developed. Test conditions, including brass-to-52100 steel coupling, simulate field conditions, and correlation with service performance has been good.

Commercial petroleum sulfonates, which are commonly used as inhibitors in instrument oils, may be separated into fractions, some of which are corrosive while others are not. Attempts to characterize these fractions are under way.

Ohio State University Research Foundation. TR 6519, Part 2.
MATERIALS FOR HANDLING FUMING NITRIC ACID. PART 2 - PROPERTIES OF FUMING NITRIC ACID WITH REFERENCE TO ITS THERMAL STABILITY. Mars G. Fontana. AF 33(038)-10381. November 1952.

Corrosion tests were conducted on several metals and alloys in white and red fuming nitric acids at room temperature, 122°, and 160°F. Titanium, titanium base alloys Ti-150A and RC-130-B, zirconium, and Haynes 25 alloy (L-605) were found very resistant to fuming nitric acid at all temperatures tested. Aluminum and some of its alloys show very good corrosion resistance to FNA. Durimet-20 has fair resistance to FNA at 160°F. Stainless steels in the AISI three and four hundred series lose their corrosion resistance to FNA when the testing temperature is increased to 160°F. The nature and mechanism of knife-line attack is discussed in detail. Results of stress corrosion of Type 347 stainless steel, effect of additives to WFNA, and galvanic couple systems are reported.

The preparation of pure nitric acid and the apparatus and experimental procedure for measuring the rate of decomposition and equilibrium decomposition pressure of nitric acid are described. During the measurements the nitric acid is confined over an inert fluorinated hydrocarbon oil in a glass tube. Pressures developed by the decomposition products of nitric acid can be measured accurately from near atmospheric pressure to 2000 psi, over a wide range of temperatures and V/L ratios. Some results of tests carried out at 167°F to check the performance of the apparatus are given which show the general relations between the decomposition pressure, composition and V/L ratio.

Armour Research Foundation of Illinois Institute of Technology. TR 53-16.
CORROSION PREVENTIVE ADDITIVES. E. J. Schwoegler and L. U. Berman. AF 33(038)-9202. February 1953. ASTIA Document No. AD 8961. PB 131459. Order from OTS $3.00.

This project was undertaken with the object of developing new corrosion inhibitors to supplement or replace petroleum sulfonates. A study of petroleum sulfonates was made to determine the nature of the compounds showing corrosion inhibition. Separation of a commercial sodium petroleum sulfonate into certain components was effected by chromatography. Both Attapulgus clay and paper were successful in fractionating the petroleum sulfonates so that physical measurements could be made on the fractions. From these studies, it appears that sodium petroleum sulfonates are alkyl benzene derivatives with the alkyl group in the para position to the sulfonic acid group.

A large number of commercially available organic compounds were evaluated by the NRL Static Water Drop Test and by the use of a galvanic couple system at 95 per cent Relative Humidity at 100°F. Several good inhibitors were found by the Static Water Drop Test method. The test presently used, employing the galvanic couple, is not as efficient for screening as the Static Drop Test method. In spite of this, certain general information concerning the type of organic compounds, which will inhibit galvanic corrosion with the system used, has been obtained. Guided by data obtained from these evaluation tests, information obtained from the chromatography and information gained from experience in the field, a large number of organic compounds were synthesized having corrosion inhibiting properties. These included glyoxalidines, alkyl aryl sodium sulfonates, amine salts of 2-ethylhexoic, oleic, nicotinic, pelargonic, linoleic, and dodecylbenzene-sulfonic acids.

The Ohio State University Research Foundation. TR 6519, Part 3.
MATERIALS FOR HANDLING FUMING NITRIC ACID AND PROPERTIES OF FUMING NITRIC ACID WITH REFERENCE TO ITS THERMAL STABILITY. Mars G. Fontana. AF 33(038)-10381. January 1954.

Polarization and corrosion studies were made on galvanic couple systems in WFNA. Aluminum was found to be anodic to stainless steel in the aluminum-stainless steel couple; this resulted in rapid corrosion of the aluminum and almost complete protection of the stainless steel. A theory on the mechanism of anodic corrosion of aluminum is proposed. The effect of water additions to WFNA on the corrosion of couples is discussed. Corrosion studies on

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welded Type 321 stainless steel show this material to be susceptible to knife-line attack similar to that reported for Type 347 stainless steel. An apparatus for studying the erosion-corrosion of metals and alloys in FNA is described.

A series of measurements of the rate of decomposition and the equilibrium decomposition pressure of pure nitric acid were carried out in glass tubes. The effect of the ratio of vapor volume to total volume of the sample, V_g/V, on the rate and equilibrium pressure was studied in detail at 76°C. The reversibility of the decomposition reaction when carried out at constant volume conditions was satisfactorily demonstrated. Evidence is given to show that the equilibrium pressure at a given temperature and V_g/V ratio, is the same whether the equilibrium is approached from a higher or lower temperature. A study of the kinetics of the reaction at 76°C indicates that the reaction occurs in the liquid phase and is one of the first order.

Armour Research Foundation. TR 53-16, Part 2.
CORROSION PREVENTIVE ADDITIVES. E. J. Schwoegler and L. U. Berman. AF 33(038)-9202. March 1954. PB 121113. Order From OTS $4.25.

This project was undertaken to develop new corrosion inhibitors that may supplement or replace petroleum sulfonates. Wright Air Development Center Technical Report 53-16 Pt 1 described a number of types of organic compounds which had been effectively screened as corrosion inhibitors by the Static Water Drop Test. This evaluation study has been continued with major emphasis on the type of compounds which previously were shown to be effective inhibitors, and the minimum effective concentration was determined. With certain amine salt inhibitors a study was made to determine the effect of heating at 150°C for three hours on corrosion inhibition.

A large number of organic compounds were screened by the use of a Galvanic Specimen Test also in order to discover inhibitors that might be effective for inhibiting corrosion in the bi-metallic system, 52100 steel-Muntz metal brass. In the course of this investigation, conditions of temperature, humidity, etc. were studied to arrive at conditions best suited for screening these compounds.

During the course of this program, the initial studies were made on the mechanism of corrosion inhibition of 1020 steel. In the elucidation of the mechanism of corrosion inhibition, attempts were made to relate effectiveness to the structural and functional group characteristics of the molecules studied. The compound types studied included morpholine derivatives, aliphatic amines and amine salts of various carboxylic acids in both AN-O-6a and Mil-L-6085A base oils.

Many organic compounds were synthesized to assist in the development of new inhibitors and in the elucidation of the mechanism of corrosion inhibition. These included morpholine and ethylenediamine derivatives, amine salts of mono- and dicarboxylic acids, fatty acid derivatives and substituted glyoxalidines.

An infrared study on 2- and 1,2-substituted glyoxalidines was made in an effort to arrive at an effective method of verifying these structures. This was essential since certain glyoxalidines were found to have potential inhibiting properties sufficient to warrant further investigation of this type of compound.

Armour Research Foundation. WADC TR 53-16 (Part 3).
CORROSION PREVENTIVE ADDITIVES. E. J. Schwoegler, L. U. Berman. AF 33(038)-9202. December 1954. PB 121108. Order From OTS $1.75.

This project was undertaken to study the development of new inhibitors that may replace or supplement petroleum sulfonates. The work was done by the Organic Chemistry Section of the Department of Chemistry and Chemical Engineering at Armour Research Foundation.

The evaluation of various effective inhibitors against corrosion of SAE 1020 steel in combination with sulfonates has shown that fair inhibition by one sulfonate, is improved by the addition of a small quantity of another inhibitor, usually an amine or amine salts. The amount added was less than that required for good inhibition by either compounds alone.

Over 200 compounds have been evaluated in the JAN-H-792 cabinet. Twelve of these passed the 100-hr requirement. Before using this test method, a study was made to determine reasons for lack of correlation between the results of the Armour Research Foundation cabinet and those of the Wright Air Development Center cabinets. Methods of correlating the cabinet conditions were found so that those compounds listed as passing would also pass the WADC JAN-H-792 test used in qualifying certain specification formulations. Six formulations containing barium dialkyl naphthalene sulfonate, phenothiazine and certain amine salts were also made which passed the 100-hr requirement.

The study of the mechanism of corrosion inhibition as related to structure and functional groups of the polar organic compounds was made on the data obtained from the galvanic system tests with a 52100 steel disk and cartridge brass clip. Most of the information was obtained from the tests conducted at 95% RH at 100°F over a 4-week period in the American Instrument Company cabinet. From the study in MIL-L-6085A base oil the sulfonates, certain amines, amine salts of carboxylic acids, certain oxidized petroleum fractions and certain fatty acid esters of pentaerythritol constituted the effective structures and groups. In the petroleum base oil, AN-O-6a, sulfonates of lower molecular weight, certain aliphatic amines, polyamine salts of carboxylic acids, certain N-alkyl- and aryl-substituted morpholine salts of carboxylic acids, glyoxalidines, and a few esters were effective.

Several additional compounds were synthesized from data obtained from previous test results but these did not show corrosion-inhibiting properties.

Ohio State University Research Foundation. AFTR 6519 (Part 4).
MATERIALS FOR HANDLING FUMING NITRIC ACID AND PROPERTIES OF FUMING NITRIC ACID WITH REFERENCE TO ITS THERMAL STABILITY. Mars G. Fontana. AF 33(038)-10381. May 1955.

Polarization studies were made to study the effect of temperature on galvanic couple systems. These results when used to determine the maximum limiting corrosion rates of the aluminum - stainless steel couple system show good correlation with observed values. RC-70 and Ti-150-A titanium show only a slight decrease in fatigue strength when tested in WFNA. Erosion-corrosion studies show that the corrosion rate increases on aluminum and decreases on stainless steel when exposed to flowing solutions; the effect is more pronounced with increasing rate of acid flow. Results of the "guinea pig" testing program, long time tests in closed containers, are summarized in this report.

A series of measurements of the rate of decomposition and the equilibrium decomposition pressure of pure nitric acid were carried out in glass tubes. The relation between the ratio and vapor volume to the total volume of the sample, the temperature, and the decomposition pressure was established over the temperature range from 76° to 125°C and was extended by extrapolation to room

temperature. The initial results of a similar study of the nitric acid - water system are reported. The results when compared with those of pure nitric acid, show that for the same V^G/V and temperature the addition of water in small amounts to the pure acid materially reduces the decomposition pressure and rate of decomposition. The apparatus and experimental techniques used are described in detail.

Ohio State University Research Foundation. AFTR 6519 (Part 5).
MATERIALS FOR HANDLING FUMING NITRIC ACID. Mars G. Fontana. AF 33(038)-10381. May 1955.

Corrosion fatigue studies were made on RC-70 titanium, titanium alloy Ti-150-A, Armco 17-7PH stainless steel, and 2S aluminum in WFNA (1.5% NO_2) and RFNA (10.5% NO_2) at room temperature. The fatigue life of the titanium and the stainless steel are shortened. Armco 17-7PH was not subject to stress corrosion. Both 3S and 61ST6 aluminum appear to be subject to concentration cell corrosion in WFNA. Polarization data on aluminum and stainless steel agree favorably with actual data obtained from natural galvanic couples of these materials. When aluminum is made anodic to platinum in FNA a phenomenon of anodic passivity occurs at certain values of impressed potential. The corrosion rate of stainless steel can be reduced to a very low value by cathodic protection. These experiments were made using platinum as the inert anode and current densities ranging from 1.5 to 10ma/in^2 depending on test conditions.

Nox-Rust Chemical Corp. WADC TR 55-84.
PACKAGE SAFETY TEST FOR VOLATILE CORROSION INHIBITORS. R. K. Johnston, J. G. Schafer, L. D. McBeth. AF 33(616)2119. June 1955. PB 111848. Order from OTS $1.75.

When a package is assembled using volatile corrosion inhibitors to protect ferrous metal components, there is no known method at present to indicate when the protection drops below a safe level. The development of such package safety tests, which can be applied without opening and repackaging, was the major objective of this contract. All tests were conducted by personnel of the Nox-Rust Chemical Corporation at their laboratories. The development was carried through the laboratory stage, and resulted in two alternate methods, both based on a rust-inhibition test of the package atmosphere. Various limits of applicability of the two methods have been determined in laboratory tests. These methods are recommended for further study in application to actual packaging operations. Differences in the apparent mode of action of currently available volatile corrosion inhibitors have become apparent during the course of the laboratory work; and it is recommended that fundamental studies be made to determine the mechanisms involved.

WADC TN 55-212.
EVALUATION OF CORROSIVITY OF ROCKET BLAST RESIDUES AND CLEANING METHODS TO MINIMIZE CORROSION. Alfred H. Pagano, 1/Lt., USAF. July 1955. ASTIA Document No. AD 74150. PB 131225. Order from OTS $1.25.

Five different cleaning methods were tested in an attempt to remove the residues from six different rocket type blasts on fifteen different metal finish combinations. Results of the test indicate that the use of the standard cleaning method prescribed in T. O. 1-1-1, and a similar type cleaning method tried during this program were most effective in removing residues produced from various rocket residues. The above two methods followed by steam cleaning proved very effective as opposed to steam cleaning alone which was not effective. It was also found that certain rocket blasts were more corrosive to the materials tested than others, and that several of the finishes tested held up very well against rocket blast and may be suitable for application on aircraft to prevent corrosion caused by rocket blast.

Bjorksten Research Laboratories, Inc. WADC TR 54-568.
RESEARCH ON TREATED MAGNESIUM SURFACES. Stanley E. Rohowetz. AF 33(616)-2032. July 1955. PB 121140. Order from OTS $4.75.

Research for the development of an accelerated performance test for treated magnesium alloys included the following corrosion resistance tests:

(1) pH increase in 1.0N KCl.
(2) Open circuit potential comparisons.
(3) Short circuit current comparisons.
(4) Hydrogen evolution rates in 1.0N KCl (gasometric method).

The following tests of adhesion of zinc chromate primer (MIL-P-6889A) to the treated surfaces were also included:

(1) Adhesion in shear (wrought alloys specimens).
(2) Adhesion in tension (cast alloys specimens).
(3) Impact, ultrasonic vibratory, and pressure-sensitive tape tests (qualitative).

The gasometric method was the most promising test for evaluation of corrosion resistance. It was critical in its evaluation if unprimed treated magnesium alloys within five hours and of primed specimens in seven.

The Dow #7 acid chromate treatment gave lower hydrogen evolution rates than the Dow #12 alkaline anodic treatment on all alloys tested.

In the shear and tension adhesion tests, the Dow #7 gave higher adhesion to the primer than the Dow #12.

Corrosion rates for unprimed treated specimens obtained from several weathering tests did not correlate with the rates from accelerated laboratory tests. The corrosion rate of the Dow #7 was nonlinear and increased after a given time due to depletion of the protective chromium ions. Painted specimen rates, however, were in agreement with the accelerated test rates and showed the superiority of the Dow #7 over the Dow #12.

The corrosion resistance of primed-lacquered specimens far exceeded the difference in resistance afforded by the various treatments.

WADC TR 55-109.
CORROSION STUDIES IN FUMING NITRIC ACID. Edson H. Phelps, Fredrick S. Lee, 1/Lt USAF; Raymond B. Robinson. October 1955. ASTIA Document No. AD 90526. PB 133194. Order from LC Mi $3.30, Ph $7.80.

Tests were conducted to determine the corrosion behavior of Armco 17-7PH stainless steel alloy in red fuming nitric acid containing various amounts of hydrofluoric acid as an inhibitor. It was found that hydrofluoric acid additions to red fuming nitric acid markedly reduced the corrosion rate of the alloy. Inhibition was obtained in both liquid and vapor phases at 120°F and 160°F, for periods up to 42 days duration. It was found that a hydrofluoric acid concentration of 0.25% by weight was marginal for good inhibition, and there was some indication that a hydrofluoric acid concentration of 0.75% was optimum. The presence of glass in the system diminishes the inhibiting effect of the hydrofluoric acid.

The corrosion potential of aluminum and stainless steel electrodes was observed in fuming nitric acids with

various nitrogen dioxide and water contents, and with added fluoride. It was found that water additions shift the potential of both aluminum and stainless steel in the anodic direction. Nitrogen dioxide content up to 30% did not produce a significant change in the potential of either electrode. Fluoride additions caused a definite anodic shift in the stainless steel potential, and had a tendency to shift the aluminum potential in the cathodic direction. The observed effects were correlated in a qualitative manner with the previously reported corrosion rate effects of the variables tested.

The Ohio State University Research Foundation. AFTR 6519 Part 6.

PROPERTIES OF FUMING NITRIC ACID. Webster B. Kay. AF 33(038)-10381. January 1956.

The equilibrium decomposition pressure of samples of HNO_3-H_2O mixtures containing 2.50 and 5.00 weight percent water and of HNO_3-NO_2 mixture containing 7.50 and 15.00 weight percent nitrogen dioxide were determined between 85° and 150°C and at vapor volume to total volume ratios varying from 0.05 to 0.8. The decomposition measurements were carried out in a glass tube in a high pressure apparatus under isochoric conditions and with continuous stirring.

The experimental data have been expressed graphically a) as plots of equilibrium pressure vs ratio of vapor volume to total volume, V^G/V, at constant temperature and b) as plots of the equilibrium pressure vs the specific volume of the mixture at constant temperature.

By appropriate cross-plots of the data a series of diagrams showing the equilibrium pressure vs temperature at constant V^G/V ratios for integral values of initial water and nitrogen dioxide content were prepared and high temperature data extrapolated to 25°C.

The kinetics of decomposition of nitric acid were studied by analyzing available pressure-time rate data for different V^G/V values and in the temperature range from 76° to 105°C. Rate measurements were also carried out in the presence of excess nitrogen dioxide and water for different V^G/V values at 85°C and with excess oxygen at an initial pressure of 454 psig and at 76°C.

Under the conditions of the investigation, the decomposition of nitric acid is believed to be a homogenious liquid-phase reaction.

The rate of decomposition can be represented satisfactorily by the equation.

$$\frac{dF}{dt} = Ae^{B(P^{oo}-P)}$$

Where P^{oo} = equilibrium pressure, t = time, e = base of natural logarithms, and A and B are constants. This equation holds over 80% of the reaction. The initial fast rate is followed by a slower apparent-first-order rate with respect to time.

The initial rates are (a) unaffected by addition of up to 15% nitrogen dioxide or by excess oxygen at an initial pressure of 454 psig at 76°C, and (b) lowered considerably by the addition of small amounts of water. The specific first-order rates are increased by excess nitrogen dioxide and oxygen but are lowered by added water.

The experimental energy of activation was found to be 30 kcal/mole, independent of the V^G/V ratio. The pre-exponential factor for the Arrhenius equation was 10^{14} sec^{-1}.

A tentative mechanism has been suggested, consistent with the available experimental data.

WADC TR 56-237.

EVALUATION OF THIN IRON FILMS AS SENSITIVE CORROSION INDICATORS. David Roller, A/lc. June 1956. PB 121536. Order from OTS $1.00.

A requirement exists for a sensitive, easily used, inexpensive, replaceable, and conveniently fabricated direct reading corrosion indicator. Particularly in the field of packaging, a direct reading visual or electrical specimen which could non-destructively determine environmental conditions within all types of containers would be very useful. A preliminary evaluation of the corrosion rates of thin, continuous, adherent vacuum deposited iron films in various humidities to determine their suitability as corrosion indicators is described. Variables investigated included film thickness and film substrate as it affected the corrosion rates in various humidities. It was evident from this work that thin pure iron films corrode readily in high static humidities. Recommendations for further investigations needed to develop a suitable visual or electrical indicator for use in packages are made.

Wyandotte Chemicals Corporation. WADC TR 55-345.

DEVELOPMENT OF AN IMPROVED CORROSION INHIBITOR FOR WATER-ALCOHOL SOLUTIONS. Dwight B. Conklin, Brock G. Peacock, James E. Cole. AF 33(616)-2442. July 1956. ASTIA Document No. AD 97141. PB 131781.

A corrosion inhibitor was developed for use with alcohol-water injection fluid for aircraft engines. It inhibits corrosion of steel, stainless steel and aluminum alloys, is soluble in methanol, ethanol, water or mixtures of the liquids, and lowers surface tension of the mixtures. Although the inhibitor is chemically compatible with hard water solutions, inhibition efficiency is inversely proportional to water hardness. Solutions made with water of more than 100 parts per million hardness, require an excessive amount of inhibitor for inhibition of corrosion. The inhibitor is a mixture of dicyclohexyl ammonium nitrite, urea and 1-nitropropane in an anhydrous methanol solution. Data gathered from initial screening of 150 corrosion inhibiting compounds is presented in detail.

Aerojet-General Corporation. WADC TR 56-310.

INVESTIGATION OF HYDROFLUORIC ACID AS A CORROSION INHIBITOR FOR FUMING NITRIC ACIDS. M. J. Keeler, E. F. Knoll. AF 33(616)-2516. November 1956. ASTIA Document No. AD 110504. PB 121864.

An investigation was made of the effect of small amounts of hydrogen fluoride in inhibiting corrosion by fuming nitric acid. This investigation revealed that corrosion rates of 6061-T6 aluminum and Type 347 stainless steel were generally reduced by a factor varying from about 10 to 100, when an initial HF content of approximately 0.75% by weight was employed. No further reduction in corrosion rate was obtained by increasing the HF content to 2.0%; 0.5% usually provided less inhibition. Therefore 0.75% was selected as the inhibitor concentration for further tests. The effect on the metals of exposure to the inhibited acids was determined in crevices and in stressed specimens, at weld zones, in galvanic couples, in containers with different ullages and with different sizes of vent holes, with various ratios of metal area to acid volume, with acids containing varying amounts of solid matter, and with acids flowing through orifices and impinging against metal at various velocities. The results indicated that acids inhibited with HF are satisfactory for general use. Tests conducted with other metals gave similar results of reduced corrosion in the inhibited acids. Methods for determining HF content and for determining other constituents of acids were also investigated.

Forest Products Laboratory. WADC TR 54-481.

PERMEABILITY OF BARRIER MATERIALS TO VOLATILE CORROSION INHIBITORS AT VARIOUS HUMIDITIES.

J. P. Hohf, A. A. Mohaupt. PO (33-600)-53-4023. December 1956. ASTIA Document No. AD 110632. PB 121893.

The comparative permeability of various barrier materials to certain volatile corrosion inhibitors (VCI) was determined at four relative humidities.

Barrier materials containing a metal foil were the most effective for retaining the VCI vapor. Polyester films and materials that conformed to grade C of Specification JAN-B-121 performed well but not as well as the foil barrier materials. Kraft papers proved ineffective for retaining VCI vapors for long time storage, but certain fiberboards performed well enough to be considered for shipping and short periods of storage.

In general, the volatility and transmission rates of the inhibitors increased as humidity increased. An inhibitor consisting of sodium nitrite and urea absorbed the greatest amount of water, while the oily inhibitors absorbed very little water.

Jet Propulsion Laboratory. WADC TR 56-414. CORROSION AND IGNITION OF TITANIUM ALLOYS IN FUMING NITRIC ACID. John B. Rittenhouse, Nicholas D. Stolica, Stephen P. Vango, Julia S. Whittick, David M. Mason. AF 33(616)-3066. February 1957. ASTIA Document No. AD 118028. PB 121940.

The corrosion, ignition reactions, and stress-corrosion cracking of titanium and its alloys resulting from storage in fuming nitric acid (FNA) were studied. The metal samples were stored in the liquid and vapor phases of various concentrations of the system[a] HNO_3-NO_2-H_2O for periods of time ranging from 1 hour to 90 days at temperatures ranging from room temperature to 71°C.

The susceptibility to ignition reactions, the tendency toward stress-corrosion cracking, and the corrosion rates of the metal were studied as a function of the chemical composition and temperature of the FNA.

The corrosion-time relationships of two titanium alloys in anhydrous FNA (20% NO_2)[b] over a temperature range from 25°C to 71°C were determined. Results of metallographic examinations of the corroded samples to ascertain the corrosion mechanism and the effects of heat treatment of the samples on the corrosion behavior are discussed.

Chemical and X-ray diffraction analyses of the alloys, the FNA used, and the corrosion products developed are reported.

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[a] The symbol NO_2 designates the equilibrium mixture of NO_2 and N_2O_4.

[b] Percentages of composition are in weight per cent throughout this report.

The Ohio State University Research Foundation. WADC TR 56-311. A STUDY OF THE VOLUMETRIC PROPERTIES AND THERMAL STABILITY OF CONCENTRATED NITRIC ACID. Webster B. Kay, Manoj K. D. Sanghvi, S. Alexander Stern. AF 33(038)-10381. February 1957. ASTIA Document No. AD 118034.

1. The phase relations of the binary and ternary mixtures of nitric acid with nitrogen dioxide and water, containing up to 30% by weight of nitrogen dioxide and 10% by weight of water were determined over the temperature range from 85° to 150°C, and a range of V^G/V (ratio of the vapor volume to the total volume of the sample) from approximately the bubble point of 0.8. For this purpose, a small sample of a mixture of known composition was maintained under isothermal and isochoric conditions in a glass tube and measurements of the pressure were made at regular intervals of time until physical and chemical equilibria were reached. The reversibility of the decomposition reaction was established and the effects of the V^G/V ratio, the temperature and the amounts of nitrogen dioxide and water on the pressure were determined within the stated ranges.

2. The pressure, which is primarily due to the oxygen formed as a result of the decomposition, increases with an increase in temperature and with a decrease in the V^G/V ration.

3. The pressure is markedly reduced by the addition of nitrogen dioxide and (or) water, particularly at the lower temperatures. On a weight basis, water is more effective in reducing the pressure than nitrogen dioxide.

4. The V^G/V ratio has a very great influence on the value of the equilibrium pressure of mixtures high in nitric acid content but this influence diminishes with the addition of nitrogen dioxide and water and becomes relatively insignificant in mixtures containing more than 20% of additive (nitrogen dioxide plus water).

5. The effect of temperature on the equilibrium pressure for a given V^G/V ratio, depends on the proportion of nitrogen dioxide in the additive. In the case of the nitric acid-water mixtures, the equilibrium pressure decreases with increasing water content at all temperatures and all V^G/V ratios studied, whereas in the case of the nitric acid-nitrogen dioxide mixtures at temperatures above 125°C and for V^G/V ratios greater than 0.6, the equilibrium pressure is found to increase with the addition of nitrogen dioxide.

6. The rates of attainment of equilibrium are increased by the addition of nitrogen dioxide and decreased by the addition of water. When nitrogen dioxide and water are added in a fixed ratio, the rate constants first show a decrease, pass through a minimum at about 10% of the additive and then increase with a further increase in the additive.

Clevite Research Center. WADC TR 56-380. INVESTIGATION OF THE APPLICABILITY OF HIGH FREQUENCY SOUND WAVES (ULTRASONICS) FOR CLEANING OF PRECISION PARTS. Oskar E. Mattiat, Paschal P. Zapponi. AF33(616)-3011. June 1957. ASTIA Document No. AD 130820. PB 131361.

Grease and preservative compounds on bearings are easily removed by several methods. The important soils, AN-C-124 preservative compound and MIL-G-3278 grease are removed by a thorough spray treatment with Stoddard solvent or an ultrasonic treatment under various conditions using trichloroethylene as the coupling liquid.

The effect of the ultrasonic factors, frequency, intensity, pulse power, coupling fluid and container for holding the parts, upon the rate of removal of field and synthetic soils from complex parts with small openings is determined. Two new methods of study are developed for evaluating available ultrasonic systems and factors, namely, the steel-removal and probe methods.

Results obtained with these methods show that accessible soils of all kinds are easily removed by any of the ultrasonic systems studied; however, inaccessible soils, such as steel particles in bearings and grease soils in blind holes require high sonic intensities and a coupling fluid with optimum cavitating and solubility or dispersability properties for the particular soil.

Low frequency systems appear to be more effective than high frequency systems for removing most types of soils investigated. However, inability to control intensity at the point of cleaning for the high frequency systems make this evaluation uncertain.

The use of pulsed power does not improve the

removal rates of the soils studies.

With the possible exception of an aqueous solution for the removal of steel particles from bearings, trichloroethylene is the most effective coupling fluid texted for the various soils.

Data submitted also show that while a coarse mesh basket for containing parts is not detrimental to soil removal at low frequencies it is at high frequencies.

It is shown by erosion measurements on bearings that the adverse effect of ultrasonic treatment on the starting torque is caused by cavitation erosion; however, the amount of erosion is so small that it may be neglected for the short cleaning time normally required.

Recommendations are made for the cleaning of bearings and other precision parts contaminated with all of the various soils submitted by the contracting agency.

Rocketdyne, A Division of North American Aviation, Inc. WADC TR 57-379.

ROCKET EXHAUST CORROSION SIMULATOR. Frank B. Cramer. AF 33(616)-3165. August 1957. ASTIA Document No. AD 155762. PB 151273. Order from OTS $2.75.

Techniques for studying the corrosive effects of rocket exhause gases upon various material surfaces have been developed. The designs of the recommended items of test equipment and the methods for executing these procedures are presented.

This study was initiated with an analysis of the problem areas: namely, sampling of the hot, high velocity rocket exhaust gases, uniform "simultaneous" exposure of a statistically adequate number of specimens, adequate development of corrosion, evaluation of the corrosion damage on the specimens and statistical evaluation of the utility of the techniques and equipment.

Designs were developed and checked out for a device capable of providing uniform exposure of large numbers of specimens to a single firing of a solid propellant charge. One design is presented for a laboratory device to burn small quantities (one pound) of propellants under very low hazard conditions. Another design was presented for a device capable of uniformly exposing 72 specimens to multiple samples of the hot, high velocity exhaust gases of an aircraft armament rocket.

An evaluation of the extent of corrosion damage to specimens examined by the techniques described in this report showed: that exposure was uniform from specimen to specimen, that differences could be demonstrated between different propellants and between different surfaces, that armament rockets could be compared statistically with propellant burned in a laboratory burner, and that specimens exposed to rocket exhausts during airflight could be qualitatively compared to the specimens exposed to propellants burned in the laboratory style test fixture.

WADC TN 55-212 Sup 2.

RESISTANCE OF ORGANIC FINISHES TO ROCKET BLAST. Sam E. Collis. September 1957. ASTIA Document No. AD 131017.

Forty-seven test panels with various organic and inorganic coating systems were exposed to an impinging blast from free fired, T-214 Rockets. None of the coating systems offered complete protection for the metal. The systems offering the best protective properties were EC 843 (Minnesota Mining and Manufacturing Company) applied over zinc chromate primer or over wash primer-zinc chromate primer. Ceramic coatings or drying oil enamels and varnishes applied over zinc chromate primer, were found to rank second in protective properties. The other coating systems, which were, in most cases, applied without a corrosion inhibiting primer, provided only fair protection.

University of Dayton Research Center. WADC TR 57-540.

THE DESIGN AND CONSTRUCTION OF A SPECIAL TEST FIXTURE FOR THE STATIC EVALUATION OF THE CORROSIVE EFFECTS OF BORON OXIDE AT HIGH TEMPERATURES. Charles R. Andrews. AF 33(616)-3737. December 1957. ASTIA Document No. AD 142263. PB 131 667.

The design and construction of a specialized high-temperature corrosion test fixture is described herein. The fixture is arranged to provide for the long-time exposure of text materials to static environments consisting of cyclic immersion in molten boron oxide (B_2O_3) at temperatures up to 3000°F under closely controlled conditions. Features of the test fixture include an extensive electrical heating control system and an automatic specimen cycling mechanism. The fixture can be operated on a continuous basis with a minimum of attention.

A description of the operating characteristics of the fixture and details of operating procedures are included in this report. A list of engineering drawings pertaining to the test fixture, as well as an outline of maintenance procedures to be followed, are contained in appendices.

Bell Aircraft Corporation. WADC TR 57-302.

INVESTIGATION OF CORROSION INHIBITORS FOR FUMING NITRIC ACID. William H. Bergdorf, Edward J. Kinsey, Jr. AF 33(616)-3056. April 1958. ASTIA Document No. AD 151116.

The major effort of this investigation was to develop a corrosion inhibitor for fuming nitric acid that has corrosion-inhibiting and scale and sludge-formation properties superior to those of hydrofluoric acid. Ammonium hexafluorophosphate in amount equivalent to 0.30% by weight hydrofluoric acid was found to provide better inhibition to the corrosion of 61S-T6 aluminum alloy, and 17-7PH and AISI Type 304L stainless steels than 0.70% by weight hydrofluoric acid under the test conditions. Optimum inhibitor concentrations for Types I and III specification acids, and inhibitor depletion rates for eight inhibitors including hydrofluoric acid were determined. Effects of inhibitor up to five times optimum inhibitor concentration of each of the several inhibitors were measured in terms of slush or freezing point of inhibited acid media which had been subjected to wide variation in temperature and short time storage conditions. Measurements of the mechanical properties of welded specimens of the aluminum and stainless steel alloys that had been immersed in the liquid and vapor phases of eight inhibited acid systems for thirty days at 120°F showed that no significant change in properties was effected by these exposure conditions. Film-formation studies were conducted on aluminum and stainless steel specimens that had been exposed to the liquid and vapor phases of the several inhibited systems and subsequently rinsed in water and methylene chloride. Both rinses removed the inhibited acids from the specimens but methylene chloride failed to remove corrosion products.

Hydrofluoric acid phosphoric acid plus hydrofluoric acid, and ammonium hexafluorophosphate at optimum concentrations dissolved in specification Types I and III acid media were tested in vessels fabricated from the aluminum and stainless steel alloys for a period of 60 days at 70°, 120°, and 160°F. Ammonium hexafluorophosphate produced the greatest pressures during the test period. This inhibitor, however, suffered the

smallest loss in concentration and in general provided the best inhibition.

Georgia Institute of Technology. WADC TR 57-662.
THIN METAL FILMS AS CORROSION INDICATORS. Richard B. Belser, Frank E. Hankinson. AF 33(616)-3879. May 1958. ASTIA Document No. AD 155516. PB 131894. Order from OTS $2.25.

In order to investigate the feasibility of using thin metal films deposited on glass or plastic substrates as integrating indicators of corrosive conditions, the corrosion rates of films of nine metals exposed to air atmospheres of controlled temperature and humidity have been studied. Of the metals iron, manganese, copper, nickel, cobalt, strontium, calcium, magnesium and zinc, only films of iron and manganese proved worthy of more than a preliminary survey. A more intensive study of the corrosion properties and rates of iron and manganese films and of the bimetal pairs iron and copper and iron and gold was undertaken.

This investigation disclosed the important role in metal film corrosion of the absorbed gas layer normally present on glass substrates. This layer furnished the oxygen necessary for small particles of the oxides of iron to form at random sites in the film. These subsequently became elements of electrolytic cells in which the iron film acted as the anode in the presence of air at 25°C and 70 percent relative humidity. Destructive oxidation of the film thereafter proceeded by electrochemical processes.

Rates of corrosion for iron films were controllable to a degree by removal of the absorbed gas layer by pre-deposition heating of the substrate to 400°C or by more complete combination of the absorbed layer with the metal of the film induced by post-deposition heating of the film. The former method reduced corrosion rates because of the removal of the oxygen supply for the formation of oxide nuclei, and the latter method increased the corrosion rate because of the greater number of oxide nuclei formed.

Control of the corrosion rates of iron films under specific conditions of temperature and humidity appears feasible by control of the absorbed gas layer and by introduction of cathodic particles either in the form of oxides or in the form of small volumes of a more electrically positive metal deposited at selected sites.

WADC TR 58-227.
CORROSION OF ALUMINUM AND MAGNESIUM ALLOYS IN ALCOHOL-WATER SOLUTIONS, EVALUATED USING THE RANDOM ASSIGNMENT STATISTICAL TECHNIQUE. Harold L. Stevens. November 1958. ASTIA Document No. AD 203381.

The effects of methanol-water and ethanol-water on magnesium and aluminum were investigated. There was no significant difference between the effects of solutions of the two alcohols. The magnesium alloys tested were more resistant to alcohol-water attack than were the aluminum alloy tested. Pure methanol catastrophically destroyed the magnesium alloys. Coupling aluminum to magnesium accelerated the attack on both metals in the presence of alcohol-water solutions. Increased exposure time did not change the corrosion rates in any solution.

The Random Assignment Statistical Technique used in this study reduced the required number of test samples from 1,152 to 84. The technique is limited to homogenous populations of approximately fifty or more samples.

WADC TR 57-743.
DETERIORATION OF MATERIALS ON OPERATIONAL AIRCRAFT. Harold L. Stevens. December 1958. ASTIA Document No. AD 206895.

Deterioration of materials on operational aircraft was studied at twenty-eight Air Force bases at various locations. Findings include data on metal corrosion, packaging, protective coatings and fungus attack with emphasis on aircraft engine corrosion. Relation of deterioration to geographical location is discussed. Recommendations for deterioration control are included.

WADC TR 58-454.
RAIN EROSION FLIGHT TEST PROGRAMS. Samuel A. Marolo, George P. Peterson. May 1959. ASTIA Document No. AD 213 599.

Various aircraft materials have been previously evaluated at subsonic speeds utilizing the "whirling arm" laboratory test method to determine the most satisfactory solution, from a materials aspect, to the rain erosion problem. As a result of this research, several neoprene coating systems were determined to have the most satisfactory rain erosion resistant properties for the protection of structural plastic materials and were required for use on aircraft external plastic leading edges.

Flight test programs in which coated plastics were flown through rain by high speed aircraft at up to 500 mph were initiated at Wright-Patterson Air Force Base and at Cornell Aeronautical Laboratory. The purpose of these programs was to accumulate data on the efficiency of the MIL-C-7439 approved neoprene coatings and to correlate the vast amount of available laboratory test data with service test data.

The results of these programs indicate that (1) while the laboratory test method does not duplicate service test conditions, it does rate materials in their relative order of rain erosion resistance, and (2) the approved neoprene coatings provide the most satisfactory means of protection for external plastic leading edges of subsonic speed aircraft and have an average service life of 3 to 4 hours in high speed flight through rain.

Georgia Institute of Technology. WADC TR 59-149.
THIN METAL FILMS AS CORROSION INDICATORS. Richard B. Belser, Frank E. Hankinson. AF 33(616)-3879. May 1959. ASTIS Document No. AD 214 886. PB 151 974. Order from OTS $1.50.

It is the purpose of this investigation to expand basic knowledge of the corrosion behavior of thin metal films, as outlined in Wright Air Development Center Technical Report 57-662, to the degree that economic and reliable corrosion indicators may be designed.

The corrosion behavior of 614 film specimens, 330 of iron, 224 bi-metal films of iron and copper, and 60 manganese, deposited on glass substrates has been observed during exposure in sealed chambers at 25°C and 70 percent relative humidity and by electron diffraction examination of films stored in the chambers and in room air. These studies have revealed that iron films deposited on glass substrates cleaned by a detergent, chromic acid, and an alcohol rinse do not exhibit visual evidences of corrosion in 60 days. Efforts to meter corrosion inducing nuclei into the iron films by evaporation at high pressure (10^{-1} mm of Hg), deposition by sputtering, over or under-coating the iron films with vaporized iron oxide, or subjecting the film to heating in air or discharges from a Tesla coil were unsuccessful in appreciably increasing the corrosion rates of the films. Coating the substrate with a metallic salt,

exposure of the deposited film to fumes of HCl and deposition of an over or underlying pattern of a copper film did increase the corrosion rates. Films sensitized by a salt or HCl corroded at 25°C when the relative humidity rose above approximately 30 percent. Natural corrosion of a film appeared to be a two-stage process: (1) sensitization of the film by a chemical more active toward iron than oxygen at 25°C, and (2) the gradual destruction of the film by subsequent local electrolytic action. The adoption of a sensitizer method negates step one above and the film becomes primarily an integrator of the conditions of temperature and relative humidity to which the film is subsequently exposed. The inference is that both sensitized and unsensitized films may be needed for a proper indication of corrosive conditions.

Manganese films, in the thickness range 250 to 1500 Angstroms, cleaned by the same method, revealed a corrosion rate related to thickness; films thicker than 1500 Angstroms did not corrode appreciably.

Further work is required to delineate the value of sensitized and unsensitized film corrosion indicators and to establish processing techniques that will reliably produce indicators of known and desired corrosion rates.

National Bureau of Standards. WADC TR 53-192, Part XIII.

MECHANISM OF RAIN EROSION, PART XIII. Mechanism Studies on Neoprene Coatings. Olive G. Engel. AF 33(616)-58-12. July 1959. ASTIA Document No. AD 225 488.

The mechanism by which neoprene coatings fail is of interest because air traffic will be carried on for many years to come in the altitude range in which rain is still encountered and in the velocity range for which neoprene coatings are a solution to the rain-erosion problems. This report is an account of studies that have been made to determine the mechanism by means of which neoprene coatings eventually fail under high-speed rain impingement. Results of tests involving antiozonant applications to the neoprene coatings are encouraging enough to warrant further experiments with such applications.

University of Dayton Research Institute. WADC TR 58-443.

EFFECTS OF MOLTEN BORON OXIDE ON HIGH TEMPERATURE MATERIALS. Charles R. Andrews. AF 33(616)-3898. September 1959. ASTIA Document No. AD 229 444.

The corrosive ability of boron oxide a combustion product of boron-containing high energy fuels, is reported herein. A sequence of thirty environmental exposures of high-temperature alloys and ceramic coatings typical of those available for use in aircraft power plants was completed. These exposures, which resulted in the destructive corrosion of the high temperature test materials, are detailed and discussed in this report. The static exposure conditions consisted of the cyclic immersion of unstressed test materials in molten boron oxide at temperatures from approximately 1600 to 2100°F and in atmospheres consisting of air, air containing water vapor, or helium. Results showed that nickel base, cobalt base, iron base, and Ni-Cr-Co-Fe alloys were corroded at temperatures less than 2163° F by combinations of intergranular corrosion, pitting, and general surface attack. The results indicated that the presence of aluminum as an alloy constituent or as a surface coating can cause catastrophic corrosion of that alloy at relatively low temperatures. The effects of other constituents are not as well defined; however, manganese is suspected of initiating the corrosive reaction in alloys not containing aluminum. The degree and type of corrosion was defined for each alloy by a combination of results from room temperature tensile tests and studies of the corroded microstructures. Several mechanisms of corrosive attack by molten boron oxide are postulated to explain the data.

Allison Division, General Motors Corp. WADC TR 59-317.

EFFECT OF BORON CONTAINING HIGH ENERGY FUEL COMBUSTION PRODUCTS ON THE PROPERTIES OF STATICALLY STRESSED HIGH TEMPERATURE ALLOYS. George L. Vonnegut, William Mahler. AF 33(616)-5753. January 1960. PB 161 658. Order from OTS $1.75.

This report presents details concerning the development and operation of test apparatus, and the results of metallurgical evaluation to determine the effects of boron containing (TMBO-TMBA) high energy fuel combustion products on the stress rupture behavior and structures of representative nickel, cobalt, and molybdenum base high temperature alloys.

Specifically, the program was designed to determine: (1) Whether rapid corrosive attack by boron oxide, B_2O_3, is dependent on temperature alone or on a combination of stress and temperature factors; (2) Are the alloys in question suitable for use in engines which burn boron containing high energy fuels; (3) What are the temperature, stress, and operating time limitations.

The selected alloys were divided into three groups with reference to the maximum temperature at which they usually employed: Group I - J1570, Hastelloy C, Inconel, M252, Waspalloy - 1600°F; Group II - Hastelloy X, L 605, Incc 713, X40, GMR 235D - 1800°F; and Group III - Molybdenum with oxidation resistant coating - 2000°F.

The results of tests demonstrated that the stress rupture behavior of either stressed or unstressed Group I and Group II alloys was not affected by the boron containing high energy fuel combustion products in times up to 50 hours at the respective exposure temperature.

University of Dayton. WADC TR 59-205.

FURTHER INVESTIGATION OF THE EFFECTS OF MOLTEN BORON OXIDE ON HIGH TEMPERATURE MATERIALS. Joseph W. Rosenbery. AF 33(616)-3898. January 1960. PB 161703. Order from OTS $2.50.

The corrosion resistance of a group of high temperature materials was evaluated during cyclic exposure to boron oxide, a combustion product of boron-containing high energy fuelr (HEF). A series of forty-six cyclic exposures of high temperature alloys and ceramics typical of those available for use in aircraft power plants as well as several experimental materials were completed. The results of the first thirty tests have previously been reported in WADC TR 58-443. The sixteen subsequent exposures and the effects on various materials are detailed and discussed in this report. Exposure consisted of cyclic immersion in B_2O_3 at temperatures of 1750°F - 2200°F for periods up to 145 hours, in air atmosphere.

Results showed that the nickel, cobalt, iron and mixed base alloys tested were incapable of resisting corrosion at temperatures above 2100°F and that siliconized SiC was the only material capable of resisting corrosion during 50 hours continuous exposure at 2200°F. The metals all exhibited surface attack and pitting, and in some cases, intergranular penetration. The results indicate Al, Mn, and C as alloy constituents are detrimental to corrosion resistance, while Si may produce some beneficial effects. The degree and type of corrosion for each alloy was defined by room temperature tensile properties and studies of the corroded

microstructures. Several possible mechanisms of attack by molten boron oxide are postulated to explain the data.

Thermodynamics Laboratories. WADC TR 53-173, Part VI.

A STUDY OF RAIN EROSION RESTING METHODS FOR SUPERSONIC SPEEDS. Donald E. Hurd, Roy F. Holmes. AF 33(616)-3421. January 1960. ASTIA Document No. AD 234 776.

To better understand the mechanisms by which materials passing through rain at supersonic speeds are damaged, the results of numerous types of impacts on metals were analyzed.

An equation which related total energy of impact to the volume of metal displaced was derived and found adequate to explain damage in the velocity range from less than one foot per hour to greater than Mach 3. This equation together with results of incidence angle tests led to an overall damage equation which was successfully applied to the problem of multiple drop rain damage. Principal parameters are target material tensile strength; impacting material shape and mass; angle of incidence; and the velocity of impact.

Facility improvements and test method refinements are described.

Crucible Steel Co. of America. WADC TR 60-115.

CORROSION OF SUPERALLOYS BY SELECTED FUSED SALTS. A. Moskowitz, L. Redmerski. AF 33(616)-6196. March 1960. PB 161 848. Order from OTS $2.25.

The corrosion of Inconel X, Inconel 702, Rene 41, M-252, and Haynes 25 by potassium chloride and lithium fluoride at 1600 to 1900°F was studied. Thin coatings of the salts (1.5 mg/cm^2) caused severe corrosion of the alloys in air, which resulted in accelerated failures of thin sheet specimens in creep-rupture testing. Rankings for the alloys based on creep-rupture tests were similar for uncoated and salt-coated materials: Haynes 25, Rene 41, and M-252 best, Inconel 702 poorer, and Inconel X poorest.

The corrosion products consist of oxides and spinels, and only very little corrosion, if any, occurs without oxygen. The presence of the salt prevents the normal formation of a protective oxide film. X-ray diffraction studies showed differences between the normal oxidation products and the oxide corrosion products produced with salt present.

The corrosion occurs as severe surface attack with consequent eroding away of metal, as intergranular penetration, and as internal voids formed in the alloy. All of the alloys were susceptible to each of these types of corrosion. Grain boundary separation effects due to stress (2,500 to 10,000 psi) were also found.

Georgia Institute of Technology. WADC TR 59-759.

THIN METAL FILMS AS CORROSION INDICATORS. Richard B. Belser, Niels Engel. AF 33(616)-3879. March 1960. PB 161 757. Order from OTS $2.00.

The purpose of the research conducted was to study the properties of thin metal films in relation to their proposed use as integrating indicators of corrosive conditions existing over a period of time within sealed packages. In particular, the evaluation of iron films as corrosion indicators was desired.

During the period of this report over 500 films deposited on glass substrates were examined by exposure in sealed containers at 25°C and at known relative humidities in the range of 0 to 70 percent; electrical resistance changes and area destruction were recorded. The specimens consisted predominantly of iron films but included approximately 80 films of manganese, 30 films of magnesium, and 50 bimetal films of iron and copper.

The observed behaviors confirmed results previously reported and extended knowledge of the basic corrosion behavior of thin metal films. In short, thin iron films deposited by vacuum evaporation onto glass slides which are essentially clean did not corrode in a manner observable to the eye at 25°C and 70 percent relative humidity in periods greater than 60 days. Careful electrical measurements, on the other hand, indicated small electrical resistance changes of one to five percent during the same period. Efforts made to increase corrosion rates by roughening the substrate, placing the film in tension, partially oxidizing the film, or employing bimetal configurations of iron and copper were only partially successful in that the corrosion rate was increased by a factor of two or three but data scatter remained large.

Iron film specimens sensitized with a salt spray such as NcCl or HCl acid fumes exhibited rapid corrosion rates, i.e., extensive area destruction or large resistance increases in 24 hours. Similarly rapid corrosion effects were observed when films were inadvertently sensitized by chemical fumes coming from nearby laboratories through the ventilation system, by fumes released from soldering fluxes, or from acid bottles opened in the laboratory during processing. Films which had been thus sensitized exhibited relatively rapid corrosion at 25°C and at relative humidities above approximately 35 percent; hence, such films became essentially coarse integrators of conditions of temperature and relative humidity.

It was evident that metal films, as usually prepared, were frequently inadvertently sensitized during fabrication and storage and that the various degrees of sensitization experienced contributed to the large data scatter observed. The scatter of corrosion rates prevents use of the films as precisely calibrated individual indicators of corrosion rates; on the other hand, their use as corrosion indicators in statistically valid numbers appears practical. In addition, it is apparent that films register the event of sensitization when exposed at 25°C and above a relative humidity of 35 percent and that sensitized films register the event of rise of relative humidity above 35 percent. Likewise, it is probable that a coarse integration of conditions of terperature and relative humidity may be obtained up until the time the film becomes totally destroyed.

Bimetal films of copper and iron and films of manganese and of magnesium exhibited, generally speaking, essentially the same corrosion characteristics as films of iron with the exception that in general their corrosion rates were slightly greater.

Concurrent studies of bulk metal specimens, and the occurrence of inadvertent sensitization of these specimens at the same time as film specimens, revealed the importance of sensitization effects in corrosion; these implied that large corrosion losses in packaged metal goods could be avoided by proper care in minimizing or removing sensitizing influences during processing and packaging.

A method of studying the corrosion of films of the semi-refractory and refractory metals by measuring the temperature coefficient of resistance of metal films in vacuo and in air over the range 25°C to 600°C was presented. It was shown that only films of gold and platinum survived temperatures of 600°C without oxidation. Films of iridium exhibited only relatively minor

oxidation and films of chromium began oxidizing at about 550°C. Films of other metals exhibited rapid oxidation at 400°C or below. Overcoatings of silicon monoxide provided considerable protection of films of the various metals from oxidation. The method of study outlined appears to be a useful one for further exploring the corrosion properties of the refractory metals and for studying protective coating systems.

National Bureau of Standards. WADC TR 53-192, Part XV.

MECHANISM OF RAIN EROSION - PART XV. Resistance of White Sapphire and Hot-Pressed Alumina to collision with Liquid Drops. Olive G. Engel. AF 33(616)-5912. June 1960.

Data are presented that show the resistance of 0.318-cm (0.125-in) thick plates of white sapphire and hot-pressed alumina to impingement with 0.2-cm-diameter water-drops and mercury drops. In collision with mercury drops, the velocity at which damage was first observed was 3.514×10^4 cm/sec (1,153 ft/sec) for white sapphire and 4.276×10^4 cm/sec (1,403 ft/sec) for hot-pressed alumina; the difference in the velocities found for the two ceramics is not considered to be significant. The velocity required to damage these ceramic materials on collision with a waterdrop was not reached experimentally. A theoretical extrapolation suggests that plates of these ceramics of the indicated thickness can be expected to survive collision with 0.2-cm waterdrops without damage up to a velocity of 33.7×10^4 cm/sec (11,100 ft/sec). For air at 0°C, this is equivalent to a Mach Number of 10.

Nitrogen Division, Allied Chemical Corp. WADD TR 60-384.

NITROGEN TETROXIDE CORROSION STUDIES. C. W. Alley, A. W. Hayford, H. F. Scott, Jr. AF 33(616)-6568. July 1960. PB 171 301. Order from OTS $1.75.

The purpose of this investigation was to determine quantitatively the corrosive effects of nitrogen tetroxide on mild steel, aluminum, stainless steels and titanium. This was done under static conditions at six water concentrations up to 3.2 wt % and four temperatures up to 74°C. The corrosion rates under dynamic flow conditions were also investigated.

The corrosion of carbon steel (ASTM A-285 Grade C) and aluminum (5086) was less than 0.5 mil per year in nitrogen tetroxide containing up to 0.2 wt % water at 74°C, increasing to 50 mils per year at 3.2 wt % water and 74°C. Negligible corrosion was observed under severe conditions with stainless steel (304-L) and titanium (75A and 6Al-4V) whereas high strength steel (PH 15-7 Mo) showed losses of 0.5 to 1.0 mils per year. No stress corrosion cracking was observed in tests of carbon steel, high strength steel or aluminum in nitrogen tetroxide containing 0.1 and 1.6 wt % water at 49°C. Significant corrosion of stainless steel (304-L) occurred in the presence of Teflon.

Dynamic tests showed no significant corrosion of 304-L and PH 15-7 Mo stainless steels and average rates of 0.05 mils per year for aluminum and 0.33 mils per year for carbon steel after 205 hours exposure to commercial nitrogen tetroxide flowing at velocity of 10 ft per second at 30°C.

Air Products, Incorporated. WADD TR 60-436.

THE COMPATIBILITY OF VARIOUS METALS AND CARBON WITH LIQUID FLUORINE. Charles J. Sterner, Alan H. Singleton. AF 33(616)-6515. August 1960. ASTIA Document No. AD 244309.

Experimental studies were made to determine the compatibility and resistance to corrosion of various metals and carbon with liquid fluorine at -320°F. The metals tested were various alloys of aluminum, stainless steel including high-strength steels, titanium, copper, Monel metal, nickel, and magnesium. Tests which were performed included: continuous immersion of stressed and unstressed samples in liquid fluorine for periods up to two weeks; impact ignition of titanium and aluminum in liquid fluorine and of titanium in liquid oxygen at impact energy levels ranging from 2.6 to 65 ft-lb; intensive and extensive impact on tubes containing liquid fluorine; passivation and storage for periods up to 64 days followed by immersion in liquid fluorine; thermal shock of samples, both by liquid fluorine and in contact with liquid fluorine; flexing of metal samples immersed in liquid fluorine; testing of metal samples while immersed in liquid fluorine; the explosibility of contaminant in liquid fluorine.

It was found that the corrosion of the metals tested in pure liquid fluorine was negligible, generally amounting to less than 1 mil penetration per year. However, contamination of liquid fluorine can result in severe corrosion. Graphitic carbon was found to be incompatible although dense amorphous carbon was affected only slightly. Titanium was found to ignite upon impact in liquid fluorine although the ignition did not propagate. No evidence was found to support the theory that a fluoride film is required to protect metals from attack by liquid fluorine.

Passivation by gaseous fluorine is recommended as an extension of the cleaning procedure despite the lack of real evidence that passivation is required for materials which have been thoroughly cleaned.

WADD TR 60-649.

COMPARATIVE EROSION RESISTANCE OF PLASTIC MATERIALS IN A SUPERSONIC ROCKET EXHAUST AND SUBSONIC AIR ARC PLASMA. Herbert S. Schwartz. September 1960. ASTIA Document No. AD 247441.

Representative thermally protective (ablative) plastics consisting primarily of organic and vitreous fiber reinforced materials were exposed in a hyperthermal subsonic air plasma stream and the supersonic exhaust of a rocket fueled with gaseous hydrogen and oxygen.

Linear and mass erosion rates were determined and correlated with materials composition. Relative performance of materials types in two different environments was obtained by establishing an erosion rate ratio parameter.

It was found that the class of materials having best performance was considerably different for the two exposure environments. In the rocket exhaust exposures, the best performing materials were high silica fiber, asbestos and glass fiber reinforced phenolics. In the subsonic arc plasma, the best performing materials were organic fiber reinforced phenolics.

Reaction Motors Division. WADC TR 57-542.

A STUDY OF THE CORROSIVE EFFECTS OF THE COMBUSTION PRODUCTS OF BORON CONTAINING FUELS ON SELECTED HIGH TEMPERATURE MATERIALS. AF 33(616)-3713. September 1960. PB 161 421. Order from OTS $5.00.

The corrosion behavior of a wide cross section of high temperature alloys, under unstressed conditions, and some ceramics and cermets, in the combustion products of boron-containing fuels has been examined. Initially, static tests were carried out on alloys in contact with molten boric oxide and yielded preliminary information

on the corrosion process.

The major effect of the program was devoted to synamic testing. Specimens were exposed to a continuous flow of combustion products from commercially available fuels (trimethylborate azeotrope and trimethoxyboroxine).

Test conditions were adjusted to simulate the combustion products from a potential boron-containing high energy duel. A special unit utilizing a ceramic liner was designed and constructed so that many long-term tests (150 hr) could be carried out in the range 1500-2500°F without the destruction of the unit.

A method is presented for treating in a quantitative manner the corrosion data obtained, as is a rating of all the alloys tested. The data show that for all alloys there exists a critical temperature (Tc) in the range 1600 to 2000°F above which the corrosion rate increases very rapidly with increasing temperature, and above which serious pitting, and even destruction, occurs within 150 hours. The rate of corrosion below Tc has been determined. A characteristic value, A, representing this rate has been listed for many alloys and tentatively related to the elemental composition. The effect of velocity of combustion products on the corrosion process has been discussed. Mechanisms of the process are presented. Finally, data on the cleaning of deposited boric oxide by saturated steam are included.

*Milan University (Italy).
METAL CORROSION, G. BIANCHI. Project 9761(802), Contract AF 61(052)-260; AFOSR, DCS.

Oxygen and hydrogen peroxide reactions with non-metals, metals and metal oxides are being studied, including interactions of electrochemical processes with reactions of oxidizing solutions.

Design Criteria

TR 5144, Part 1.
INVESTIGATION OF STRESS CORROSION. PART I - TEST METHODS AND PROGRESS. Baxter C. Madden, Major. September 1944.

TR 5311.
METHOD OF TESTING THIN SHEET MATERIAL IN COMPRESSION. Baxter C. Madden, Major. October 1945.

TR 5521.
METHOD OF TENSILE TESTING AT ELEVATED TEMPERATURES (200°F - 600°F.) Baxter C. Madden, Major and E. L. Horne. August 1946.

TR 5144, Part 2.
INVESTIGATION OF STRESS CORROSION. PART 2 - RESULTS OF TESTS AND CONCLUSIONS INCLUDING A METHOD OF EVALUATING STRESS CORROSION SENSITIVITY. Baxter C. Madden, Major. October 1946.

TR 5662, Part 1.
INVESTIGATION OF MECHANICAL PROPERTIES AND PHYSICAL METALLURGY OF AIRCRAFT ALLOYS AT VERY LOW TEMPERATURES. PART I - IMPACT AND HARDNESS DATA OF SEVERAL AIRCRAFT METALS AT LOW TEMPERATURES. Donald A. Shinn. January 1948.

California Institute of Technology. TR 5742, Part 1 and 2.
RAPID LOADING PROPERTIES OF AIRCRAFT STRUCTURAL METALS. PART 1 - DESIGN AND CONSTRUCTION OF A HYDRO-PNEUMATIC MACHINE FOR RAPID LOAD TENSILE TESTING. PART 2 - INFLUENCE OF RAPID LOAD AND TIME AT LOAD ON THE TENSILE PROPERTIES OF SEVERAL ALLOYS. D. S. Wood, D. A. Elmer, and D. S. Clark. W33-038-ac-14102(15772). December 1948.

P. R. Mallory and Company, Inc. TR 5897.
THE PROTECTIVE COATING OF MOLYBDENUM. A. M. Suggs. W33-038-ac-19697. June 1949.

TR 5899.
TENSILE PROPERTIES OF SOME AIRCRAFT STRUCTURAL MATERIALS AT VARIOUS RATES OF LOADING. Richard F. Klinger. August 1949.

P. R. Mallory and Company, Inc. TR 5947.
MANUFACTURE OF METAL-CERAMIC COMBINATIONS. E. F. Swazy. W33-038-ac-19697. May 1950.

Armour Research Foundation Illinois Institute of Technology. TR 6517, Part 1.
DETERMINATION OF PHYSICAL PROPERTIES OF NON-FERROUS STRUCTURAL SHEET MATERIALS AT ELEVATED TEMPERATURES. D. D. Doerr. AF 33(038)-8681. December 1951.

In order to establish important design criteria, the compressive, bearing, and shear properties have been determined for (1) 24S-T3 aluminum alloy at room temperature and at elevated temperatures between 212 and 700°F, with exposure periods ranging from 0.5 to 1000 hr; (2) 75S-T6 aluminum alloy, FS-1H and MH magnesium alloys at room temperature and at elevated temperatures ranging from 300 to 600°F, for exposure periods of between 0.5 and 1000 hrs; (3) annealed and cold rolled titanium materials at room temperature and at several elevated temperatures between 400 and 1000°F for exposure periods of 0.5 to 100 hr. These properties have been compared with the tensile data in an attempt to establish a possible correlation of the compressive, bearing, and shear characteristics with the tensile properties of the individual material at elevated temperatures.

The test specimens, equipment, and procedure are described in detail. The test results are presented in the form of tables and curves to illustrate the effect of temperature and exposure time on physical properties of the various materials investigated. It is concluded that the results of this investigation will establish reliable data concerning the quantitative relationships between tensile and concomitant mechanical properties of selected structural sheet materials at elevated temperatures.

This is a summary report covering work conducted during the first year of the project. The project is being continued.

University of Illinois. TR 52-89, Part 2.
PLASTIC BEHAVIOR OF ENGINEERING MATERIALS. PART 2 - PARTIALLY PLASTIC THICK-WALLED CYLINDERS. M. C. Steele. AF 33(038)-15677.

August 1952. PB 116840. Order from LC Mi $3.25, Ph $9.00.

This report presents experimental and theoretical work on the overstraining of thick-walled cylinders. Four mild steel cylinders (2:1 wall ratio) were subjected to internal fluid pressure and strains at the bore and the outside surfaces were measured. In addition, the mechanism of flow was studied by polishing the end and outside surfaces for the observation of Lueders lines. A theoretical analysis is given which is based on results from a quantitative comparision of certain previous theories and available experimental data. The solution is in closed form and is applicable to strain-hardening materials.

Observations disagree with theoretical assumptions concerning the progression of yielding; wedge regions of overstrained material, occupying a small fraction of the total volume, characterize the yielding process. Discrepancies with theory are observed in the measured strains; fully plastic load-carrying capacities predicted from theory are higher than those observed in the experiments. Instability of deformation (creep) under maintained constant load is discussed.

It is concluded that theoretical analyses, in their present form, do not cope adequately with the inelastic problem concerning the wedge type of yielding in two and three dimensional, non uniform stress fields. Suggestions are given for further research.

Ohio State University Research Foundation. TR 5662, Part 4.

INVESTIGATION OF MECHANICAL PROPERTIES AND PHYSICAL METALLURGY OF AIRCRAFT ALLOYS AT VERY LOW TEMPERATURES. PART 4 - LOW TEMPERATURE MECHANICAL PROPERTIES, INCLUDING FATIGUE OF TITANIUM-BASE ALLOYS RC-130-B AND Ti-150-A. Joseph W. Spretnak and Mars G. Fontana. W33-038-ac-15698. November 1952.

The yield and ultimate strengths of both alloys increase as temperature is lowered. The increase in ultimate strength at -196°C over the room temperature values is 72% in the case of RC-130-B and 61% in the case of Ti-150-A. Elongation and reduction of area of both materials are decreased at low temperatures. The effect of notching on tensile properties is to raise the yield and ultimate strengths at room temperature and 78°C and to decrease these values at -196°C. Apparently the notched tensile strength passes through a maximum between room temperature and -196°C. Elastic moduli increase as temperature decreases.

Both alloys show low values of impact strength at room temperature, considering their tensile strengths, and the absorbed energy in the impact test drops to low values at temperatures below -78°C.

Ti-150-A shows a remarkable high room temperature fatigue strength of 110,000 psi at 10^7 cycles. The fatigue strength at room temperature of RC-130-B is 84,000 psi. At -196°C these values increase to 130,000 psi for RC-130-B and 141,000 psi for Ti-150-A. RC-130-B is less sensitive to notching in fatigue than TI-150-A, except at -196°C, at which temperature Ti-150-A is less notch sensitive than RC-130-B.

RC-130-B is slightly harder than Ti-150-A at all test temperatures. At -253°C the hardness of both materials increases about 106% over the room temperature hardness.

The thermal expansion coefficient of RC-130-B is higher than that of Ti-150-A at all temperatures from room temperature down to -196°C. The thermal expansion coefficients of both alloys decrease as temperature is lowered.

University of Minnesota. TR 52-320.

EFFECT OF DAMPING CONSTANTS AND STRESS DISTRIBUTION ON THE RESONANCE RESPONSE OF MEMBERS. B. J. Lazan. AF 33(038)-18903. December 1952. PB 116818. Order from LC Mi $2.00, Ph $2.75.

The amplitude of vibration of a member at resonance, as defined by its resonance amplification factor, is analyzed in relationship to the damping properties of materials. Data are presented on damping energy to indicate the effect of stress magnitude, stress history and temperature. Based on the mathematical relationship found to exist between damping and stress magnitude the resonance amplification factors are determined for a variety of direct stress members and beams. It is shown that the amplification in vibration caused by resonance may be considered to be the product of three basic factors: (a) the mathematical factor, (b) the cross-sectional shape factor, and (c) the longitudinal stress-distribution factor. The first of these factors may be calculated from the damping and dynamic modulus properties of the material and the last two from the shape and loading characteristics of the member. Diagrams are presented to show these basic factors as functions of the damping exponent and other variables for members commonly encountered in engineering practice. Experimental data are presented to confirm the equations derived for resonance amplification factors of members having various shapes and stress distribution.

University of Minnesota and Syracuse University. TR 52-252.

DYNAMIC TESTING OF MATERIALS AND STRUCTURES WITH A NEW RESONANCE-VIBRATION EXCITER AND CONTROLLER. B. Lazan, A. Gannett, and P. Kirmser. AF 33(038)-18903. December 1952. PB 116825. Order from LC Mi $2.75, Ph $6.50.

The nature of near-resonance vibration and response characteristics are discussed to clarify the relationships among resonance amplification factor, damping energy and dynamic modulus of elasticity. A newly developed machine is described for exciting and controlling resonance or near resonance vibrations in materials and joints under various types of stress. This machine imposes in adjustable-while-running mechanical exciting force at a controllable frequency and by means of automatic electronic controls maintains (a) the desired vibration phase angle (usually 90° for resonance) by controlling the frequency of the exciting force, and (b) the desired magnitude of the excited force by automatically controlling the magnitude of the exciting force. Equations are developed for determining the resonance amplification and other properties from the machine readings. The stability and accuracy of the machine are discussed. Data are presented on the damping and elasticity properties of aluminum and mild steel, and these are compared with results produced in rotating cantilever beam equipment. The resonance response, damping, and elasticity properties of a bolted joint were determined and the effects of bolt tension and molybdenum disulfide lubrication are illustrated and partially analyzed.

University of Illinois. TR 53-10.

THE INFLUENCE OF TEMPERATURE AND RATE OF STRAIN ON THE PROPERTIES OF METALS IN TORSION. C. E. Work and T. J. Dolan. AF 33(038)-21587. February 1953. PB 113980. Order from LC Mi $8.50, Ph $30.25.

An experimental study was made to determine the effect of temperature and rate of strain on the strength, ductility and energy absorbing capacity of seven different structural metals in torsion. Cylindrical specimens 0.25 in. in diameter were tested at four different constant

strain-rates from 0.0001 in./in./sec. to 12.5 in./in./sec. and at four different temperatures from room temperature up to 1200°F. Two series of tests were conducted: (A) specimens were held at the test temperature for one-half hour before loading, and (B) specimens were given a two-hundred hour aging treatment at the test temperature before testing.

Torque, angle of twist, and time were continuously recorded and the torsional properties determined. The detailed results are presented in three-dimensional charts analyzed in terms of the mechanisms altering the material behavior. In general, it was found that an increase in strain-rate caused an increase in strength, whereas an increase in temperature reduced the strength of all metals except in the blue-brittle temperature range for steel. Extremely great ductility was exhibited by some of the metals at the highest elevated temperatures employed, particularly at the slower rates of straining. The two-hundred hour aging treatment had no appreciable effect on the properties of most of the metals tested; significant changes were produced only in the aluminum alloys at 400°F and 600°F and alloy steel at 1200°F.

The experimental observations were compared with several theories that have been proposed to express mathematically the effects of strain-rate and temperature on mechanical properties. By proper selection of empirical constants, several equations involving a general relation for flow stress or new parameters of a "temperature-modified" strain-rate or a "velocity-modified" temperature were found to express approximately the variations obtained in mechanical properties.

TR 52-89, Part 3.
PLASTIC BEHAVIOR OF ENGINEERING MATERIALS. PART 3 - CRITICAL REVIEW AND INTERPRETATION OF THE LITERATURE ON PLASTIC (INELASTIC) BEHAVIOR OF ENGINEERING METALLIC MATERIALS. June 1953. PB 122464. Order from LC Mi $11.10, Ph $39.35.

National Bureau of Standards U. S. Department of Commerce. TR 53-7.
POISSON'S RATIO OF AIRCRAFT SHEET MATERIALS FOR LARGE STRAINS. Stanley Goodman and Stanton B. Russell. P. O. (33-038)-51-4061. June 1953. PB 116820. Order from LC Mi $3.25, Ph $9.00.

Tests were conducted to determine the value of Poisson's ratio in the plane of the sheet for six aircraft sheet materials under tensile load through an axial stress range from zero to a stress producing either severe necking or fracture. Specimens were loaded at various orientations relative to the direction of rolling of the sheet.

Maximum values of Poisson's ratio obtained were: 0.434 for XA78S-T6 alclad aluminum alloy; 0.473 for 75S-T6 alclad aluminum alloy; 0.445 for 24S-T3 alclad aluminum alloy; 0.622 for FS-1h magnesium alloy; 0.769 for commercially pure titanium; and 0.544 for RC-130-A titanium alloy.

Density determinations were made of unstrained and highly strained portions of sheet. Poisson's ratio in the thickness direction was measured over a range of large axial strains for two of the materials. It was markedly different from Poisson's ratio in the transverse direction. Values of Poisson's ratio in the transverse direction computed from the measured values in the thickness direction were in good agreement with measured values in the transverse direction.

The results obtained in this investigation are compared with the results of earlier investigations conducted by the National Bureau of Standards.

Rem-Cru Titanium, Inc. TR 53-71.
EFFECT OF STRAIN RATE ON THE MECHANICAL PROPERTIES OF TITANIUM-BASE MATERIALS. D. R. Luster, W. W. Wentz and J. P. Catlin. AF 33(038)-21912. September 1953. PB 138686. Order from LC Mi $7.80, Ph $25.80.

The results of this investigation show that at intermediate rates of strain, high purity titanium is not significantly more sensitive to strain rate changes than are some other metals. Nitrogen additions, however, increase the rate sensitivity markedly so that the presence of nitrogen in all commercial titanium-base materials is the probable reason for their high rate sensitivity at intermediate and low strain rates.

All of the representative types of titanium alloys were investigated. The substitutional all-alpha alloy showed the most desirable strength -vs- strain rate characteristics. This alloy type exhibited the lowest rate sensitivity at the slow testing speeds thus promising good long-term strength, and also exhibited a relatively rapid strengthening with high rate of strain, thus promising excellent impact resistance. The high impact resistance of the all-alpha alloy was confirmed by Charpy V-notch impact tests.

Analysis of the test data and examination of microstructures suggests that all titanium-base material undergo a basically similar change in mode of deformation with changing strain rate and temperature. It is proposed that this change is from slip and/or twinning at high strain rates and/or low temperatures to grain boundary microflow at low strain rates and/or high temperatures. Some evidence is given although further confirmation is required.

The effect of strengthening the base material by cold working or alloying appears to be to alter the range of conditions over which this changing mode of deformation takes place. Cold working and alloying also superimpose strain aging, recovery, and transformation effects which further alter the individual characteristics of the various representative titanium-base materials investigated.

Northrop Aircraft, Inc. TR 53-300.
MATERIAL PROPERTY DESIGN CRITERIA FOR LARGE MAGNESIUM ALLOY CASTINGS. Keith F. Finlay. AF 33(600)-8496. November 1953. ASTIA Document No. AD 38626. PB 133507. Order from LC Mi $7.20, Ph $22.80.

A study was made of the correlation between casting size, metallurgical quality as determined by radiographic classification, and mechanical properties of large magnesium alloy castings.

Twelve 40-inch panels and one 16-foot wing section were cast. Standard separately cast test bars were produced as controls for each casting. The 40-inch and 16-foot castings were radiographed in their entirety to determine the sections which represented a range of metallurgical imperfections. Representative imperfect sections were removed and machined into test coupons. The coupons were in turn radiographed and classified. Tensile tests, compression tests, shear tests, and strain measurements were carried out on the coupons, and statistical analysis was conducted on the resulting data.

Data, graphs showing the results of statistical analysis, and conclusions and recommendations are presented in this report. In addition, photomicrographs representing typical radiographic classifications are included. A supplement contains glossy prints of the radiographs for all test specimens.

The Ohio State University Research Foundation. TR 5662, Part 5.
INVESTIGATION OF MECHANICAL PROPERTIES AND PHYSICAL METALLURGY OF AIRCRAFT ALLOYS AT

VERY LOW TEMPERATURES. PART 5 - MECHANICAL PROPERTIES OF METALS AND A PLASTIC LAMINATE AT LOW TEMPERATURES. M. G. Fontana, S. M. Bishop and J. W. Spretnak. AF 33(038)-15698. December 1953.

The materials and tests covered in this supplementary report are as follows:

(1) Tensile and notched fatigue tests at -253°C on 24S-T4, 75S-T6, NE(SAE) 8630 steel, titanium, and 18-8 stainless steel type 304.
(2) Tensile compression, and fatigue tests at room temperature and -196°C on a glass-fabric-polyester resin laminate.
(3) Hardness, tensile and impact tests of titanium at room temperature and -78°C.
(4) Hardness and impact tests of a Cr-Ni-Cu steel at room temperature, - 78°C, -127°C, -196°C, and -253°C.
(5) Dilatometric measurements over the range room temperature to -253°C on 24S-T4, 75S-T6, NE(SAE) 8630 steel, titanium, and 18-8 stainless steel.
(6) Thermal cycling tests of the glass fabric polyester resin laminate consisting of cooling to -196°C and warming to room temperature.

The results may be summarized as follows:

(1) In tension, all the materials are notch sensitive at -253°C; the ductility of the NE 8630 steel is greatly reduced; the other materials retain some ductility at -253°C. At -253°C, all the materials tested show an improvement in fatigue resistance at low stresses. The resistance to overstressing is reduced.
(2) Both the compressive and tensile strengths of the polyester resin laminate are improved at -196°C. Some increase in fatigue strength results when this material is cooled to -196°C.
(3) Titanium increases in hardness and tensile strength at -78°C, but its impact resistance is lowered.
(4) The hardness of the Cr-Ni-Cu steel is increased as temperature is lowered. This material undergoes a gradual transition from ductile to brittle impact fracture as the temperature is lowered to -253°C.
(5) The aluminum alloys have the highest coefficients of expansion at low temperatures. Titanium shows a low expansion coefficient.
(6) The glass fabric polyester resin laminate is not damaged by repeated thermal cycling between room temperature and -196°C.

University of Minnesota. TR 53-510, Part 1.
DYNAMIC CREEP, STRESS-RUPTURE, AND FATIGUE PROPERTIES OF 24S-T4 ALUMINUM AT ELEVATED TEMPERATURES. PART 1 - UNNOTCHED SPECIMENS. Fred W. Demoney and Benjamin J. Lazan. AF 33(038)-20840. March 1954. PB 122865. Order from LC Mi $3.60, Ph $9.30.

Fatigue, stress-rupture, and creep data obtained under various combinations of mean and alternating axial stress are presented and discussed for rolled aluminum alloy 24S-T4 at 300° and 500°F. The data are presented as S-N curves and stress range diagrams to show the effect of temperature, alternating-to-mean load ratio, and stress magnitude on the fatigue, stress-rupture, and creep properties.

The effect of temperature and alternating-to-mean ratio on the characteristics of the creep curve is discussed on the basis of "static" and "dynamic" types.

The role of both creep and fatigue as factors in rupture is discussed with particular reference to temperature and alternating-to-mean ratio.

Cornell Aeronautical Laboratory, Inc. WADC TR 53-24 (Part 1).
INTERMITTENT STRESSING AND HEATING TESTS OF AIRCRAFT STRUCTURAL METALS. G. J. Guarnieri. AF 33(038)-10958. May 1954. PB 133693. Order from LC Mi $4.50, Ph $12.30.

Battelle Memorial Institute. TR 52-251, Part 2.
INVESTIGATION OF COMPRESSIVE-CREEP PROPERTIES OF ALUMINUM COLUMNS AT ELEVATED TEMPERATURES. PART 2 - STABILITY PROBLEMS. R. L. Carlson and G. K. Manning. AF 33(038)-9542. May 1954. ASTIA Document No. AD 43525. PB 120290. Order from LC Mi $4.80, Ph $13.80.

A method for estimating allowable load capacities of columns subject to creep is presented. The method, which utilizes approximate stress distributions derived from isochronous-stress-strain curves to estimate column load capacities, is shown to be conservative for the time for which the estimate is made.

An application of the method is made to test data on as-received and on stabilized 24S-T4 aluminum alloy. A comparision of the computed column capacities with experimental capacities indicates that the method is satisfactory for estimating the decrease in capacity with increasing time.

Easily obtained, time-dependent tangent-modulus loads are discussed. They are interpreted as being approximations to allowable load-capacity estimates. A limited application is made to test data, and the results appear promising. It is concluded that if certain limitations are recognized, the method may prove to be useful because of its simplicity.

A presentation of the results of an experimental investigation of the effects of column imperfection and column-material variation is made. It is found that column-capacity variations of the order of 10 percent can result from column-imperfection differences and column-material variation.

The results of an experimental study of the variation of column capacity with temperature of exposure are presented.

They indicate that column efficiency, as measured by decrease in capacity, can be acceptable for very short times at higher temperatures. The efficiency at these higher temperatures falls rapidly, however, with increasing time.

Armour Research Foundation Illinois Institute of Technology. TR 6517, Part 3.
DETERMINATION OF PHYSICAL PROPERTIES OF FERROUS AND NONFERROUS STRUCTURAL SHEET MATERIALS AT ELEVATED TEMPERATURES. Donald E. Miller. AF 33(038)-8681. June 1954.

In order to establish important design criteria, tensile, compressive, bearing, and shear properties have been determined for the following materials and conditions: (1) 14S-T6 aluminum alloy sheet (clad) at room temperature and at elevated temperatures ranging from 200° to 600°F, for exposure periods between 0.5 and 1000 hours; (2) 24S-T81 and 24S-T86 aluminum alloy sheet (clad) at room temperature and at 200°, 300°, and 400°F for exposure periods between 0.5 and 1000 hours; (3) FS1-H24 magnesium alloy sheet at 200°F, for exposure periods of 0.5 and 1000 hours; (4) 75S-T6 aluminum alloy sheet (clad) at 200°F, for exposure periods between 0.5 and 1000 hours; (5) cold rolled titanium and annealed titanium at 200°F, for exposure periods of 0.5 and 1000 hours; and (6) RC-130-A titanium alloy at room temperature and at temperatures ranging from 300° to 800°F, for exposure periods of 0.5, 100, and 1000 hours. A comparison was

made between the tensile data and the data on other properties in an attempt to formulate a method for estimating all other elevated temperature properties from a knowledge of tensile elevated temperature properties and room temperature values of other properties. The conclusion was reached that the various properties are not related in a simple, consistent manner.

Test specimens, equipment, and procedures are described in detail. Test results are presented in the form of tables and curves to illustrate the effect of temperature and exposure time on the mechanical properties of the various materials under investigation.

University of Michigan. WADC TR 54-175 (Part 1).
NOTCH SENSITIVITY OF HEAT-RESISTANT ALLOYS AT ELEVATED TEMPERATURES. Howard R. Voorhees, James W. Freeman. AF 18(600)-62. August 1954. ASTIA Document No. AD 45580. PB 140005. Order from LC Mi $6.00, Ph $18.30.

Tests have been performed seeking to understand the factors affecting notch sensitivity of heat-resistant alloys under sustained loads at elevated temperatures. The investigation was based on the belief that varied response to notches must be related to relaxation characteristics of alloys at service temperature. A material was postulated to be strengthened or weakened by a notch according to the portion of total rupture life consumed while initial stress concentrations around a notch are reduced and redistributed by a creep-relaxation process. A procedure was proposed whereby the history of representative fibers in a notched specimen would be followed to the point of rupture.

Data from other sources comparing strengths for smooth and notched bars of materials of interest are included. Additional data required for the proposed analysis were obtained under the present program for three alloys with conventional heat treatments:

S-816 at 1350°F
Waspaloy at 1500°F
Inconel X-550 at 1350°F

Test results included stress - rupture time properties, short-time tensile properties, and creep properties when stresses were changed from one level to another during a test. Relaxation characteristics were measured for initial stresses both below and above the proportional limit.

The notch strengthening observed for S-816 and Waspaloy, and the notch weakening for Inconel X-550 at the test temperatures has been satisfactorily explained in terms of comparative relaxation and stress-rupture time characteristics, though further work is indicated before a quantitative correlation is attempted.

Tests were conducted to determine the effect of some metallurgical variables on the notched bar rupture test characteristics. Cold working had the greatest effect on notch sensitivity of the several conditions investigated, but no severe case of notch weakening was observed for either S-816 at 1350° and 1500°F, or for Waspaloy at 1500°F in the limited number of tests.

University of California. WADC TR 54-402.
CREEP BUCKLING OF COLUMNS. D. Rosenthal, D. Hasanovitsh. AF 33(616)-379. September 1954. ASTIA Document No. AD 51800. PB 130405. Order from LC Mi $4.80, Ph $14.80.

An upper and lower bound to the exact solution of creep buckling has been evolved. In both cases time-deflection curves are computed using isochronous curves which are cross plotted from creep tension data. In addition to the usual assumptions of the beam theory the solution is based on the assumption that the variation of stress in the cross section can be approximated by a parabola in terms of the position in the plane of bending. Under these assumptions the method of solution is quite general and applicable to prismatic columns of any type and initial eccentricity. Comparison with experimental data on Stabilized 24S Aluminum Alloy suggests that the upper bound is a closer approximation to the solution than the lower one. There is also an indication that for moderately crooked columns subjected to high average stress the tangent modulus to the isochronous curve at the average stress level would provide a closer lower bound than the one given by the theory. The deflection at a given time could then be determined directly from the isochronous curve at this time. However, the justification for this procedure could not be established theoretically and it must come from the experiment.

An attempt to extend the theory to columns with high slenderness ratio shows that the solution depends critically on the initial eccentricity, a result which is in qualitative agreement with the experiment.

The investigation shows a need for further work, both experimental and theoretical. In particular, there is a need for duplicate tests to determine the scatter of experimental data.

Cornell Aeronautical Laboratory, Inc. WADC TR 53-24, Part 2.
INTERMITTENT STRESSING AND HEATING TESTS OF AIRCRAFT STRUCTURAL METALS. G. J. Guarnieri. AF 33(038)-10958. September 1954. PB 133694. Order from LC, Mi $5.40, Ph $15.30.

The high-temperature creep-rupture properties of six sheet alloys having application to aircraft design were investigated under conditions of intermittent load and temperature for comparison with their corresponding constant temperature-constant load behavior. Included are type 321 stainless steel at 1200° and 1350°F, N-155 alloy at 1350° and 1500°F, Inconel X at 1350° and 1500°F, RC-130-A titanium at 800°F, 24S-T3 clad aluminum at 300°, 450°, and 600°F, and FS-1H magnesium at 300° and 450°F.

Intermittent load-constant temperature tests were conducted with a cycle of one-hour-load-on, one-hour-load-off in most cases although several of the alloys were subjected also to an eight-hour-on, eight-hour-off load cycle. Intermittent temperature-constant load tests were made using a two-hour cycle with the specimen at temperature one hour and cooled one hour. While these relatively simple cyclic conditions do not duplicate the complex load and temperature patterns encountered in aircraft service, the results obtained do provide some qualitative guidance in applying available static creep-rupture data to design of aircraft structural parts.

A wide variety of effects were produced by the intermittent load and temperature conditions. Stability of microstructure was one of the more significant variables controlling the alloy response to the cyclic conditions. Acceleration of creep and rupture was induced by intermittent loading where such processes as overaging, relaxation, recrystallisation, and loss of ductility occurred. Retarding of creep and rupture occurred in those alloys where increase in ductility and creep recovery developed because of the intermittent-load cycle. Intermittent heating produced acceleration of creep and rupture in a number of cases, particularly where susceptibility to intergranular oxidation and cracking was aggravated by thermal stresses.

Rem-Cru Titanium, Inc. WADC TR 53-71, Part 2.
EFFECT OF STRAIN RATE ON THE MECHANICAL PROPERTIES OF TITANIUM BASE MATERIALS. J. P. Catlin, W. W. Wentz. AF 33(038)-21912. February 1955. PB 134784. Order from LC, Mi $6.90, Ph $21.30.

Constant strain rate tensile tests at a variety of strain rates and temperatures were conducted on four commercial-purity alloys representing the three basic types of titanium-base materials (alpha, beta, and combined alpha-beta). The room temperature rate sensitivity ratings, in order of increasing rate sensitivity are:

1. Ti-4%Mn-4%Al (combined alpha-beta).
2. Ti-5%Al-2.5%Sn (alpha).
3. Ti-10%Mn-5%Cr-5%Mo (beta).

Tensile results on high purity unalloyed materials containing individual additions of C, O, N, and H indicate that the C, O, and N additions (0.3, 0.2 and 0.1% respectively) cause an increase in rate sensitivity at room temperature. The hydrogen addition (0.01%) had little effect on rate sensitivity.

The above interstitial additions, nitrogen excepted, also minimized the extent of twinning during deformation. Some evidence of increased amounts of twinning with decreasing strain rates was also obtained.

WADC TR 54-122.
EFFECT OF STRAIN RATE ON THE STRENGTH PROPERTIES OF SINGLE AND MULTIPLE RIVETED LAP AND BUTT JOINTS. Richard F. Klinger. February 1955. ASTIA Document No. AD 67338. PB 130351.

Slow and rapid loading tensile shear tests were conducted at room temperature on single and multiple rivet lap joints and also on butt joints with multiple riveted cover plates. The rivets were made of 24S-T31 and A17S-T3 aluminum alloys in 1/8 inch and 3/16 inch diameters. The sheet material used was 0.064 inch and 0.072 inch clad 75S-T6 aluminum alloy. The joints were fabricated by standard machine riveting and shop practice. The ultimate strengths of the joints were determined with times to the ultimate load of 0.03 seconds and one minute. All failures were by rivet shear. No pronounced effect of rapid loading on the ultimate strength of any of the riveted joints tested was found although a slight trend toward decreasing strength was observed.

WADC TR 53-507.
EFFECT OF STRAIN RATE ON THE TENSILE PROPERTIES OF SAE 4340 STEEL. Richard F. Klinger. February 1955. PB 111948. Order from OTS $1.00.

The effect of tensile loading rate was determined at room temperature on SAE 4340 steel ranging from the annealed condition to a nominal ultimate strength of 220,000 psi. Stress-strain curves were obtained and the mechanical properties determined over a strain rate range of from 0.00002 in/in/sec to 20 in/in/sec. The results indicate that the strength properties at the higher strain rates are greater than at the low strain rates and that this strain rate effect is less as the strength level of the steel is increased. This trend continues to the 220,000 psi level which shows no change or a slight loss in strength at the higher strain rates. The elongation was not affected by the change in strain rate at any strength level tested.

WADC TR 54-214, Part 1.
ELEVATED-TEMPERATURE TESTING PROCEDURES. PART 1. CONTINUOUS RECORDING OF TIME-DEFORMATION READINGS DURING CREEP-RUPTURE TESTING AT TEMPERATURES UP TO 1200°F. William H. Rector, Charles A. Townsley. February 1955.

A new method of recording deformation during creep-rupture testing at temperatures up to 1200°F is presented. This method has the advantages of: (1) obtaining a continuous time-deformation record up to failure, compared to the intermittent readings obtained using the former manual method and (2) being completely automatic and saving the technician's time required to make manual measurements. This method employs cameras to record the data. A description of the equipment is given.

Syracuse University. WADC TR 55-18, Part 1.
DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS-CONCENTRATIONS AND HYDROGEN EMBRITTLEMENT. PART 1. EFFECTS OF HYDROGEN EMBRITTLEMENT ON HIGH STRENGTH STEELS-STATIC PROPERTIES. E. P. Klier, B. B. Muvdi, George Sachs. AF 33(616)-2362. May 1955.

In Part I data on the hydrogen embrittlement of one heat of 4340 steel are presented and evaluated. The data represent the mechanical behavior of the embrittled steel in the presence of stress-concentrations. Two different methods of embrittling the steel were employed. The first was to cathodically embrittle the specimens in a bath of 10 percent NaOH, and the second was to introduce embrittlement through the commercial plating of the specimens.

Three different tests were performed to evaluate the effect of hydrogen embrittlement on the steel under different test conditions. The first was a tensile test and the second a bend test. In both of these tests several loading rates were used to evaluate the effect of embrittlement under different loading conditions. The third test performed was a stress-rupture test to render the evaluation of embrittlement under sustained-load applications possible.

Several of the tests examined are suitable for the evaluation of hydrogen embrittlement. However, the bend test is the simplest, and most economical of these tests. At the same time it is a sensitive method of evaluating hydrogen embrittlement in ultra-high strength steels.

Southern Research Institute. WADC TR 55-199, Part 1.
TENSILE PROPERTIES OF AIRCRAFT STRUCTURAL METALS AT VARIOUS RATES OF LOADING AFTER RAPID HEATING. Clifford L. Dotson, J. Robert Kattus. AF 33(616)-424. August 1955. PB 121137. Order from OTS $4.50.

The purpose of the work described in this report was to determine the effects of the following variables on the tensile properties of seven aircraft structural sheet metals after they had been heated within 10 seconds to temperatures up to 1200°F:

1. Strain rates from 0.00005 in./in./sec to 1.0 in./in./sec.
2. Holding times at test temperature from 10 seconds to 30 minutes.

Special testing apparatus for heating and controlling the temperature of the test specimens, loading the test specimens, and recording load-strain curves was developed.

The test results showed that the tensile properties of the structurally stable test alloys -- Type 321 stainless steel, RC-70A titanium and RC-130A titanium alloy -- were not appreciably affected by changes in holding time. Because of the structural changes which occurred, the tensile

properties of the precipitation hardening test alloys -- Alclad aluminum alloys 2014-T6, 2024-T3, and 7075-T6 -- and hard rolled AZ-31 magnesium alloy changed as the holding time was varied at certain temperatures.

With the exception of the results at one particular test condition, the strength properties of each alloy increased with increasing strain rates. The exception occurred in Alclad 2024-T3 alloy at 450°F, at which temperature a strongly mitigating structural change occurred. Percent elongation in the different test alloys varied inconsistently with increasing strain rates dependent upon the alloy, temperature, and holding time.

University of Minnesota. WADC TR 55-372.
THE EFFECT OF SURFACE PREPARATION AND CONDITION ON MICROHARDNESS. F. H. Vitovec, H. F. Binder. AF 33(616)-2803. October 1955. ASTIA Document No. AD 887663. PB 128519. Order from LC, Mi $3.00, Ph $6.30.

Observations on the load dependence of the pyramid microhardness are discussed and various explanations of it are briefly reviewed. Experimental results are presented which indicate the effect of the polishing procedure on the hardness-load relationship. A simplified mathematical analysis is presented which shows the relationship between the hardness-load behavior and the stress-strain curve under uniaxial deformation. The various hardness-load trends are explained in terms of strain hardening, effect of the free surface, and cold working introduced by the polishing procedure.

Battelle Memorial Institute. WADC TR 55-150, Part 2.
MATERIALS PROPERTY DESIGN CRITERIA FOR METALS Pt 2 A STUDY OF METHODS OF PRESENTING CREEP DATA FOR AIRFRAME DESIGN. W. S. Hyler, H. J. Grover. AF 33(616)-2303. November 1955.

A study was made of a number of methods of presenting creep data from the standpoint of usefulness to the designer of airframes. These methods included: stress-time graphs, stress-temperature graphs, Larson-Miller graphs, and isochronous stress-strain graphs. Consideration was given to current practices and needs of personnel in the aircraft structures field. Also, consideration was given to the use of creep data in particular problems associated with high-speed flight.

On the basis of this study, it appears that a particularly useful method of presenting creep data is that of the stress-temperature graphs (with time as a parameter). It is also believed in some applications that the isochronous stress-strain graph may be a particularly desirable presentation, since useful engineering approaches for certain high-temperature problems have been advanced based on these graphs. This latter presentation should be considered tentative pending verification of these engineering approaches by experimental studies.

Syracuse University. WADC TR 55-103, Suppl. 1.
DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS-CONCENTRATIONS EFFECTS OF A NUMBER OF VARIABLE ON THE MECHANICAL PROPERTIES OF AIRCRAFT HIGH-STRENGTH STEELS. B. B. Muvdi, E. P. Klier, G. Sachs. AF 33(616)-2362. January 1956. ASTIA Document No. AD 92582. PB 133208. Order from LC, Mi $9.90, Ph $34.80.

Supplement 1 of WADC TR 55-103, is a summary of all test results assembled in this investigation. The results are presented in graphs which indicate all the important parameters.

Syracuse University. WADC TR 55-103, Suppl. 2.
DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS-CONCENTRATIONS EFFECTS OF A NUMBER OF VARIABLES ON THE MECHANICAL PROPERTIES OF AIRCRAFT HIGH-STRENGTH STEELS. B. B. Muvdi, E. P. Klier, G. Sachs. AF 33(616)-2362. January 1956. PB 121155s. Order from OTS $3.00.

Supplement 2 of WADC TR 55-103 is a summary of all test results assembled in this investigation. The results are presented in tables which indicated both individual and average test values.

Syracuse University. WADC TR 55-103.
DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS-CONCENTRATIONS Pt 1 EFFECTS OF A NUMBER OF VARIABLES ON THE MECHANICAL PROPERTIES OF AIRCRAFT HIGH-STRENGTH STEELS. G. Sachs, B. B. Muvdi, E. P. Klier. AF 33(616)-2362. January 1956. PB 121155. Order from OTS $3.25.

This report presents a large amount of test data on a number of low-alloy steels, heat treated to strength values between 210,000 and 290,000 psi. The steels investigated were four heats of 4340 and one heat of each of the following: V-Mod. 4330, 98B40, Hy-Tuf, Super Hy-Tuf, Super TM-2 and Inco. Furthermore, the results of tests previously performed on an additional heat of 4340 (small dia. bar) are included for purposes of comparison and completeness. The tests performed were the following: (a) Tensions, which yielded information concerning the tensile and yield strengths as well as the ductility (reduction of area and elongation) of the steels studied. (b) Notch-Tension, from which the notch strength, notch-strength ratio and information on the notch sensitivity of the steels were obtained. (c) Impact, which permitted evaluation of impact characteristics of the steels at various test temperatures, as well as some information regarding the transition from impact-ductile to impact-brittle behavior. (d) Fatigue and Notch-Fatigue, from which the endurance limit and the fatigue strength at various numbers of cycles were obtained for both smooth and notched specimens. (e) Stress-Rupture, which permitted investigating the behavior of high-strength steels under sustained load conditions.

In addition, hardness measurements as well as metallographic studies were performed on all steels.

This report comprises first an extensive discussion of the effects of all fundamental factors investigated here. These factors are: (a) specimen position, (b) various heats of 4340 steel, (c) as-processed section size, (d) as-tested section size, (e) tempering temperature, (f) 500°F temper brittleness, (g) stress concentration, (h) directionality, (i) eccentricity, (j) loading time and (k) test temperature. This is followed by a comparison of the properties of the various steels, those of 4340 steel being used as basis.

The appendixes (Supplement 1 and 2) assembles the individual data in graphical and tubular form. Various parametric representations are used to facilitate their utilization.

Cornell Aeronautical Laboratory, Inc. WADC TR 53-24, Part 3.
INTERMITTENT STRESSING AND HEATING TEST OF AIRCRAFT STRUCTURAL METALS. G. J. Guarnieri. AF 33(616)-2226. January 1956. ASTIA Document No. AD 90895. PB 135127. Order from LC, Mi $4.50, Ph $12.30.

Additional data have been gathered as part of a continuing program to evaluate the effect of intermittent load and temperature upon the high-temperature creep and

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rupture properties of aircraft structural alloys. The results obtained for the four alloys of 24S-T3 aluminum, RC-70 and RC-130-A titanium, and 4130 steel, when considered with respect to data previously reported for FS-1H magnesium, 321 stainless steel, Inconel X, and N-155, further emphasize that each alloy system behaves in its own characteristic manner when subjected to intermittent temperature and load. Data of the type being accumulated are of value to design engineers in the sense that some guidance is provided in the application of available static creep and rupture data to the construction of parts for intermittent service conditions.

Emphasis in the test program is now turning towards consideration of effects of combined cycling of load and temperature inasmuch as the creep and rupture behavior has been established for eight alloys under the simpler conditions of constant temperature-constant load, constant temperature-intermittent load, and constant load intermittent temperature.

Battelle Memorial Institute. WADC TR 55-150, Part 1.
MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS. AF 33(616)-2303. January 1956.

The research program described in this report was undertaken to review the field of materials-property design criteria for metals used in aircraft and missiles, and to bring up to date a compilation of information of maximum or typical value for the design of aircraft and missiles. The information obtained in this study has been presented in such form as to be directly applicable to Bulletin ANC-5 (issued by the Air Force-Navy-Civil Panel on Strength of Metal Aircraft Elements).

A great deal of work along this line has been done by the Air Force and other agencies. Much of this information has been published in WADC technical reports.

In the past few years, new metals and alloys have been developed, new properties were recognized as being important in design, and new information was obtained on the design uses of material properties. While much of this information is available in such sources as WADC technical reports, some is less readily available for the designer, and some gaps and inconsistencies in the existing information are apparent.

As a result of mutual agreement between representatives of the Materials Laboratory of Wright Air Development Center and Battelle Memorial Institute, investigation of seven specific subjects was undertaken. These are:

1. The development of recommended design values at elevated temperatures for SAE 4130, SAE 8630, 17-7(301), and 18-8 (302) steels.
2. A study of available data on bending moduli of steel and aluminum round tubes.
3. A study of available data on torsional modulus of rupture of steel and of aluminum round tubing.
4. A study of fatigue data on magnesium alloys in ANC-5.
5. A survey of available data on several stainless steels and superalloys.
6. A study of available data on spot-welded joints at depressed and elevated temperature.
7. Aluminum- and magnesium-base alloys for sand-, permanent-mold-, and die-casting applications.

Since the various phases were initiated and carried out separately, each will be reported in a separate section of this Final Report.

University of Michigan. WADC TR 54-175, Part 2.
NOTCH SENSITIVITY OF HEAT-RESISTANT ALLOYS AT ELEVATED TEMPERATURE PT 2. "Analysis of Notched-Bar Rupture Life in Terms of Smooth-Bar Properties." Howard R. Voorhees, James W. Freeman. AF 18(600)-62. January 1956. PB 121184. Order from OTS $2.25.

Rupture lives of smooth and notched bars have been compared in tests on three heat-resistant alloys with conventional heat treatments. Additional rupture tests were run for two of the alloys using notched and smooth specimens from material cold rolled between solution and aging treatments. The method of notch preparation was demonstrated to have a large effect on notched-bar life for some conditions. Variable results were attributed to residual stresses remaining from the machining operation.

Other experimental data obtained on smooth bars for some alloys and conditions included short-time tensile properties, creep curves, relaxation characteristics, and rupture life when the stress was changed from one level to another during the test.

A definite qualitative agreement was noted between notch strengthening or weakening and the rate of experimental stress relaxation under creep conditions. A quantitative stepwise analysis was developed to compare notch rupture behavior with properties of smooth bars. This analysis was based on the postulate that for a material to be notch strengthened it must be able to relax high initial stress concentrations quickly before a major portion of the total life has been expended in fibers initially at stresses above the nominal stress applied to the specimen. Further, the effective stress in such fibers must eventually drop below the nominal stress for notch strengthening to occur.

Completed calculations for one condition of notch strengthening and one of notch weakening showed satisfactory agreement with experimental findings and indicates that stress redistribution by creep-relaxation could by itself explain the wide differences in notch behavior observed for different materials and test conditions.

Further work is indicated to ascertain influence of other factors and to establish the range of validity of the proposed analytical method. Suggested extensions include tests on flat notched bars and use of other types of alloys.

Syracuse University. WADC TR 55-18, Suppl. 1.
DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS CONCENTRATIONS AND HYDROGEN EMBRITTLEMENT. B. B. Muvdi, G. Sachs, E. P. Klier. AF 33(616)-2362. February 1956.

In Part II of this report data on the low-cycle rotating beam fatigue tests performed on hydrogen embrittled specimens of one heat of 4340 steel are presented and evaluated. The specimens were cathodically embrittled in a 10 percent sodium hydroxide solution, and tested on an R. R. Moore fatigue machine operated at approximately 250 rpm. All tests were limited to cycles ranging between 10 and 10,000.

The present report comprises a continuation of the work presented in Part I and as such it concerns itself with the same general purposes, namely, (1) the evaluation of hydrogen embrittlement of a steel heat treated to high strength levels and (2) the determination of suitable and sensitive means of evaluating such brittleness. While the low-cycle fatigue test reported here yielded valuable information, it is in no way as economical and sensitive as the static bend test reported in Part I.

Battelle Memorial Institute. WADC TR 56-26, Part 1.
AN INVESTIGATION OF THE INTERCHANGE OF TENSILE CREEP FOR COMPRESSIVE CREEP Pt. 1. Types

2024-T4 And 1100-0 Aluminum. O. K. Salmassy, R. L. Carlson, R. J. MacDonald, G. K. Manning. AF 33(616)-2738. March 1956. PB 121327. Order from OTS $1.75.

Aluminum 1100-0 and aluminum alloy 2024-T4 were subjected to tension- and compression-creep testing at varying stress levels and temperatures. Creep data were compared to establish whether significant differences existed between tension and compression behavior. Room-temperature and elevated-temperature static properties of each material were obtained. Metallographic and hardness studies were used to supplement the results of creep and static tests. Test equipment and test techniques were developed which permitted creep measurements approaching 10 microinches per inch in sensitivity and ± 25 microinches accuracy. Data obtained on 2024-T4 and 1100-0 aluminum in all instances indicated greater creep resistance in compression. Differences in tension- and compression-creep strain decreased with increasing temperatures. Results indicated that a reversal in the interchange of 2024-T4 may take place between 375°F and that interchange may be a temperature-dependent phenomenon.

Battelle Memorial Institute. WADC TR 52-251, Part 4.

INVESTIGATION OF COMPRESSIVE-CREEP PROPERTIES OF ALUMINUM COLUMNS AT ELEVATED TEMPERATURES Part 4. Additional Studies. R. L. Carlson, E. G. Bondine, G. K. Manning. AF 33(038)-9542. April 1956. PB 121465. Order from OTS $2.00.

A summary and an analysis of the results of an experimental study of the creep buckling of columns is presented. The results include investigations of the behavior of short, inelastic columns of the aluminum alloy 2024-T4, of square tubing columns of aluminum alloy 2024-T3, and columns of the stainless steel 17-7PH (THD 1050).

Data from all phases of the investigation indicate that column imperfection variations can cause marked scatter in lifetime. It is concluded that the effect of imperfection variations will exert a strong influence on the evolution of practical methods of designing for creep buckling.

Shanley's time-dependent tangent modulus is applied to the test data, and it is found to be fairly successful in providing an estimate of the possible loss in load capacity with time. It is observed, however, that the agreement with test data can be expected to vary significantly with column imperfection.

The possible existence of a lower load limit below which collapse will never occur is discussed. From a consideration of know creep behavior, it is concluded that there may be a temperature below which finite lower limits do exist and above which the lower limit is zero.

University of California. WADC TR 53-336, Part 4.

THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS, Part 4. Creep Results for Alclad 7075-T6 Aluminum Alloy and Comparison with Results for Other Materials. N. H. G. Daniels, H. B. Masuda, John E. Dorn. AF 33(038)-11502. May 1956. PB 121435. Order from OTS $2.75.

A study was made of the creep properties of clad aluminum alloy 75S-T6 under intermittent heating and loading conditions at 300°F and 600°F. A few tests were also conducted at 450°F under intermittent heating conditions.

The results were correlated by means of the empirical functional equation $\varepsilon = F\{tf(\sigma)\}$

where ε = total time
t = time
σ = stress
F = strain function
f = stress function

As suggested in previous reports, the function f was shown to be approximately independent of strain and of the type of intermittent testing cycle. The prediction of intermittent condition creep data can therefore be carried out from a knowledge of the constant load isothermal data, and a single test under the intermittent condition.

A review of the literature has shown that although in a significant proportion of cases the above analysis can be used successfully, the function f often varies considerably with strain, and in some instances is not independent of the type of intermittent condition. Nevertheless even the approximate prediction of intermittent creep data by flexible use of the method is likely to be of value.

The test results were also compared on a basis of net time at temperature under load. It appears that any intermittent cycle permitting recovery and overaging of the material in the absence of load has a deleterious effect on its creep resistance. Under other intermittent conditions the creep of the material is approximately the same on a net time basis as its creep under steady load isothermal conditions.

University of California. WADC TR 53-336, Part 5.

THE CREEP PROPERTIES OF METALS UNDER INTERMITTENT STRESSING AND HEATING CONDITIONS, Part 5. Further Creep Results on Alclad 7075-T6 Aluminum Alloy and Consideration of Analytical Procedures. N. H. G. Daniels, H. B. Masuda. AF 33(038)-11502. May 1956. PB 121476. Order from OTS $2.50.

A further study of the creep properties of aluminum alloy Alclad 7075-T6 (Clad 75S-T6) under isothermal intermittent stressing and under cyclic temperature steady load conditions has been made at 300°F, 450°F, and 600°F.

For the intermittent stressing conditions, cycles in which the load was on for 5 minutes out of a 2 hour cycle, 5 minutes out of a 12 minute cycle, and 24 hours out of a 48 hour cycle were used. The first cycle was shown to have seriously deleterious effects, particularly at 300°F, where the net times under load to reach specified strains or fracture were reduced to approximately one eighth of the corresponding times for steady load isothermal creep.

At 300°F it was shown that the ratio of on-load time to off-load time controlled the scale of the effect of the intermittent condition, which was apparently independent of the total duration of each cycle. Empirical methods of analysis and prediction based on the equation used in previous work $\varepsilon = F\{tf(\sigma)\}$

where ε = total strain
t = time
σ = stress
F = strain function
f = stress function

were successfully applied to the results both at 300°F and 450°F.

Neither the above correlation method nor the use of comparisons based on net time under load were successful at 600°F for the extreme types of cycle used. Correlations for other alloys are also given.

The cyclic temperature, steady load creep data were also subjected to attempted analysis using other correlation methods available in the literature, but these did not seem superior to the above empirical method. Other intermittent condition creep data in the literature which

have become available since the last report were also analyzed, and the limitations of the method, as discussed therein, were confirmed.

Battelle Memorial Institute. WADC TR 55-150, Part 3.
MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS, Part 3. Fatigue Evaluation of Magnesium Alloys. W. S. Hyler, F. H. Lyon. AF 33(616)-2303. August 1956. ASTIA Document No. AD 97212. PB 121 579.

WADC Technical Report 55-150, "Materials-Property-Design Criteria for Metals," indicated that the ANC-5 document shows discrepancies in fatigue data for various magnesium alloys as obtained in rotating-bending, plate-bending, and axial-loading fatigue tests. A study was made of three magnesium alloys FS-1a (AZ 31A-0), J-1 (AZ 61A-F), and O-1 (AZ 80A-F) under conditions of completely reversed stress for the three kinds of loading.

Results suggest that the discrepancies noted in ANC-5 data between rotating-beam fatigue data and data from the other two types of tests are real. Certain other inconsistencies in the data suggest that additional fatigue studies should be made to provide more reliable information.

A review of methods of presenting fatigue data in ANC-5 was made. It is suggested that a more consistent method of presentation be followed for the various alloy systems for which fatigue data are reported.

Battelle Memorial Institute. WADC TR 55-150, Part 4.
MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS, Part 4. Elastic Moduli: Their Determination and Limits of Application. S. A. Gordon, R. Simon, W. P. Achbach. AF 33(616)-2303. October 1956. ASTIA Document No. AD 110475. PB 121857.

A study was made of the modulus of elasticity at elevated temperatures for several materials as it is derived from the conventional stress-strain curve and as it is derived from the determination of the velocity of propagation of elastic waves. The two methods of determination give modulus values which agree closely in regions of low stress and where time effects are unimportant. At higher stress levels, where the stress-strain relationships are not linear and where time effects are important, moduli determined by the two methods do not agree, the dynamic modulus being higher than the statically determined modulus.

These differences determined for the magnesium alloy AZ-31 and for the aluminum alloy 2024-T4 for various temperatures are discussed with respect to their applicability to airframe design.

Syracuse University. WADC TR 56-216.
THE PROPERTIES OF CONSTRUCTIONAL METALS AS A FUNCTION OF TEMPERATURE AND STRAIN RATE IN TORSION. E. P. Klier, N. Feola, A. Viggiano, V. Weiss. AF 33(616)-2606. November 1956. ASTIA Document No. AD 110559. PB 121912.

Seven structural metals have been tested in torsion at four strain rates and at selected testing temperatures. The torque-twist relationships have been determined at the three lowest strain rates.

The experimental results confirm the trends established by Work and Dolan (1). These trends have been shown to arise from changes in the torque-twist diagrams for the respective materials in strain-rate intervals which are essentially unchanged for the different metals and which center on a strain rate of about 0.1 in./in./sec.

Both solid and tubular specimens have been tested and for the tubular specimens the normal strain rate modification of the modulus of rupture is observed at high strain rates. Differences in the trends of the data for the solid and hollow specimens are attributed to the action of the heat sink which the core of the solid specimen constitutes.

It has been possible to explain the several types of properties vs. strain rate curves by means of alterations in the torque-twist curve arising from strain hardening, temperature increase in the specimen due to plastic working, and to strain rate modification of the yield strength.

Southern Research Institute. WADC TR 55-199, Part 2.
TENSILE PROPERTIES OF AIRCRAFT-STRUCTURAL METALS AT VARIOUS RATES OF LOADING AFTER RAPID HEATING. Joseph D. Morrison, J. Robert Kattus. AF 33(616)-424. November 1956. ASTIA Document No. AD 110540. PB 121812.

The purpose of the work described in this report was to determine the effects of variations in strain rate and holding time on the tensile properties of several aircraft-structural metals after they had been heated within 10 seconds to test temperatures up to 1200°F. The work was primarily directed toward determining these effects on the yield and ultimate strength. Other properties such as modulus of elasticity, proportional limit and total elongation were also determined but only as by-products and for the purpose of establishing trends. These properties should be used only after careful consideration of the complex conditions under which they were obtained. The investigation covered strain rates from 0.00005 in./in./sec to 1.0 in./in./sec, holding times at test temperature from 10 seconds to 30 minutes, and the following materials:

1. Sheet metals: Annealed Stellite-25, precipitation-hardened Inconel-X half-hard and full-hard Type 301 stainless steel, 17-7PH stainless steel in both the annealed and the TH 1050 conditions, AISI-4130 steel in both the normalized and the quenched and tempered conditions, hot-rolled SAE-1020 steel, and A110-AT and Ti-140A titanium alloys.
2. Cast metals: ZH-62T5 magnesium alloy and 356-T6 aluminum alloy.

With a few exceptions, the strength properties of the test metals increased appreciably with increasing strain rates, whereas percent elongation reacted erratically to changes in strain rate and modulus of elasticity was affected only slightly.

At certain temperatures and strain rates, variations in holding time significantly affected the strength and ductility of 356-T6 aluminum alloy and affected only to a minor degree the strength of Inconel-X, full-hard Type 301 stainless steel, both normalized and heat-treated AISI-4130 steel, and 17-7PH-TH1050 stainless steel. These effects are believed to be caused by structural changes. Variations in holding time had no effect on the properties of the other alloys.

Up to 1000°F, Type 17-7PH-TH1050 stainless steel was the strongest material tested, but above 1000°F precipitation-hardened Inconel-X was the strongest.

Syracuse University. WADC TR 56-395, Part I.
DESIGN PROPERTIES OF HIGH STRENGTH STEELS IN THE PRESENCE OF STRESS CONCENTRATIONS, Part I. Dependence of Tension and Notch-Tension Properties of High-Strength Steels on a Number of Factors. B. B. Muvdi,

G. Sachs, E. P. Klier. AF 33(616)-2362. December 1956. ASTIA Document No. AD 110637. PB 121847.

In this report are presented the results of tension and notch-tension tests performed on hot rolled sections from commercial, electric furnace heats of 4340, V-Mod. 4330, 98B40, Tricent (Inco), Super Hy-Tuf, Hy-Tuf and Super TM-2 steels. Tension tests were conducted on 0.28 in. dia. specimens. An exception was a single test completed on a smooth 0.9 in. dia. 4340 steel specimen in order to examine the effect of section size on the tension properties of this steel. Notch-tension tests were performed on 0.3, 0.5 and 0.9 in. dia. specimens that were heat treated to strength levels ranging between 180,000 and 300,000 psi approximately. These specimens were provided with notches leading to stress-concentration factors, K, of 3, 5 and 10. In both instances (tension and notch-tension tests) longitudinal and transverse specimens were examined. Furthermore, information from the literature pertaining to the effects of as-processed section size is considered and evaluated.

In general, the tensile strength was found to be independent of the specimen orientation, but to decrease gradually with increase in the specimen size. The ductility of smooth specimens, however, was observed to depend on both specimen orientation and specimen size.

The notch strength decreased with increase in stress concentration, specimen diameter and as-processed section size. It also decreased as the specimen orientation was changed from longitudinal to transverse. These effects were pronounced at high strength levels and diminished, with decrease in the tensile strength, to insignificant values at strength levels below 200,000 psi.

Syracuse University. WADC TR 56-395, Part II. DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS CONCENTRATIONS, Part II. Axial-Load Fatigue Properties of High-Strength Steels. B. B. Muvdi, G. Sachs, E. P. Klier. AF 33 (616)-2362. December 1956. ASTIA Document No. AD 110619. PB 121883. Order from OTS $1.25.

In this report are presented the results of axial-load (completely reversed) fatigue tests on Tricent (Inco), Cru. SHS-260 and Super TM-2 steels heat treated to strength levels between approximately 250,000 and 300,000 psi. The fundamental effects of several variables on the fatigue properties of these steels are discussed and evaluated. These variables included the notch sharpness or the stress concentration, the strength level and specimen orientation. The results indicated that the fatigue strength was lowered as the stress-concentration factor was increased, a maximum lowering effect occurring for stress concentration factors between one and three. This effect was observed to depend upon the strength level. The endurance limit for both smooth and notched specimens developed minimum values at a strength level between 240,000 and 260,000 psi, and maximum values at a strength level between 270,000 and 300,000 psi approximately. In general, the endurance limit was found to be lower for transverse than for longitudinal specimens. Furthermore, this effect was much more pronounced for smooth than for notched specimens and was observed to be severe at high strength levels and to decrease with decrease in this quantity.

Syracuse University. WADC TR 56-395, Part III. DESIGN PROPERTIES OF HIGH-STRENGTH STEELS IN THE PRESENCE OF STRESS CONCENTRATIONS AND HYDROGEN EMBRITTLEMENT, Part III. The Response of High-Strength Steels in the Range of 180,000-300,000 psi to Hydrogen Embrittlement from Cadmium Electroplating. E. P. Klier, B. B. Muvdi, G. Sachs. AF 33(616)-2362. March 1957. ASTIA Document No. AD 118167. PB 131034.

The embrittlement of high strength steels due to the action of hydrogen introduced by Cd-electroplating has been studied in sustained-load, rotating beam fatigue, and bending tests. Strength levels from 180,000 to 300,000 psi as suitable for the various steels were examined for a variety of initial conditions of stress concentration.

All steels were found to be embrittled in some measure after Cd-plating and this embrittlement could not be fully eliminated, as determined in the bend test, through the baking treatment used. The improvement in properties which did result from baking was promoted by a redistribution and not an elimination of hydrogen from the steel.

Failure promoted by Cd-plating is affected by the experimental conditions and has been discussed at length in the report. In the hydrogen bearing zone a crack is initiated and then depending on the experimental conditions may propagate to failure of the cross section through overloading. Crack development is apparently dependent, in part, on the composition and is minimized by reduction in carbon content or by an increase in silicon content.

Both the sustained-load and bend tests are suitable tests for evaluation of hydrogen embrittlement in ultra-high strength steels. The rotating beam fatigue test is a relatively insensitive test of hydrogen embrittlement, but can be used to provide an excellent measure of the "static" notch strength of the steel.

Cornell Aeronautical Laboratory, Inc. WADC TR 53-24, Part 4.
INTERMITTENT STRESSING AND HEATING TESTS OF AIRCRAFT STRUCTURAL METALS. John Salvaggi. AF 33(616)-2226. May 1957. ASTIA Document No. AD 118293. PB 131210.

Previous studies on the high-temperature creep and rupture behavior of a number of aircraft structural materials exposed to relatively simple intermittent-load or intermittent-heat test conditions emphasized the fact that each alloy system behaves in its own characteristic manner. Five of these materials, C-110M titanium, A-70 titanium, type 321 stainless steel, N-155 alloy, and 4130 steel were selected for further evaluation under the more complex conditions of combined intermittent temperature and load. Analysis of these test results disclosed little difference in creep and rupture behavior relative to the intermittent-load or intermittent-heat results.

In general, the basic mechanisms or causes responsible for the behavior patterns noted in the simpler environments of constant-temperature intermittent-load and constant-load intermittent-temperature are relatively unaffected by the phase relationships of the combined cyclic-load and temperature test conditions utilized in this study.

University of Minnesota. WADC TR 56-44. ANALYTICAL METHODS FOR DETERMINING SPECIFIC DAMPING ENERGY CONSIDERING STRESS DISTRIBUTION. E. R. Podnieks, B. J. Lazan. AF 33(616)-2803. June 1957. ASTIA Document No. AD 130777. PB 131250.

Various energy and energy ratio units for expressing the damping properties of materials are discussed. Specific damping energy (in-lb/cu in/ cycle) is proposed as the most useful unit for comparing materials and performing engineering calculations. Analytical methods are presented for converting the total or average damping energy of a specimen to its specific damping. The role of the volume-stress function in this analysis is discussed in general and specific cases are illustrated. Additional

factors involved in converting such energy ratio units as logarithmic decrement are analyzed. Data on the dimensionless damping and strain energy integrals are included for a range of conditions to facilitate data conversion. Caution is urged in interpreting damping data in which stress distribution and other factors, analyzed in this report, are not fully considered.

Southern Research Institute. WADC TR 56-340. DETERMINATION OF TENSILE, COMPRESSIVE, BEARING, AND SHEAR PROPERTIES OF FERROUS AND NON-FERROUS STRUCTURAL SHEET METALS AT ELEVATED TEMPERATURES. John V. Melonas, J. Robert Kattus. AF 33(616)-2741 and AF 33(616)-3224. September 1957. ASTIA Document No. AD 131069. PB 131461.

The tensile, compressive, bearing, and shear properties of the following sheet metals were determined at various temperatures after exposure times from 1/2 to 1000 hours at the test temperature:

1. AISI-4130 alloy steel, 150,000 psi nominal strength level
2. AISI-4130 alloy steel, 180,000 psi nominal strength level
3. AISI-4130 alloy steel, 200,000 psi nominal strength level
4. A110-AT titanium alloy
5. HK31-H24 magnesium alloy
6. Type 301 stainless steel, full-hard

The magnesium alloy was tested over a temperature range from 75°F to 600°F, whereas the other test metals were tested at temperatures to 1000°F.

The test results show that all strength and modulus properties decreased to some degree with increasing temperature over the range studied. Increasing exposure time, which had no significant effects on modulus-of-elasticity values, resulted in some decreased strength properties in all test metals at the higher test temperatures but had no appreciable effect at the lower temperatures. In general, the effects of variations in temperature were greater than the effects of variations in exposure time.

A correlation and comparison of the test results indicated some crude relationships between various properties. These relationships provide a means of calculating rough estimates of certain properties on the basis of a different property determination. The accuracy of these calculations decreases as the range of materials and conditions, to which they are applied, increases. For the entire range of materials and conditions used in this work the accuracy of the various property relationships ranged from ±11% to ±55%. Precise and reliable data on the tensile, compressive, bearing, and shear properties of aircraft-structural materials can be obtained only by testing under the desired conditions.

Southern Research Institute. WADC TR 55-199, Part III.
TENSILE PROPERTIES OF AIRCRAFT-STRUCTURAL METALS AT VARIOUS RATES OF LOADING AFTER RAPID HEATING. William P. Roe, J. Robert Kattus. AF 33(616)-424. September 1957. ASTIA Document No. AD 142003. PB 131511.

The purpose of the present phase of this program was to determine the effects of variations in strain rate and holding time at temperature on the tensile properties of several aircraft-structural metals after they had been heated within 10 sec to test temperatures approaching the melting points of the alloys involved. Major emphasis was placed on an accurate determination of these effects on the ultimate tensile strength and 0.2%-offset yield strength. The modulus of elasticity, percent elongation, and proportional limit were determined with less accuracy as by-products of the data for the purpose of establishing trends. This investigation covered strain rates from 0.00005 in./in./sec to 1.0 in./in./sec, holding times at test temperature from 10 sec to 1800 sec, and the following sheet materials over the range of test temperatures indicated:

Annealed Stellite-25, 1600°F-2250°F
Heat-treated Inconel-X, 1600°F-2250°F
Full-hard 301 stainless steel, 1600°F-2250°F
Annealed A110-AT titanium alloy 1200°F-2770°F
Alclad 2024-T3 aluminum alloy, 800°f-900°F

In general, the ultimate tensile strength and 0.2%-offset yield strength of all test materials increased appreciably with increasing strain rates at each test temperature. With only minor variations, the apparent modulus of elasticity showed a consistently increasing trend with increasing strain rates. The same was true for the percent elongation with the exception of annealed A110-AT titanium alloy, which showed a decreasing trend with increasing strain rates.

Because of atmospheric attack, the properties of the A110-AT alloy deteriorated with increasing times at the higher test temperatures. Holding times at test temperature had no significant effect on any of the properties of the other materials investigated.

At 2250°F, annealed Stellite-25 had the greatest strength, followed by heat-treated Inconel-X, annealed A110-AT titanium alloy, and full-hard 301 stainless steel.

The oxidation resistance of annealed Stellite-25 and heat-treated Inconel-X was good at 2250°F, full-hard 301 stainless was fair to poor, and annealed A110-AT titanium alloy was poor. Alclad 2024-T3 aluminum alloy showed good oxidation resistance at 900°F.

Battelle Memorial Institute. WADC TR 55-150, Part V.
MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS, Part V. The Conventional Short-Time, Elevated-Temperature Properties of Selected Stainless Steels and Super Alloys. Ronald J. Favor, William P. Achbach, Walter S. Hyler. AF 33(616)-2303. October 1957. ASTIA Document No. AD 142069.

Presented in this report is a compilation of data on the conventional short-time, elevated-temperature properties of selected corrosion-resistant and high-temperature alloys applicable to airframe and missile fabrication. The resultant recommended design data obtained in this study have been presented in such form as to be directly applicable to the ANC-5 Bulletin (issued by the Air Force-Navy-Civil Panel) on "Strength of Metal Aircraft Elements."

Battelle Memorial Institute. WADC TR 55-150, Part VI.
MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS, Part VI. The Conventional Short-Time Elevated-Temperature Properties of Selected Light Alloys. W. P. Achbach, R. J. Favor, W. S. Hyler. AF 33(616)-3965. October 1957. ASTIA Document No. AD 142043. PB 131515.

Described in this report is a compilation of data on the conventional short-time elevated-temperature properties of selected light alloys applicable to airframe and missile fabrication. The resulting recommended design data obtained in this study has been presented in such form as to be directly applicable to Bulletin ANC-5 (issued by the Air Force-Navy-Civil Panel) on "Strength of Metal Aircraft Elements."

Battelle Memorial Institute. WADC TR 55-150, Part VII.
MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS, Part VII. The Conventional Short-Time, Elevated-Temperature Properties of Selected Low- and Medium-Alloy Steels. Ronald J. Favor, William P. Achbach, Walter S. Hyler. AF 33(616)-2303. October 1957. ASTIA Document No. AD 142064.

Presented in this report is a compilation of data on the conventional short-time, elevated-temperature properties of selected low- and medium-alloy steels applicable to airframe and missile fabrication. The resulting recommended design data obtained in this study have been presented in such form as to be directly applicable to the ANC-5 Bulletin (issued by the Air Force-Navy-Civil Panel) on "Strength of Metal Aircraft Elements."

Armour Research Foundation. AF TR 6517 Pt V.
DETERMINATION OF THE TENSILE, COMPRESSIVE AND BEARING PROPERTIES OF FERROUS AND NON-FERROUS STRUCTURAL SHEET MATERIALS AT ELEVATED TEMPERATURES. Donald E. Miller. AF 33(616)-3145. December 1957. ASTIA Document No. AD 142218. PB 131595.

To provide data for the establishment of design criteria, the compressive and bearing properties of six aluminum alloys, three magnesium alloys, two titanium alloys, two stainless steels, and one alloy steel have been determined at elevated temperatures. Tensile data were also obtained for all materials except the stainless steels. Each material was tested at several temperatures as follows: 2014-T6, 7075-T6, and X7178-T6 aluminum alloys and M1-H24, AZ31-H24, and AZ31-0 magnesium alloys at 200°, 300°, 400°, 500°, and 600°F; 2024-T3 aluminum alloys at 212°, 300°, 400°, 500°, 600°, and 700°F; 2024-T81 and 2024-T86 aluminum alloys at 200°, 300°, and 400°F; annealed titanium, cold-rolled titanium, Type 301 half-hard stainless steel, Type 302 annealed stainless steel, and SAE 4130 alloy steel at 400°, 600°, 800°, and 1000°F. Specimens of all materials were aged for 0.5 hr at test temperature prior to testing.

The test materials used in the program were left-over portions of sheets which had been tested previously. They had been in storage for periods ranging from 3 to 5 yrs. Analysis of the test data suggested that long room temperature aging has a substantial effect on the elevated temperature properties of certain hard-rolled and heat-treated alloys. The properties of the fully annealed materials, however, appeared to be unaffected by protracted aging at room temperature.

Battelle Memorial Institute. WADC TR 52-251, Pt 5.
THE COMPRESSIVE CREEP BUCKLING OF METAL COLUMNS. Part 5 - Cyclic Loading. A. Rudnick, R. L. Carlson, G. K. Manning. AF 33(616)-3317. June 1958. ASTIA Document No. AD 155604. PB 151218.

Tests were conducted to determine the effect of load cycling on the creep buckling behavior of metal columns. Two materials were investigated: 2024-T4 aluminum alloy columns with length to radius of gyration ratios of 81.2 and 55.7 were tested at 350°F and 450°F; C-110M titanium alloy columns having a length to radius of gyration ratio of 90 were tested at 700°F and 800°F. The cycling period used was 24 hours, the time the load was on in that period was the variable. In addition to the cyclic load tests, constant load tests were conducted to obtain background information.

A linear relationship was found between the time to creep buckling under constant load and the reciprocal of the minimum creep deflection rate. This relationship was used in interpreting the cyclic load test results.

The effect of load cycling proved to be dependent upon the properties of the material at the testing temperature. Two criteria, based upon material stability characteristics, are set up to help classify the cyclic load data.

Southwest Research Institute. WADC TR 58-246.
DETERMINATION OF MATERIALS DESIGN CRITERIA FOR 6Al-4V TITANIUM ALLOY AT ROOM AND ELEVATED TEMPERATURES. J. K. Childs, M. M. Lemcoe. AF 33(616)-3348. August 1958. ASTIA Document No. AD 155863. PB 151274. Order from OTS $4.00.

In order to establish design criteria on the 6Al-4V titanium alloy, tensile, compressive, bearing and shear properties have been determined on both bar and sheet material at temperatures from 75 to 1000°F. In addition, tensile properties of sheet material were determined after 100-hour exposure at 600, 800 and 1000°F.

Test results were compared with recently established minimum requirements for tensile strength in order to derive design values for compression, bearing, and shear.

Other properties investigated include minimum bend radius for 105° bend and notched tensile strength at temperatures from 75 to 1000°F, and torsional properties at room temperature.

Axial load fatigue characteristics were investigated at temperatures from 75 to 1000°F for both smooth and notched specimens at stress ratios of 0 (stress-rupture), 0.3, 1.0, and infinity.

Descriptions of the test specimens, equipment, and procedures used are included. Test results are reported in tables and in curves showing the effect of temperature on the various mechanical properties. The derived design data and curves are presented in a form suitable for inclusion in ANC-5.

Battelle Memorial Institute. WADC TR 52-251, Part VI.
THE COMPRESSIVE CREEP BUCKLING OF METAL COLUMNS, Part VI. Effect of Initial Imperfection. Alfred Rudnick, Robert L. Carlson, George K. Manning. AF 33(616)-3317. October 1958. ASTIA Document No. AD 202491. PB 140907. Order from LC Mi $3.60, Ph $9.30.

Tests were conducted to determine the effect of the initial imperfection of a column on its creep-buckling characteristics. The material used was 6Al-4V titanium alloy. Columns with initial imperfections ranging from $\frac{L}{1000}$ to $\frac{L}{70}$ (where L is the column length) were tested at temperatures of 750, 850 and 900°F.

Initial imperfection was found to influence very strongly the creep-buckling behavior of a column. In general, a significant inter-relationship was found to exist between column load, lifetime, temperature, and imperfection, i.e., the effects of any one factor are dependent upon the magnitudes of the other factors.

The time-dependent secant- and tangent-modulus methods for estimating column capacity were investigated. It is shown that both estimates can be either conservative or nonconservative, depending upon the temperature and initial imperfection of the column.

The Larson-Miller time-temperature parameter technique was applied to the data. The usefulness of this and similar parameters for correlating column-creep data is discussed.

Southern Research Institute. WADC TR 58-365.
DETERMINATION OF TENSILE, COMPRESSIVE, BEARING AND SHEAR PROPERTIES OF SHEET STEELS AT ELEVATED TEMPERATURES. J. Robert Kattus, James

B. Preston, Herman L. Lessley. AF 33(616)-3876. November 1958. ASTIA Document No. AD 206075. PB 151592. Order from OTS $4.00.

The tensile, compressive, bearing, and shear properties of the following sheet metals were determined at various temperatures after exposure times of from 1/2 hour to 1000 hours at the test temperature:

1. A-286 austenitic alloy, quenched and tempered
2. 17-7 PH stainless steel, RH950 condition
3. Thermold J. alloy steel, quenched and tempered
4. Type 420 stainless steel, quenched and tempered
5. Type 422 stainless steel, quenched and tempered
6. 17-22 A (S) alloy steel, quenched and tempered

The A-286 alloy was tested over a temperature range from 75°F to 1200°F, the Thermold J from 75°F to 1100°F, and the other alloys from 75°F to 1000°F.

In all of the test alloys, the strength properties and moduli of elasticity decreased with increasing temperatures. The strength properties of the Thermold J, Type 420, Type 422, and 17-22 A (S) tended to decrease by varying amounts with increasing exposure times at the higher test temperatures. These decreases in strength are believed to be associated with structural changes produced by tempering. At lower temperatures, the properties of these materials did not vary significantly with exposure time, indicating that the structures were stable at those temperatures. The strength properties of the A-286 alloy and the 17-7 PH (RH 950) stainless varied somewhat erratically with increasing exposure times at the higher test temperatures as a result, probably, of aging phenomena in both of these precipitation-hardening alloys.

The simple ratio relationships between various properties under equivalent test conditions were approximately equal in magnitude and in consistency to those previously determined for other materials and reported in WADC Technical Report 56-340. For the entire ranges of materials and conditions used in this work, the consistency of the various property relationships ranged from ± 17% to ± 71%. Precise data on the mechanical properties of aircraft-structural materials can be obtained only by testing under the desired condition.

Southern Research Institute. WADC TR 58-440, Part I.
TENSILE PROPERTIES OF AIRCRAFT-STRUCTURAL METALS AT VARIOUS RATES OF LOADING AFTER RAPID HEATING. Haywood E. Dedman, Edmund J. Wheelahan, J. Robert Kattus. AF 33(616)-3996. November 1958. ASTIA Document No. AD 206074. PB 151566. Order from OTS $3.50.

The purpose of this work was to evaluate the short-time tensile properties of aircraft-structural materials under conditions of rapid heating, short times at temperature, and moderate to rapid rates of loading. The test specimens were heated to the test temperatures within ten sec, held for periods of time ranging from 10 sec to 1800 sec, and loaded at strain rates from 0.00005 to 1.0 in./in./sec. Major emphasis was placed on the accurate determination of 0.2% offset yield strength and ultimate tensile strength. The modulus of elasticity, percent elongation, proportional limit, and full stress-strain curves were determined with somewhat less accuracy but, nevertheless, with sufficient accuracy to establish trends.

Four steels--Chro-Mow tool steel, Thermold-J tool steel, Peerless-56 tool steel, and AM-350 precipitation-hardening stainless steel--and 6Al-4V titanium alloy were fully evaluated, the steels being tested at temperatures up to 1200°F and the titanium alloy up to 1000°F. Before being tested, all of these alloys were heat treated to high strength levels of about 300,000 psi for the tool steels, 200,000 psi for the AM-350, and 160,000 psi for the 6Al-4V.

Similar evaluations, except for the determination of complete stress-strain curves, had previously been carried out on seventeen alloys under contract AF 33(616)-424 and reported in WADC Technical Report 55-199 Parts I, II, and III. For these alloys, complete stress-strain curves were obtained in the present contract under all test conditions employed in the previous contract.

The test results show that, at the lower test temperatures, the strength properties of the test alloys tended to increase slightly or to remain constant with increasing strain rates. Near the maximum test temperatures, the effect of strain rate became much greater, the strength properties increasing greatly with increasing strain rates.

Probably as a result of structural changes associated with tempering and with overaging the strength of the steel alloys decreased with increasing holding times at 1200°F. The tensile properties of the steel alloys at lower temperature and of the titanium alloy at all test temperatures were not affected by variations in holding time.

WADC TR 58-400.
MECHANICAL PROPERTIES OF 17-7 PH AND PH 15-7 MO. STAINLESS STEEL. Alton W. Brisbane. January 1959. ASTIA Document No. AD 208322. PB 151703. Order from OTS $1.00.

Two stainless steels were investigated in this work, 17-7PH (RH-950 Condition) and PH 15-7 Mo. (RH-950 and TH-1050 Conditions).

Tensile stress-strain test results for the 17-7 PH and the PH 15-7 Mo. stainless steel are presented for temperatures from room temperature to 1000°F. Compression and bearing data were also obtained at room temperature.

A comparison is made between these two stainless steels and other materials.

WADC TR 58-672.
MECHANICAL PROPERTIES OF AM350 and AM355 STAINLESS STEELS. Alton W. Brisbane. February 1959. ASTIA Document No. AD 208664. PB 151706.

In this report are presented the results of smooth and notched tension tests and compression, bearing, bending and Charpy V-notched impact tests. The tests were conducted on two heats of Allegheny-Ludlum AM-350 stainless steel sheet which is a Cr.-Ni.-Mo. stainless steel hardenable by subzero cooling or double aging, and one heat of AM-355 3/4 inch diameter bar material. This material was tested at room and elevated temperatures in tension plus some subnormal and elevated temperature Charpy impact tests.

The material was given several heat treatments to determine the effect on mechanical properties. Varying the aging temperatures had some effect on the strength properties and ductility.

The sub-zero treatment with no elevated temperature aging gives the maximum strength but not the best ductility.

The -100°F +950°F heat treatment gives the best ductility for this material, as shown in the elongation, reduction in area and energy absorbed in the impact test.

Southern Research Institute. WADC TR 58-440, Part II.
TENSILE PROPERTIES OF AIRCRAFT-STRUCTURAL METALS AT VARIOUS RATES OF LOADING AFTER RAPID HEATING. J. Robert Kattus. AF 33(616)-3996. May 1959. ASTIA Document No. AD 213834. PB 151895. Order from OTS $3.00.

In this report, a summary and an analysis are presented of the results of five years of investigation of the short-time tensile properties of structural metals. The specimens that were tested in this investigation were heated to various elevated test temperatures within 10 sec, were held at test temperature for periods of time from 10 sec to 30 minutes, and were then loaded to failure at strain rates from 0.00005 to 1.0 in./in./sec.

The strength properties of all of the test metals tended to decrease with increasing temperatures. Variations in holding time at test temperature had no significant effect upon the tensile properties of the structurally stable metals. The tensile properties of the unstable alloys changed with variations in holding time at certain temperatures as a result of time-temperature-dependent structural changes. In general, increasing strain rate produced increased strength in the test metals, the percentage increases in strength being relatively small in the temperature ranges of low-temperature behavior and quite large in the temperature ranges of high-temperature behavior. Under certain conditions, the effects of time-temperature-dependent and strain rate-temperature-dependent structural changes were superimposed upon, and sometimes obscured, the inherent effects of temperature and of strain rate.

An evaluation of time-temperature and rate-temperature parameters indicated that short-time tensile properties cannot be expressed with a high degree of accuracy as invariant functions of such parameters. Nevertheless, illustrations of short-time tensile properties as functions of the Larson-Miller parameter are quite useful for general comparisions of the strength levels of various materials over wide ranges of conditions. A duplex method that was developed for the presentation of tensile-strength data is useful primarily for the accurate condensation of tensile data over wide ranges of conditions into simple one-page plots. Such presentations, however, are not particularly useful for comparisions among different materials or for extrapolations of tensile data beyond the experimental conditions.

University of Illinois. WADC TR 56-330, Part XI.
THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS Part XI, Photoelastic Analysis of I-Beams With Elliptic-Type Web Cutouts. Will J. Worley. AF 33(616)-5658. September 1959. ASTIA Document No. AD 229931. PB 161303. Order from OTS $0.75.

This is the final report on the photoelastic study of I-Beams, and covers the period from May 1, 1958 to date.

The data presented are intended to supplement the data presented in WADC TR 56-330, Part VII under Contract AF 33(616)-2753. Report VII presented information on the fully plastic strength while the present report discusses the elastic stress distribution in the web section of I-Beams with various shapes of elliptic-type web cutouts.

The prediction of the location of the fully plastic hinges using the shear difference method is discussed for one shape of web cutout. The method shows reasonable agreement when compared with available data on the actual aluminum alloy I-Beam.

Battelle Memorial Institute. WADC TR 55-150, Part 8.
MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS, Part 8. The Creep Behavior of Selected Materials in the Range Up to 1 Per Cent Net Creep Strain and 1000 Hours. R. J. Favor, W. P. Achbach, H. J. Grover. AF 33(616)-3965. September 1959. ASTIA Document No. AD 229933. PB 161302. Order from OTS $0.75.

The objectives of this study were to compile and to evaluate creep strain versus time data on airframe structural materials. The range of interest, as recommended by the Elevated Temperature Task Group of the ANC-5 Panel, includes creep strain up to 1 per cent and time up to 1000 hours. Only two reference sources dealt with creep of airframe structural materials within the conditions of strain and time imposed. Although both of these sources have considerably extended the knowledge of creep behavior in the early stages of creep, it appears that neither has encompassed adequately the regions of interest specified. Reasons for this conclusion are discussed in this report. It should be emphasized that for creep strain above about 1 per cent, both sources of data appear adequate.

National Bureau of Standards. WADC TR 56-645, Part III.
PROPERTIES OF GLASSES AT ELEVATED TEMPERATURES. M. J. Kerper, C. C. Diller, E. H. Eimer. AF 33(616)-56-13. October 1959. ASTIA Document No. AD 230656.

In order to establish realistic design criteria applicable to several special glasses certain physical properties have been determined throughout their useful temperature range.

The modulus of rupture and modulus of elasticity at several temperatures are presented for Corning Glass Works glasses, 1723 (Aluminosilicate), 7740 (Borosilicate), 7900 (95% Silica), and 7940 (Fused Silica). Stress-rupture and creep results for semi-tempered and tempered soda-lime-silica glass are also presented.

Data are given in the form of tables and graphs together with a complete statistical analysis of all results.

Marquardt Corp. WADC TR 59-484.
SHORT TIME, ELEVATED TEMPERATURE STRESS-STRAIN BEHAVIOR OF TENSILE, COMPRESSIVE, AND COLUMN MEMBERS. Eugene C. Bernett. AF 33(616)-6043. December 1959. PB 161492. Order from OTS $2.75.

The short time tension and compression load carrying properties of four aircraft type sheet materials (namely: 2024-T81 aluminum, 17-7PH (TH 1050) stainless steel, annealed N-155 (multimet) alloy, and annealed 6Al-4V titanium alloy were evaluated. Tensile and compressive strength properties were measured at strain rates ranging from 0.00001 to 0.1 in./in./second. Test temperatures up to 2000°F and hold times at temperature of 2 and 30 minutes were investigated. The compressive creep properties (for times up to 15 minutes) were also evaluated and methods are shown for use of these data to define the behavior of columns subjected to high temperature and stresses.

Changes in testing speed produced major differences in the tensile and compressive strengths, particularly at the elevated temperatures. It was also demonstrated that time-dependent deformation rates at high stresses and high temperatures are very rapid. Large amounts of plastic strain can occur even when the time involved is of the order of one second or less.

Columbia University. WADC TR 58-69, Part II.
ON STRESS INTERACTION IN FATIGUE AND A CUMULATIVE DAMAGE RULE PART II. 7075 Aluminum Alloy. Alfred M. Freudenthal, Robert A. Heller. AF 33(616)-3982. January 1960. ASTIA Document No. AD 234448. PB 161837. Order from OTS $0.75.

The object of this investigation was the determination of the effects of stress interaction, under randomized stress distributions representing gust and maneuver loads on aircraft wings, on the fatigue life of 7075 aluminum alloy specimens.

On the basis of numerous rotating bending fatigue tests, it has been demonstrated that the linear cumulative damage rule does not provide a safe fatigue life estimate of unnotched specimens. A non-linear rule representing the test results fairly well has been proposed.

University of Illinois. WADC TR 56-330, Part XII.
THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS PART XII Eccentrically-Loaded Tension Members and Columns Made of 17-7PH Stainless Steel and Ti 155A Titanium Alloy and Tested at Various Temperatures. O. M. Sidebottom, S. Dharmarajan, J. L. Gubser, J. D. Leasure. AF 33(616)-5658. February 1960. PB 161836. Order from OTS $2.25.

Experimental data were obtained from eccentrically-loaded columns made of 17-7PH stainless steel and Ti 155A titanium alloy and tested at room temperature, from eccentrically-loaded columns made of 17-7PH stainless steel and tested at 1000°F, and from eccentrically-loaded tension members and columns made of Ti 155A titanium alloy and tested at 800°F. In most cases the creep tests were limited to 30 mins. At the elevated temperatures the inelastic deformation was time dependent for the 17-7PH stainless steel and was mostly time independent for the Ti-155A titanium alloy.

The theoretical analysis of room temperature tests data obtained from Ti-155A titanium alloy members tested at 800°F was based on the interaction curve-moment-load curve theory. The arc hyperbolic sine theory was used in the analysis of data obtained from 17-7PH stainless steel members tested at 1000°F. In all cases, good agreement was found between theory and experiment.

WADC TR 59-25.
INVESTIGATION OF THE UNNOTCHED AND NOTCHED FATIGUE BEHAVIOR OF SEVERAL HEAT RESISTANT MATERIALS FOR ENGINE BOLTS. D. M. Forney, Jr., Douglas Y. Wang. February 1960. ASTIA Document No. AD 236684. PB 161930. Order from OTS $1.50.

An investigation of the fatigue behavior of 3/8 inch engine bolts at various temperatures is described for the heat resistant alloys AMS 5735 (A-286), Udimet 500, Inconel 700 and Incoloy 901. Tests were also performed under axial stress on unnotched fatigue specimens and fatigue specimens with a theoretical stress concentration factor of 3.4. The data are presented as S-N diagrams to show the effect on fatigue strength of temperature, stress concentration, stress magnitude and bolt thread rolling.

The results of this investigation suggest the possibility of using fatigue test data obtained with single notched specimens to evaluate other heat resistant alloys for possible engine bolt applications thereby reducing the significant expense of fabrication of experimental lots of bolts for test.

Advanced Metals Research Corporation. WADD TR 60-793.
DIFFUSION IN REFRACTORY METALS. N. L. Peterson. AF 33(616)-7382. March 1960. ASTIA Document No. AD 257860. Order from OTS $3.00.

Data available in the open literature on diffusion in tungsten, tantalum, molybdenum, niobium, platinum, hafnium, zirconium, vanadium, chromium, and titanium is reviewed and evaluated. Information on ninety-five binary systems and thirteen ternary or higher order systems is reported.

Care was taken to make this report as complete as possible at this time. For some systems where no diffusion data was available, diffusion coefficients were estimated from existing theories or from data on diffusion controlled processes.

Cornell Aeronautical Laboratory, Inc. WADC TR 59-572.
INVESTIGATION OF THE COMPRESSIVE, BEARING, AND SHEAR CREEP-RUPTURE PROPERTIES OF AIRCRAFT STRUCTURAL METALS AND JOINTS AT ELEVATED TEMPERATURES. Luke A. Yerkovich. AF 33(616)-5772. March 1960. PB 161717. Order from OTS $2.50.

The determination of the high-temperature mechanical properties of aircraft structural materials is a prerequisite to efficient design when elevated temperature service is to be expected. Normally, these properties are obtained from the conventional short-time tensile and creep-rupture tests and as such are not necessarily applicable for stress conditions other than tension. The present program was conducted to examine the high-temperature strength and deformation characteristics of two high-strength airframe alloys when exposed independently to a variety of stresses under both short and long time loading. Specifically, the object of the program was to determine the high-temperature tension, compression, bearing and shear properties of selected airframe alloys with the ultimate purpose of correlating tension behavior with behavior under the various other types of loads and also of applying these basic data to predict the behavior of riveted joints undergoing creep deformation in tension, bearing and shear.

This report summarizes in tabular and chart form the high temperature properties of 16 V- 2.5 Al titanium alloy and Vascojet 1000 alloy steel in tension, compression, bearing and shear. In addition, correlations of the tensile creep-rupture properties with corresponding compression, bearing and shear creep-rupture properties are presented.

The creep-rupture characteristics of single-rivet lap type joints and two-rivet doubler type joints, which represent components of multiple riveted assemblies, prepared from the test alloys are presented herein. Correlations between measured joint creep-rupture and predicted joint creep-rupture are also included.

Bell Aircraft Corp. WADC TR 59-702, Part I.
MECHANICAL PROPERTIES OF SELECTED ALLOYS AT ELEVATED TEMPERATURES. Harry A. Pearl, George F. Kappelt, Edmund J. King. AF 33(616)-5760. March 1960. PB 161761. Order from OTS $4.00.

Six materials in plate and bar form were subjected to tension, compression, bearing, and shear stresses at various temperatures after exposure times of 1/2, 10, 100, and 1000 hours at selected test temperatures. The materials studied and the temperature range of testing were:

Materials	Testing Temperature Range
AM 355	R. T. - 1000°F
PH15-7Mo	R. T. - 1000°F
HK31-H24	R. T. - 600°F
A 286	R. T. - 1200°F
Udimet 500	R. T. - 1700°F
Inconel X	R. T. - 1500°F

In general, the test results show that most of the material properties, regardless of the type of stress imposed, decrease with temperature and time. Time of exposure was not as effective in lowering the properties as

the temperature. The A-286 alloy did show mechanical properties higher after prolonged exposure due to aging at some temperatures. The sharp drop off in properties that the other alloys exhibited at the higher temperatures was not characteristic of the A-286 alloy.

Since mechanisms of failure are different, precise data under a given stress can be obtained only by testing under the desired conditions. Comparisons between alloys are made only where the temperature range of operation is common to both.

Bell Aircraft Corporation. WADC TR 59-702, Part II.

MECHANICAL PROPERTIES OF SELECTED ALLOYS AT ELEVATED TEMPERATURES PART II. Design Criteria of Silicon Carbine. Harry A. Pearl, John M. Nowak, Harry G. DeBan. AF 33(616)-5760. March 1960. PB 161723. Order from OTS $2.75.

A study was made of nondestructively testing silicon carbide by density and density uniformity, dynamic modules by sonic technique, X-ray diffraction under transverse load, electrical resistivity, and internal friction. Dynamic modules of silicon carbide was experimentally determined at 80°F and 2200 F. Modules of rupture tests were conducted at 80°F, 2200°F and 2400°F.

The variability of the properties of the silicon carbide and the lack of simple correlations between the properties and geometry require the use of a statistical approach to correlate mechanical properties and geometry. A theoretical analysis is presented on the effect of specimen size, surface finish, and methods of loading on the strength properties of silicon carbide.

Available literature and manufacturers' property data for various types and forms of commercially available silicon carbide are tabulated. Various areas of possible application of silicon carbide in aircraft and missiles and design parameters for leading edge applications are given.

Westinghouse Electric Corporation. WADC TR 57-344, Part IV.

DEVELOPMENT OF NIOBIUM-BASE ALLOYS. Richard T. Begley, William N. Platte. AF 33(616)-5754. May 1960. PB 161 936. Order from OTS $2.75.

Vacuum tensile data were obtained for pure niobium from room temperature to 1371°C (2500°F). The data for niobium followed the general pattern exhibited by other pure refractory metals. The ductile-brittle transition (in impact) for commercial purity, arc melted niobium was close to room temperature.

The effect of binary additions of Ti, Zr, Hf, V, Mo, W, Re, Al, and Y on the hardness and workability of niobium was determined. Nb-Ti and Nb-Y alloys had excellent cold workability.

Mechanical property data were obtained at room temperature and 1093°C (2000°F) on niobium containing additions of Ti, Zr, Hf, V, Mo, and W. Of the elements studied, vanadium additions were the most effective strengtheners. Nb-Zr and Nb-Hf alloys having high oxygen contents had very good high temperature properties. It appears that the interaction of oxygen with the alloy addition may be responsible for the high strength. Tensile data were also obtained on alloys having ternary and quaternary additions of Ti, Zr, Hf, and Mo. Nb-Ti-Zr-Hf alloys exhibited yield strengths well in excess of 40,000 psi at 1093°C (2000°F).

Welding studies on a number of niobium alloys containing additions of Ti, Zr, Hf, V, and W were carried out. Satisfactory welds were obtained in all the alloys studied. Bend test data were obtained on the weld specimens.

Forest Product Laboratory. WADC TR 55-319, Sup. 2.

EFFECTS OF WEATHERING ON THE MECHANICAL PROPERTIES OF FOUR REINFORCED PLASTIC LAMINATES. Kenneth Kimball. DO 33(616)-58-1. September 1960.

Four different reinforced plastic laminates were subjected to outdoor weathering at three sites having entirely different weather conditions. After completion of the exposure, the laminated panels were tested in flexure at the U. S. Forest Products Laboratory. Data on the effect of 3 and 12 months' exposure on the flexural properties and the appearance of the laminates are presented in this report.

Another series of panels are still undergoing exposure at all three sites, and this series will be tested after 3 years' exposure. Upon completion of the tests on these panels, all data will be compiled in a final report.

Data obtained from the tests to date show that the effect of outdoor exposure varies greatly with different types of laminates and conditions of exposure. The greatest loss in strength was usually associated with exposure at the Florida site. In general, normal and wet flexural strengths of weathered glass-fabric laminates were 90 percent or more of the control strengths, but the high-temperature strength was only about 60 to 80 percent of the control. The phenolic-asbestos laminate had a higher over-all strength retention than the glass-fabric laminates.

Results of strength tests of over 700 specimens are presented in this interim report.

National Bureau of Standards. WADD TR 60-56, Part I.

A COMPENDIUM OF THE PROPERTIES OF MATERIALS AT LOW TEMPERATURE (PHASE I) PART I. Properties of Fluids. Victor J. Johnson, General Editor. AF 33(616)-58-4. October 1960. ASTIA Document No. AD 21-9644. PB 171618. Order from OTS $6.00.

This first phase of the Compendium covers ten properties of ten fluids (Part I), three properties of solids (Part II), and an extensive bibliography of references (Part III). Density, expansivity, thermal conductivity, specific heat and enthalpy, transition heats, phase equilibria, dielectric constants, absorption, surface tension and viscosity for the solid, liquid and gas phases of helium, hydrogen, neon, nitrogen, oxygen, air, carbon monoxide, fluorine, argon and methane are given wherever adequate data could be collected. Thermal expansion, thermal conductivity and specific heat and enthalpy are given for a number of solids of interest in cryogenic engineering. Data sheets, primarily in graphic form, are presented from "best values" of data collected. The source of the material used, other references and tables of selected values with appropriate comments are furnished with each data sheet to document the data presented. Conversion tables and other helpful information are also included.

National Bureau of Standards. WADD TR 60-56, Part II.

A COMPENDIUM OF THE PROPERTIES OF MATERIALS AT LOW TEMPERATURE (PHASE I) PART I. Properties of Solids. Victor J. Johnson. AF 33(616)-58-4. October 1960. ASTIA Document No. AD 249786. PB 171619. Order from OTS $4.00.

This first phase of the Compendium covers ten properties of ten fluids (Part I), three properties of solids

(Part II), and an extensive bibliography of references (Part III). Density, expansivity, thermal conductivity, specific heat and enthalpy, transition heats, phase equilibria, dielectric constants, adsorption, surface tension and viscosity for the solid, liquid and gas phases of helium, hydrogen, neon, nitrogen, oxygen, air, carbon monoxide, fluorine, argon and methane are given wherever adequate data could be collected. Thermal expansion, thermal conductivity and specific heat and enthalpy are given for a number of solids of interest in cryogenic engineering. Data sheets, primarily in graphic form, are presented from "best values" of data collected. The source of the material used, other references and tables of selected values with appropriate comments are furnished with each data sheet to document the data presented. Conversion tables and other helpful information are also included.

National Bureau of Standards. WADD TR 60-56, Part III.

A COMPENSIUM OF THE PROPERTIES OF MATERIALS AT LOW TEMPERATURE (PHASE I) PART III. Bibliography of References (Cross-Indexed). Victor J. Johnson, General Editor. AF 33(616)-58-4. October 1960. ASTIA Document No. AD 249777. PB 171620. Order from OTS $3.00.

This first phase of the Compendium covers ten properties of ten fluids (Part I), three properties of solids (Part II), and an extensive bibliography of references (Part III). Density, expansivity, thermal conductivity, specific heat and enthalpy, transition heats, phase equilibria, dielectric constants, adsorption, surface tension and viscosity for the solid, liquid and gas phases of helium, hydrogen, neon, nitrogen, oxygen, air, carbon monoxide, fluorine, argon and methane are given wherever adequate data could be collected. Thermal expansion, thermal conductivity and specific heat and enthalpy are given for a number of solids of interest in cryogenic engineering. Data sheets, primarily in graphic form, are presented from "best values" of data collected. The source of the material used, other references and tables of selected values with appropriate comments are furnished with each data sheet to document the data presented. Conversion tables and other helpful information are also included.

WADD TR 60-419.

EFFECTIVE STRESS CONCENTRATION FACTORS FOR FLIGHT VEHICLE MATERIALS UNDER VARIOUS CONDITIONS DURING FATIGUE TESTING. V. F. Lardenoit. October 1960. PB 171410. Order from OTS $3.00.

This report presents in the form of tables and curves, the effective stress concentration factors, K_f, for a number of aircraft materials subjected to various conditions during fatigue testing. The influence on K_f is shown for such parameters as temperature, stress ratio, specimen size, direction of testing, test frequency, fabrication of notches, heat treatment, grain size, and for titanium and its alloys, interstitial constituents and hydrogen level.

Because K_f is sensitive to so many variables, no attempt is made to relate it to any of the above listed parameters.

Crucible Steel Company of America. WADD TR 60-489.

INVESTIGATION TO DEVELOP OPTIMUM PROPERTIES IN FORGED Ti-7Al-4Mo. James E. Hamer. AF 33(616)-6122. October 1960. PB 171 546. Order from OTS $2.75.

The influence of six processing sequences on the microstructure, mechanical properties and heat treatment response of the Ti-7Al-4Mo alloy has been studied. The information developed demonstrates the critical effects of processing history in controlling the material performance levels and emphasizes the importance of such considerations in attaining "optimum" values.

Forging entirely above the beta transus produced the usual Widmanstatten type of structure found in titanium alloys after such processing. As expected, the creep resistance of this structure was excellent; however, heat treatment response and tensile ductility were poor, and a reduction in charpy impact strength of 3/4" rod was noted for all thermal treatment conditions. Forging entirely below the beta transus developed an equiaxed microstructure and generally good properties except for reduced creep resistance. Duplex forging, i.e., forging initially above the beta transus, cooling to room temperature, and reheating to below the beta transus for finish forging, produced a nearly equiaxed microstructure and general property levels quite similar to all below-beta-forging but with improved creep resistance.

In general, notch tensile and notch stress rupture did not show any direct relationships with processing history. Notch rotating beam fatigue tests for above-beta forged material showed more scatter than those on material forged below the beta transus though the endurance limit was not greatly affected.

National Bureau of Standards. WADC TR 57-374, Part V.

PHYSICAL PROPERTIES OF HIGH TEMPERATURE MATERIALS - PART V. Thermal Diffusivity of Magnesia-Stabilized Zirconium Oxide at High Temperatures. Howard W. Flieger, Jr., Friedrich P. Knudsen, Defoe C. Ginnings. AF 33(616)-56-21. October 1960. ASTIA Document No. AD 269130. Order from OTS $2.75.

As a part of a program developing standards of thermal conductivity and thermal diffusivity of refractory materials at high temperatures, thermal diffusivity measurements have been made on dense polycrystalline zirconium oxide which had been stabilized with magnesium oxide. The results indicate that the stabilization of the zirconium oxide was inadequate, giving progressively increasing values of thermal diffusivity after cycling to temperatures of about 1200°C. Thermal expansion and X-ray measurements indicate that the increase in thermal diffusivity values correspond to a change from cubic to monoclinic crystalline form.

Southern Research Institute, WADC TR 59-744, Volume V.

INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOLUME V. Mechanical Properties of Bare and Coated Molybdenum Alloy. J. Robert Kattus, James B. Preston, Herman L. Lessley. AF 33(616)-6034. November 1960. ASTIA Document No. AD 251810. PB 171 986. Order from OTS $3.00.

The mechanical properties of arc-cast, fully recrystallized molybdenum 1/2%-titanium alloy - bare, W-2 coated, and Durak-MG coated -- were determined at temperatures up to 3000°F in short-time tension and compression and in tension and compression creep for times up to 10 hr. The purpose of the work is to provide data, for this alloy upon which to base the design of leading edges for hypersonic boost-glide vehicles.

In inert atmospheres, this molybdenum alloy retains useful strength and good ductility at temperatures up to 3000°F. Both coatings have an extreme

embrittling effect and a weakening effect on the base metal at room temperature. At 1200°F and above, the coated material is equivalent in strength and only slightly less ductile than the bare material. When properly applied, both coatings provide good oxidation protection in still air at temperatures up to 3000°F and for times up to 10 hr. Under tension and compression loads, the coatings can absorb, without failure, elastic strain and varying degrees of plastic strain from less than 1% up to 18% depending upon the conditions.

With W-2 or Durak-MG coatings of optimum quality, the molybdenum 1/2%-titanium alloy is recommended for applications that are limited to no plastic strain at room temperature and to small amounts of plastic strain at elevated temperatures to a maximum of 3000 F for times up to 10 hr. Although the coatings can absorb considerable plastic strain under many conditions, further research is needed to safely extend their usefulness beyond small amounts of plastic strain at 2100 F and at room temperature. Future research is recommended to minimize or to eliminate the embrittling effects of coatings on the base material and to develop quality-control methods to assure consistent optimum quality in the protective coatings.

Battelle Memorial Institute. WADC TR 59-744, Volume VI.
INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOLUME VI. Determination and Design Application of Mechanical Properties of Bare and Coated Graphite. Alfred Rudnick, Robert L. Carlson, George K. Manning. AF 33(616)-6034. November 1960. ASTIA Document No. AD 257731. Order from OTS $3.50.

The purpose of this contract was to investigate the feasibility of utilizing available heat-resistant materials in the fabrication of leading edges for hypersonic boost-glide vehicles. This volume presents the results of a study on the mechanical properties of a siliconized Grade ATJ graphite. The mechanical properties were studied as functions of temperature (to 2950°F), strain rate, size, and stress state. Supplementary studies were conducted to determine the room temperature properties of the uncoated material and the degree of anisotropy of both the coated and uncoated material.

Gross variations in the material tended to obscure the effects of the parameters under consideration. Inspection techniques were developed, however, which will reduce these variations. With proper selection of material quality, it is believed that the siliconized Grade ATJ graphite can be used effectively as a structural component at elevated temperature in an oxidizing atmosphere.

Douglas Aircraft Company, Inc. WADD TR 60-363.
INVESTIGATION INTO "MORE COMPLETE USE OF STRUCTURAL MATERIALS" THROUGH A STUDY OF THE STRESS-TEMPERATURE-TIME CONDITIONS OF A RE-ENTRY VEHICLE. J. E. Davis, J. E. Fischler, J. W. Lobbett, D. Saltman, J. W. Stanwood, N. A. Tiner. AF 33(616)-6680. November 1960. ASTIA Document No. AD 249787.

In the past, vehicles operated in a fairly low temperature environment. The selection of materials were primarily dependent on the quasistatic and dynamic loads. With the advent of manned hypersonic glide reentry vehicles, the temperature and time parameters were found to significantly affect the selection of materials. In this report, the criteria for including the effects of temperature and time are clarified by using six "measuring sticks." Two materials, Inconel 718 and Haynes 25 were evaluated for repeated exposure to a typical hypersonic re-entry glide trajectory.

Bell Aerosystems Company. WADC TR 59-744, Volume VII.
INVESTIGATION OF FEASIBILITY OF UTILIZING AVAILABLE HEAT RESISTANT MATERIALS FOR HYPERSONIC LEADING EDGE APPLICATIONS VOLUME VII. Oxidation Resistance of Bare and Coated Molybdenum Alloy and Graphite. Donald J. Powers, John A. Dickson, Joseph C. Conti, Frank M. Anthony. AF 33(616)-6034. December 1960. ASTIA Document No. AD 257732. Order from OTS $3.50.

The purpose of this contract was to investigate the feasibility of utilizing available heat resistant materials in the fabrication of leading edges for hypersonic boost-glide vehicles. This particular volume presents the results of oxidation resistance tests of bare and coated 0.5% titanium alloy of molybdenum and ATJ graphite. Chromalloy W-2 and Durak-MG coatings were evaluated on the molybdenum alloy while the ATJ graphite was coated by the National Carbon Company's siliconizing process.

Tests were conducted in five different facilities including three arc plasma jets. Specimen temperatures ranged from 2000°F to 3400°F. Test times ranged from approximately 1 minute to 4 hours.

Both uncoated materials reacted exothermically under oxidizing conditions. Surface recession was the major result of the high temperature exposure in the oxidizing environments. The surface recession was quite predictable and uniform; it was a function of specimen temperature and environmental pressure.

A high degree of failures were encountered on all coated specimens. The failures were dependent on time and temperature but there was considerable scatter in the results. For the coated molybdenum specimens the edges and corners were most prone to failure. Failures of the siliconized ATJ specimens were more or less randomly located over the surface area.

Reactive Metals, Inc. WADD TR 60-404.
A STUDY OF THE EFFECT OF ELECTRON BEAM MELTING ON COMPOUNDS AND METALS. R. L. Martin, S. R. Seagle, O. Bertea. AF 33(616)-5603. January 1961. ASTIA Document No. AD 258588. Order from OTS $2.25.

Because of the high temperatures and vacuum that can be obtained during electron beam melting, this process offers the potential of purifying high melting point materials. This program consists of a study of the effect of the electron beam melting process on boron, boron-carbon alloys, tantalum carbide, titanium carbide, zirconium di-boride, hafnium, tungsten, cobalt, vanadium and beryllium. In addition, deoxidation studies were carried out on beryllium, vanadium, cobalt and molybdenum. When possible, microstructures, tensile properties, and chemical compositions of the materials melted were obtained.

The compounds studied either could not be melted or were brittle after melting. The interstitial content of all the metals was reduced by the electron beam melting process. By the addition of deoxidants, the interstitial contents of molybdenum and beryllium were reduced. Improvement in the tensile properties of vanadium and molybdenum were obtained by the addition of deoxidants to these metals.

California Inst. of Tech. Pasadena. Technical Report No. 1. AFOSR-TN-55-124.

GENERAL METHODS OF SYNTHESIS FOR ELECTRICAL-MECHANICAL ANALOGIES OF AIRCRAFT STRUCTURES. G. D. McCann and R. H. MacNeal. AF 18(600)669. October 1954. ASTIA Document No. AD 58331.

A general treatment is presented of the methods of analysis which have been developed successfully for the vibration studies of airframes together with the corresponding electrical circuit analogies that must be programmed into the electric analog computer. This computer type is distinguished from electronic differential analyzers in that passive circuit elements consisting of decade resistors, capacitors, inductors, and multiwinding, multitap transformers are utilized directly, as well as amplifiers and other electronic devices. The passive electrical elements are used to represent analogous passive elements of the physical system under study when a 1:1 physical analogy can be developed between electric circuit theory and physical system; thus, both currents and voltages in the computer take on physical significance, whereas in electronic differential analyzer techniques only voltages are used to represent voltages. In other cases, where amplifiers are employed, the passive elements are utilized to simulate transfer functions, reducing the number of electronic amplifiers that would be required if only electronic differential analyzers were used.

California Inst. of Tech. Pasadena. Technical Report No. 4. AFOSR-TN-55-120.

VIBRATIONS OF COMPOSITE SYSTEMS. R. H. MacNeal. AF 18(600)669. October 1954. ASTIA Document No. AD 63118.

This study is concerned exclusively with the vibrations of linear, conservative mechanical systems and their electrical equivalents. The concepts of "normal mode" and of "impedance" are brought together and interrelated in a number of ways. It is shown, for example, that normal modes can be used to compute "impedances", and that impedance measurements, in turn, can be utilized to obtain normal modes. The methematics used in this analysis of vibration is presented in the language of matrix algebra. With the exception of matrix theory, no "advanced" mathematical concepts are employed. The solution is given for several examples relating to the vibrations of a conventional airplane, and a comparison is made with analog computer results.

California Inst. of Tech., Pasadena. Technical Report No. 2. AFOSR-TN-55-121.

A STUDY OF THE EFFECTS OF DAMPING ON NORMAL MODES OF ELECTRICAL AND MECHANICAL SYSTEMS. S. F. Crumb. AF 18(600)669. January 1955. ASTIA Document No. AD 58338.

A general investigation is presented to some of the properties of free and forced vibrations in linear, nonconservative systems. Particular emphasis is placed upon the problems which arise in normal mode studies made on the electric analog computer at the California Institute of Technology. In Part I the major problems are defined, and limitations of the study are discussed. Part II contains a review of the basic theory of normal modes. In Part III, modifications of normal mode concepts, as applied to damped systems, are examined. "Small damping" criteria are discussed, and a set of theorems of small damping is presented. In Part IV, a series of normal mode analog circuits for damped systems are developed. Part V is a study of uniform damping, generalizing and extending some of the work of Rayleigh, Bode, and Guillemin. It is shown that for any type of uniform damping, all of the basic normal mode concepts are preserved. In Part VI, the theory of mode separation in uniformly damped systems is considered. Criteria for determining mode frequencies and mode parameters are developed. A multiple drive method of exciting normal modes is proposed. In Part VII, some of the methods of Part VI are extended to nonuniformly damped systems. Equivalent orthogonal systems are proposed to approximate the behavior of systems with moderately nonuniform damping. A quantitative measure of nonuniformity is presented. In Part VIII, numerical examples and experimental results in support of the theory are presented. Concluding remarks are made in Part IX. (Contractor's abstract)

Carnegie Inst. of Tech. (Dept. of Mathematics) Pittsburgh, Pa. Technical Report No. 2. AFOSR-TN-55-50.

A NOTE ON THE TRANSVERSE VIBRATION OF A TUBE CONTAINING FLOWING FLUID. G. H. Handelman. AF 18(600)1236. February 1, 1955. ASTIA Document No. AD 56337.

A study was made to determine the characteristics of the low-vibration frequencies for various end conditions for 2 ranges of the flow velocity solely from the structure of the differential equation without determining specific solutions. The differential equation in nondimensional form, developed by Long (Experimental and Theoretical Study of Transverse Vibration of a Tube Containing Flowing Fluid, American Society of Mechanical Engineers, Preprint 54-A-22), was used. A discussion is included on the determination of small flow velocities and velocities near the critical velocity. Data indicated that the perturbation terms can be computed step-by-step by quadratures. In the supported-end cases, the critical velocity can be determined first; perturbation from this point in powers of D serve as a check on the perturbation solution in terms of power u, where D is the eigenvalue found by removing the time-dependent term from the partial differential equation, and u is the flow velocity in nondimensional terms.

California Inst. of Tech., Pasadena. Technical Report No. 3. AFOSR-TN-55-148.

A STUDY OF THE ACCURACY OF LUMPED PARAMETER AND ANALOG COMPUTER REPRESENTATIONS OF CANTILEVERED BEAMS UNDER CONDITIONS OF STATIC STRESS AND DYNAMIC VIBRATIONS. G. D. McCann and H. S. Braham. AF 18(600)669. April 1955. ASTIA Document No. AD 63173.

The cantilevered beam in simple bending is a basic element of airframes as analyzed for stress, dynamic vibration, and aero-elastic analysis. The accuracy with which this element can be represented by lumped parameter approximations, and on the direct analogy electric analog computer, has been studied in detail. Concerning static loading, finite difference analogies with both full and half cell end termination and the Russell analog were investigated. Exact solutions, calculated lumped parameter, and analog computer solutions were obtained for comparison. It was found that for the wide range of beam conditions studied, the computer parasitic errors in recording deflections and slopes lie within a maximum of 2.6% with the error 1% or less for most cases. Considering the effects of finite difference lumping alone, it was found that half cell

termination gives greater accuracy for beams having variation in the (EI) properties that do not exceed the second power of distance along the beam, while the full cell termination analog gives better accuracy for higher rates of taper. Four finite difference cells are required for the uniform beam to reduce the slope and deflection errors to 3% and 8 cells for 1%. For the extreme fourth power taper, 4 cells produce a 10% error and 16 cells are required to reduce the error to 1%. The Russell analog is exact if loading is applied only at the ends of cell divisions. Concerning dynamic vibrations, 3 types of analogies were considered; the finite difference analog with both full and half cell end terminations, the modified Russell analog, which is the finite difference analogy with mass lumped at the center of the cells (half cell termination only was used), and the Russell analog. Exact, calculated lumped parameter and analog computer solutions were obtained for the first 15 free vibration mode frequencies and shapes. Shears and moments were also measured on the computer and compared with exact values. The analog with mass lumped at the cell division points and half cell termination was found to be the most accurate of the finite difference analogies. The Russell analog was found to provide considerably better accuracy in all cases except for the higher modes of the beam with large taper. It was found that the transformer parasitic parameters are the only significant factors affecting the accuracy of the computer for structures of this type. The effect of these can be calculated reliably and optimum computer constants chosen for each case. Thus, for complex problems requiring the representation of a large number of modes, the computer set up can be so designed as to ensure that the important modes are represented to the greatest accuracy or to about 1%. (Contractor's abstract, modified)

California Inst. of Tech., Pasadena.
N6ori-10503, Project Squid see under Princeton U. James Forrestal Research Center, N. J. (Project SQUID) item no. PRI. 11:023.

California Inst. of Tech. Guggenheim Aeronautical Lab., Pasadena. Report No. 2;
GALCIT Report No. 86. AFOSR-TN-55-237.
THE FLUTTER OF A BUCKLED PLATE IN A SUPERSONIC FLOW. Y. C. Fung. AF 18(600)1142. July 1955. ASTIA Document No. AD 70403.

The flutter of a thin elastic plate in a "2-dimensional" supersonic flow is considered. The edges of the plate are hinged to supports that are perfectly rigid and fixed in space. A sufficiently large compressive load is assumed to act in the plane of the plate, so that the plate would buckle if it were free from all tractions normal to the surface of the plate. The solution is based on Galerkin's method, and under the assumption of small reduced frequency and linearized aerodynamic theory. Nonlinearity in the problem is caused by the dependence of the compressive load in the plane of the plate on the lateral deflection. Both the linear, small amplitude flutter about equilibrium positions and the nonlinear, finite amplitude flutter are considered. Certain theoretical difficulties and unsolved problems are discussed. (Contractor's summary).

California Inst. of Tech. Guggenheim Aeronautical Lab., Pasadena. Report No. 4;
GALCIT Report No. 86. AFOSR-TN-56-296.
THE FLUTTER OF A TWO-DIMENSIONAL BUCKLED PLATE WITH CLAMPED EDGES IN A SUPERSONIC FLOW. J. G. Eisley. AF 18(600)1142. July 1956. ASTIA Document No. AD 90008.

The flutter of a 2-dimensional buckled panel with clamped edges is studied both theoretically and experimentally. In the first part, the flutter mode is described by a series expansion of functions which satisfy the boundary conditions for clamped edges. Quasi-steady linearized aerodynamics is used. Large deflections of the plate are considered. Numerical calculations have been made for only the first 2 terms of the series expansion. An experimental program designed to study the nature of panel flutter is described. The results of the experiments are used to check the validity of the assumptions used in the theoretical study and to shed some light on the process of panel flutter for use in future studies. (Contractor's abstract)

California Inst. of Tech. Guggenheim Aeronautical Lab., Pasadena. Report No. 1;
GALCIT Report No. 86. AFOSR-TN-55-236.
THE FLUTTER OF SIMPLY SUPPORTED RECTANGULAR PLATES IN A SUPERSONIC FLOW. J. G. Eisley. AF 18(600)1142. July 1955. ASTIA Document No. AD 70404.

The problem of flutter of a simply supported rectangular plate placed in a supersonic airstream is studied. Only small deflections of the plate are considered so that linear plate theory may be used. The flutter mode is described by a series expansion in terms of the normal modes of oscillation of the plate in a vacuum. Linearized aerodynamic theory is used. Additional simplification is introduced in 2 forms. In 1 case, the strip assumption is introduced, and in another the quasi-steady approximation is made. The exact solution is carried out, and the result is compared with the 2 approximate results. Numerical calculations were made to determine the flutter boundaries for plates of varying aspect ratio using strip theory aerodynamics. These calculations were made for $M=\sqrt{2}$ and $M=2$ over a range of values of the reduced frequency. For $M=2$ the flutter mode was described by considering 2 or 3 normal modes. For $M=\sqrt{2}$ only 2 normal modes were considered. (Contractor's summary)

*Bell Aircraft Corp., Buffalo, N. Y.
ADVANCED STUDIES IN FLUTTER, M. Zisfein, Project 9782(806), Contract AF 49(638)-749; AFOSR, DAS.

The intent of this program is to conduct advanced investigations of the dynamics of airframes including: (1) a theoretical study of the effects of damping on flutter and high Mach numbers. The purpose of this research is to obtain a better understanding of the significance of damping on the flutter stability of structural components at high supersonic and hypersonic speeds. Particular attention is directed toward evaluating the variety of approximate methods presently in use for the prediction of flutter and to study the phenomenon of velocity-damping "loopbacks"; and (2) study of the linear and non-linear vibration characteristics of a "jet-mass" on flutter speed. The term "jet-mass" as used here consists of applying an external force which is at all times proportional to the acceleration of the point of application of the force. The ultimate objective is to alter the flutter characteristics of an aerodynamic surface by "effective mass" changes to the systems.

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*Abstract of unclassified basic research project supported by the U. S. Air Force Office of Aerospace Research, Fiscal year 1960.

*Midwest Research Inst., Kansas City, M. EFFECT OF EXTERNAL FORCE EXCITATION ON PANEL FLUTTER, Y. L. Luke. Project 9782(806), Contract AF 49(638)-389; AFOSR, DAS.

This is a theoretical investigation of the effect of external-force excitation on the flutter of flat panels. The equations of motion for a flat panel in both two- and three-dimensional supersonic flow will be studied, taking into account external forces due to sound presures and frame transmitted vibrations.

*California Inst. of Tech., Pasadena. THEORETICAL AND EXPERIMENTAL STUDIES CONCERNING PANEL FLUTTER, E. E. Sechler. Project 9782(806), Contract AF 49(638)-220; AFOSR, DAS.

Theoretical and e perimental studies will be conducted in certain areas of panel flutter, namely, the flutter of buckled plates of finite aspect ratio, the limiting amplitudes of flutter, flutter at transonic speeds, the effect of thermal stresses on flutter, and flutter of a membrane. One of the first projects of the experimental program will be to develop a technique of observing the onset of panel flutter. A variable frequency excitor will be developed to measure the response of the panel at increasing and decreasing excitation frequencies. An approximate aerodynamic theory will be developed for the two-dimensional supersonic flow problem to bridge the gap between the quasi-steady aerodynamic theory and the full linearized theory. The investigations will be extended to take into account the case of flat finite aspect ratio rectangular plates in a supersonic flow. It is felt that simplified aerodynamic theories can be developed in this area for particular cases and employed with success in prototype flutter analyses.

Joining

TR 4554.
TESTS OF SCIAKY PMCO. 2S SPOT WELDER. R. E. Bowman and R. W. Ego. July 1940.

TR 4659.
SHUNT FOR THE MEASUREMENT OF WELDING CURRENT IN SPOT WELDERS. R. E. Bowman and R. W. Ego. July 1941.

TR 4657.
TESTS OF TAYLOR-WINFIELD HI-WAVE SPOTWELDER. R. E. Bowman and R. W. Ego. July 1941.

TR 5310.
FLASH WELDED CENTRIFUGAL CASTINGS FOR AIRCRAFT. Edward L. Horne, Ture T. Oberg, and L. W. Katz, 1/Lt. October 1945.

TR 5749.
TESTS AND EXAMINATION OF GAS PRESSURE WELDS. Robert E. Bowman, Walter Trapp, and C. B. Hartley. January 1949.

TR 5931.
DETERMINATION OF IMPURITIES IN INERT GASES. R. E. Bowman and C. B. Hartley. September 1949.

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*Abstract of unclassified basic research project supported by the U.S. Air Force Office of Aerospace Research, Fiscal year 1960.

Battelle Memorial Institute. TR 52-313, Part I. BRAZING TITANIUM TO TITANIUM AND TO MILD AND STAINLESS STEELS. W. J. Lewis, P. S. Rieppel, and C. B. Voldrich. AF 33(038)-23338. November 1952. PB 111244. Order from OTS $1.00

Procedures and alloys suitable for brazing titanium were investigated. Commercial brazing alloys were evaluated by making brazed joints of titanium in a furnace containing an atmosphere of high purity argon. The most satisfactory alloys in this type of brazing were silver and silver base alloys. Joints with shear strengths averaging 15,000 psi were obtained by furnace brazing with the following alloys:

100% silver
85% silver, 15% maganese
45% silver, 15% copper, 16% zinc, 24% cadmium

Brazed joints of titanium were also made with the oxyactylene torch and a commercial brazing flux. The best alloy found in torch brazing was a 45% silver, 15% copper, 16% zinc, 24% cadmium alloy, which produced shear strengths averaging 13,000 psi.

The strengths of brazed joints in titanium were somewhat lower than that of similar brazed joints of the same alloys in carbon steels. The lower strengths are believed to be associated with the inter-metallic compounds which formed at the boundaries between the brazing alloy and titanium. Also, broad zones of diffusion were present at some of the boundaries. Some of the inter-metallic compounds appeared to be brittle. Silver and the 85% silver-15% manganese alloy were the only brazing alloys that produced joints exhibiting some ductility.

In order to reduce compound formation and diffusion, a few preliminary tests were made using shorter brazing cycles. This was accomplished with induction, resistance, and shielded carbon-arc brazing methods. These tests indicated that the formation of inter-metallic compounds can be reduced by using shorter heating cycles. This phase of the investigation will be covered in more detail in a second report.

Battelle Memorial Institute. TR 52-322, Part I. CAUSES OF CRACKING IN HIGH-STRENGTH WELD METALS. A. J. Williams, A. J. Jacobs, P. J. Rieppel and C. B. Voldrich. AF 33(038)-12619. November 1952. PB 11153. Order from OTS $1.25

In this investigation, the major part of the effort was devoted to making and testing a special apparatus for determining the hot ductility of weld metals. The apparatus was designed so that the test specimen could be tested in tension after the center section had been cooled directly from the molten state to a predetermined temperature. The center section was melted by induction heating and was retained in place by a mold of fused silica. Special equipment was designed and constructed to measure the load required to fracture the specimen and to measure the elongation. Techniques were developed to measure the temperature at the center section. The operation of the apparatus was checked by testing SAE 1018 and SAE 4340 steel specimens in the temperature range from 2588° to 1800°F after the center section was cooled directly from the melting temperature. The equipment was satisfactory and will be used in future tests to determine the effects of weld-metal composition on hot strength and hot ductility. Seven special heats of SAE 43XX steels were made with different sulfur and carbon contents. These steels will be included in future tests to determine the effects of carbon and sulfur on hot strength and hot ductility.

Some studies were made with weld-metal cracking test specimens to develop a specimen that could be used in conjunction with the hot-ductility studies. Techniques were also developed for using the electron microscope in the study of grain boundaries of weld metals.

Battelle Memorial Institute. TR 52-294.
EFFECTS OF CARBON, OXYGEN, AND NITROGEN ON THE PROPERTIES OF WELDS IN TITANIUM SHEET. D. C. Martin and C. B. Voldrich. AF 33(038)-21385. November 1952. ASTIA Document No. AD 4526.

Three series of titanium alloys were melted and rolled into sheet. The first series included four titanium-carbon alloys with the carbon ranging from 0.13 per cent to 0.74 per cent. The second series contained three titanium-oxygen alloys with the oxygen ranging from 0.15 per cent to 0.55 per cent. The third series had two titanium-nitrogen alloys, one containing 0.13 per cent nitrogen, the other, 0.24 per cent nitrogen. A 0.50 per cent nitrogen alloy was melted but could not be rolled into sheet. Inert-gas-shield arc welds were made in one-sixteenth inch and one-eighth inch sheets of each alloy. Spot welded specimens were made with 0.032 inch and 0.064 inch sheets of each alloy. The physical properties of both arc welds and spot welds in each alloy were determined. The data that was obtained will be useful in establishing the allowable percentages of carbon, oxygen and nitrogen impurities in titanium sheet.

TR 53-67.
DETERMINATION OF HYDROGEN AND OXYGEN IN INERT GASES. Robert E. Bowman, Major and Charles B. Hartley. May 1953. PB 116838. Order from LC Mi $2.25, Ph $4.00.

This report describes tests of a commercial apparatus which was used to determine the amount of oxygen and hydrogen impurities in inert gases. The determination consists of measuring the temperature rise which occurs when oxygen and hydrogen combine in the presence of oxygen (or hydrogen) in the inert gas. The operation of the apparatus was checked by measuring the oxygen and hydrogen impurities in a cylinder of welding grade argon. Oxygen and hydrogen impurities as low as 0.001% could be detected.

Battelle Memorial Institute. TR 52-313, Part 2.
BRAZING TITANIUM TO TITANIUM AND TO MILD AND STAINLESS STEEL. W. J. Lewis, G. E. Faulkner, P. J. Rieppel and C. B. Voldrich. AF 33(038)-23338. December 1953.

The brazing characteristics of titanium when joined to titanium, mild steel, and stainless steel were studied. Joints between these materials were induction brazed, torch brazed, and furnace brazed with commercial and experimental brazing alloys.

Induction-brazed joints produced higher strengths than furnace- and torch-brazed joints. Joints in titanium were more easily brazed by all methods and using all alloys than those between titanium and mild and stainless steels.

High-strength joints in titanium were obtained using titanium-base experimental brazing alloys, but ductility was poor.

Armour Research Foundation Illinois Institute of Technology. TR 53-231.
RECRYSTALLIZATION WELDING OF ALUMINUM AIRCRAFT ALLOYS. Nickolas A. DeCecco and John M. Parks. AF 18(600)-92. December 1953.

Recrystallization is established as the mechanism for solid phase welding. 2S, 24S, 52S and 61S aluminum alloys were welded by pressing together two cleaned and cold worked surfaces and heating to the recrystallization temperature. The shear strengths of the welded joints are equivalent to that of the annealed metal.

The fundamental forces in operation when making a recrystallization welded joint have been investigated and analysed. The basic principles and techniques described are applicable to metals other than aluminum and aluminum base alloys.

Battelle Memorial Institute. TR 53-401.
PRODUCTION OF SOUND DUCTILE JOINTS IN MOLYBDENUM. M. I. Jacobson, D. C. Martin and C. B. Voldrich. AF 33(616)-10. January 1954. PB 123916. Order from LC Mi $4.50, Ph $13.80.

Methods of welding and brazing molybdenum with the objective of producing sound ductile joints were investigated by Battelle Memorial Institute for the Wright Air Development Center during the period 25 March 1952 to 25 September 1953. The results of tungsten-arc welding tests showed that, although ductile weld metal could be produced, the welded joints were brittle transverse to the direction of welding because of recrystallization in the base metal. Several brazing methods were investigated, with induction brazing in an argon atmosphere appearing to be the most satisfactory from the standpoint of braze quality and practicability. Brazed joints were tested at room temperature and at 1800°F. The brazing alloys that gave the best results at 1800°F were Inconel and Haynes Alloy 25, which produced joints with 100-hour shear strengths of 5000 psi and 4500 psi, respectively.

Battelle Memorial Institute. TR 52-322, Part 2.
CAUSES OF CRACKING IN HIGH-STRENGTH WELD METALS. A. J. Jacobs, P. J. Rieppel and C. B. Voldrich. AF 33(038)-12619. February 1954. PB 111532. Order from OTS $1.25.

This report discusses an experimental investigation of the causes of cracking in high-strength weld metals. The research was conducted at Battelle during the third contract period, from August 12, 1952, to August 12, 1953. Specifically, the work included hot-ductility and weld-metal cracking tests on special high-strength steels and a metallographic examination of grain boundaries and inclusions in two of the steels. The results, although in an early stage, indicate that low-sulfur contents are associated with high hot ductility and high resistance to hot cracking. They also suggest a possible relationship between low-melting intergranular films and low hot ductility.

Battelle Memorial Institute. WADC TR 52-322, (Part 3).
CAUSES OF CRACKING IN HIGH-STRENGTH WELD METALS. A. J. Jacobs, R. P. Sopher, P. J. Rieppel. AF 33(038)12619. August 1954. ASTIA Document No. AD 65474. PB 145332. Order from LC Mi $3.00, Ph $6.30.

This report summarizes the third year of experimental work at Battelle on the causes of cracking in high-strength weld metals. During the period from August 12, 1953, to August 12, 1954, hot-tension and weld-metal cracking studies were conducted on SAE 43XX-type steels and other selected steels. Results from these studies showed a correlation, inasmuch as an increase in carbon, sulfur, and phosphorus tended to lower hot ductility and promote hot-crack susceptibility, and a misch metal addition seemed to have the opposite effects.

Westinghouse Electric Corporation. WADC TR 54-17.
JOINING OF MOLYBDENUM. W. N. Platte.

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AF 18(600)114. August 1954. ASTIA Document No. AD 29379. PB 128527. Order from LC Mi $5.70, Ph $16.80.

By the use of a closed chamber and a continuous flow of gas through this chamber a controllable welding atmosphere has been provided for experimental work. The atmosphere may be varied from 100% argon, 99.95% pure, to 100% O_2, N_2 or any combination of these or other gases. The chamber provides a fresh gas shield in an atmosphere of the same composition as the shield.

Using the controlled atmosphere chamber, welds in carbon deoxidised arc cast molybdenum are shown to be subject to hot short cracking when the oxygen content in the argon atmosphere around the arc exceeds 0.2%. The ductility of these welds was drastically reduced by the presence of more than 0.05% oxygen in the welding atmosphere.

Oxygen in sintered molybdenum is shown to produce porosity and hot short cracking. However, crack and porosity free welds have been produced by using deoxidizing agents in sintered molybdenum.

Rensselaer Polytechnic Institute. WADC TR 54-97. STUDIES AND EXPERIMENTAL INVESTIGATIONS ON THE FLASH WELDING OF ALLOYED STEELS. Ernest F. Nippes, Warren F. Savage, Gordon Brotke, Salvatore M. Robelotto. AF 18(600)-71. September 1954.

Weld centerline rates of cooling at 1300, 1000, and 900°F were determined as functions of three welding variables, the rate of platen acceleration, the final clamping distance, and the thickness of the material, for flash welds employing parabolic flashing patterns. Three rates of platen acceleration, 0.120, 0.042, and 0.0166 in./sec.^{2}, were investigated, with a range of final clamping distances of 0.3 to 1.2 in., for rectangular bar material of 0.250 and 0.375 in. thickness.

Flash welding variables within these limits resulted in weld centerline cooling rates, at 1300°F, ranging from a minimum of 40°F/sec. to a maximum of 365°F/sec. Increasing platen acceleration, increasing section thickness and decreasing final clamping distance resulted in an increased rate of cooling. In both alloy steels studied, AISI 4130 and 4340, the formation of considerable amounts of martensite at the weldline could not be avoided by any reasonable adjustment of flash welding variables studied.

Flash welds were prepared using AISI 1020, 4130, and 4340 steels at four predetermined rates of cooling. Photomicrographs and the results of hardness and tensile tests are presented and discussed to relate the weld properties and the weld-zone micro-structures.

Handy and Harman. WADC TR 55-22. OXIDATION RESISTANT BRAZING ALLOYS. George H. Sistare, Jr., Allen McDonald. AF 33(616)-2205. April 1955. ASTIA Document No. AD 75471. PB 130403. Order from LC Mi $3.30, Ph $7.80.

During the period from 30 June 1953 to 31 December 1954 some 117 experimental brazing alloys were prepared at the Handy and Harman research laboratories, and evaluated as possible candidates for joining heat conducting metal fins to inconel tubing for service at temperatures in the order of 1400°F to 1600°F where oxidation resistance of the joint was mandatory.

Alloys of gold-nickel-chromium, and palladium-nickel base alloys with chromium and silicon were developed which can be used to join stainless steel to inconel at brazing temperatures in the range of 1900°F to 1950°F. The alloys can be torch brazed using flux, or used without flux in a protective atmosphere. The resulting joints resist oxidation at 1600°F service temperature.

It is still possible that with further development palladium-nickel base alloys with silicon-phosphorus-boron additions can be produced which will braze in the temperature range from 1850°F to 1900°F.

The report also contains a note on graphite brazing.

Rensselaer Polytechnic Inst. WADC TR 54-97, (Part 2). STUDIES AND EXPERIMENTAL INVESTIGATIONS ON THE FLASH WELDING OF ALLOYED STEELS. Ernest F. Nippes, Warren F. Savage, Gordon Grotke, Salvatore M. Robelotto. AF 18(600)-71. April 1955.

Weld centerline rates of cooling at 1300, 1000 and 900°F in flash welds in AISI 1020 and 4340 steels, prepared under similar conditions, were measured and compared. The cooling rate is not influenced by the composition of the steel if the specimen geometry, platen acceleration, and final clamping distance are maintained constant.

Weld zone cooling rates were determined for welds in AISI 1020 steel subjected to 1.5 sec. of upset current at an average current density of 18,500 amp/in.^{2}. The reduction in cooling rate produced by this upset current flow was obtained by comparision with previous data on welds without post-upset current.

The temperature distribution in the vicinity of the weld zone was measured experimentally during the application of 4.75 sec. of temper current at an average density of 21,400 amp/in.^{2}.

Three steels of differing elevated-temperature strength were employed for welds prepared at four rates of platen acceleration to determine the influence of the composition of the steel and the influence of the temperature gradient, established during flashing, on the upset force requirements.

Wall Colmonoy Corporation Research Laboratory. WADC TR 55-213. DEVELOPMENT OF BRAZING ALLOYS FOR JOINING HEAT RESISTANT ALLOYS. Forbes M. Miller, Homer S. Gonser, Robert L. Peaslee. AF 33(616)-2287. July 1955. PB 121001. Order from OTS $2.00.

Fifteen different alloy systems were tested and studied for brazing characteristics, chemical and physical properties. These alloys were nickel base binary and ternary systems containing such metals and metalloids as phosphorus, silicon, chromium, maganese, molybdenum, tungsten, and iron.

The nickel-phosphorus-chromium system and nickel-phosphorus-silicon system showed the best properties of the phosphorus bearing alloys. The nickel-silicon-manganese system showed the best properties of the silicon bearing alloys. Of the alloys studied the silicon bearing alloys in general are higher melting than the phosphorus bearing alloys.

Studies also showed that phosphorus and manganese contributed the most toward improving the flow and wetting properties of the alloys, while silicon and chromium contributed the most toward improving the oxidation resistance and strength properties of the alloys.

Battelle Memorial Institute. WADC TR 52-322, Part 4. CAUSES OF CRACKING IN HIGH-STRENGTH WELD METALS. A. L. Lowe, Jr., R. P. Sopher, P. J. Rieppel. AF 33(616)-2734. November 1955. PB 124150. Order from LC Mi $3.00, Ph $6.30.

This report summarizes the experimental work conducted at Battelle in a study to determine the causes of cracking in high-strength weld metals. A new hot-tension

machine was designed and built to facilitate the testing of SAE 4340 weld metals on cooling from the molten state. The tests were conducted over the temperature range from 2600°F to 100°F. Results from the studies showed phosphorus to be detrimental to weld-metal cracking resistance. As the phosphorus was increased the ductility was lowered at temperatures near the solidus. Nitrogen content within the normal range of SAE 4340 steels appeared to have little influence on the cracking resistance of the weld deposits. Rare-earth metal additions improved the hot ductility and hot-cracking resistance of the weld metals. An increase in silicon lowered the hot ductility and hot-cracking resistance slightly.

Westinghouse Research Laboratories. WADC TR 54-17, Part 2.
JOINING OF MOLYBDENUM. W. N. Platte. AF 18(600)-114. November 1955. PB 111833. Order from OTS $3.25.

A study of the factors which influence the physical properties, especially the ductility, of molybdenum welds is discussed in this report. Welds in both arc-cast and vacuum sintered molybdenum made in commercially pure inert atmospheres and in contaminated inert atmospheres are examined.

The effects of several deoxidizers on the welding properties of vacuum sintered molybdenum are examined. The specific requirements of deoxidizing agents are established and it is shown that titanium between 0.2 and 0.5% meets these requirements.

The effects of interstitial elements, oxygen, nitrogen and carbon, on the weld properties of arc-cast molybdenum have been examined. It is shown that the limits for oxygen in the inert gas welding atmosphere can be predicted. The deleterious effects of nitrogen and oxygen are shown quantitatively by means of bend test data. Both oxygen and nitrogen reduce the ductility of molybdenum weld metal. Carbon is also believed to have a similar effect.

Oxygen and nitrogen are shown to have an interaction effect when used in combination in the inert welding atmosphere. Nitrogen reduces the deleterious effects of oxygen and vice versa, but the combination is not as effective as high purity inert gas as a welding atmosphere.

Exploratory studies are described for slightly improved inert gas atmospheres and post-weld heat treatment. But welds were made to establish the validity of using "bead on plate welds" as a test criterion.

Westinghouse Research Laboratories. WADC TR 54-17, Part 3.
JOINING OF MOLYBDENUM. William N. Platte. AF 18(600)-114. November 1956. ASTIA Document No. AD 110570. PB 121848.

The use of welding atmospheres of purified argon and helium is shown to cause very little contamination of the weld metal in molybdenum fusion welds. However, the use of high purity atmospheres provides only a small improvement in ductility over welds made in welding grade argon.

Increasing the carbon content of the base material within the range 0.04% to 0.06% appears to improve the ductility of the weld metal. Additions of 0.5% titanium in the presence of 0.05% carbon gave welds which could be deflected 120° at 80°F. Increasing the carbon content from 0.015 to 0.05% improved the ductility of the molybdenum welds containing 0.5% titanium. Additions of 0.16% aluminum to the molybdenum yielded welds which could be deflected 120° at 120°F.

Exploratory studies on the effectiveness of diffusion as a method of removal of nitrogen from the weld metal are discussed. The use of thermal treatment as a method of improving mechanical properties by over-aging and polygonization was explored. Weld metal additions are also discussed briefly. The use of Ti and Zr as deoxidizers in vacuum sintered molybdenum is described.

An exploration into the effects of residual welding stresses showed that the weld crater cracking encountered in many cases is caused by measurable stresses in the region of the weld which approximate the rupture strength in material.

Battelle Memorial Institute. WADC TR 56-550.
DEVELOPMENT OF HIGH-STRENGTH FILLER WIRES FOR WELDING SAE 4130, 4140, AND 4340 STEELS. Herbert W. Mishler, Raeman P. Sopher. AF 33(616)-2339. April 1957. ASTIA Document No. AD 118210. PB 131074. Order from OTS $2.00.

Filler wires were developed for welding high-strength low-alloy aircraft steels by the inert-gas tungsten-arc and inert-gas consumable-electrode processes.

The major factors that were considered in the evaluation and development work were: (1) weld-metal porosity, (2) weld-metal cracking tendency, (3) heat-treatment response compared with base metal, (4) strength of heat-treated weld joint, (5) weld-metal toughness, and (6) weld-metal hardenability. Two filler wires were developed for welding each of the three steels, SAE 4130, 4140, and 4340. One of each pair of filler wires had a composition similar to the base material, while the other was of a dissimilar composition. Sound, crack-free welds, heat treatable to 200,000 psi were deposited under a high degree of restraint with all these filler wires. The best results were obtained with the filler wires of compositions similar to the base material, although satisfactory welds were made also at most of the strength levels with the dissimilar filler wires.

Battelle Memorial Institute. WADC TR 58-479.
RESEARCH AND DEVELOPMENT OF PROCEDURES FOR JOINING OF SIMILAR AND DISSIMILAR HEAT-RESISTING ALLOYS BY ULTRASONIC WELDING. Norman E. Weare, John N. Antonevich, Robert E. Monroe, David C. Martin. AF 33(616)-5342. February 1959. ASTIA Document No. AD 208323. PB 145015.

An investigation was conducted to apply ultrasonic welding to joining similar and dissimilar heat-resisting alloys and to study the fundamentals of this process.

Ultrasonic spot welds were made in various combinations of heat-resisting alloys. Room temperature tension-shear and cross-tension strengths were obtained for each combination. Metallographic examinations of the heat-resistant weldments showed in presence of cracks at the edges of many of the spot welds. This cracking is probably caused by high cyclic stresses at the edge of the weld producing fatigue failures in this area. Methods of preventing such cracking can probably be determined.

Efforts in the fundamental studies were aimed at studying the mechanism of bonding by ultrasonic welding. Effects of times, temperatures, and forces involved or closely related to bonding were determined although the exact mechanism was not established. Also determined were the effects of factors associated with the process and materials involved.

Sylvania-Corning Nuclear Corp. WADC TR 58-674.
THE CLADDING AND WELDING OF STAINLESS STEEL TO MOLYBDENUM AND NIOBIUM. Joseph Fugardi, John L. Zambrow. AF 33(616)-3492. October 1959.

Methods were investigated for the fabrication of composite sheets consisting of 310 stainless steel bonded

to one side of either molybdenum or niobium sheet. A barrier material between the two sheets was used to minimize diffusion. The best barrier material found, on the basis of tensile strength, thermal shock resistance, minimum diffusion during long time high termperature tests and minimum formation of intermetallic compounds at the bonding surfaces, was nickel for the molybdenum to Type 310 stainless steel composite and iron for the niobium to Type 310 stainless steel composite. Butt welds were made on composite sheets of stainless-molybdenum and stainless-niobium. The difference in the thermal coefficient of expansion between the stainless and the refractory metals used caused stresses and distortion in the weldments.

Armour Research Institute. WADC TR 59-404. DEVELOPMENT OF PARTIALLY VOLATILE BRAZING FILLER ALLOYS FOR HIGH-TEMPERATURE APPLICATION AND RESISTANCE OXIDATION. W. M. Lehrer, H. Schwartzbart. AF 33(616)-5654. December 1959. ASTIA Document No. AD 232 969. PB 161 716. Order from OTS $1.25.

Experimental brazing filler alloys have been developed containing temperature depressants which have been volatilized during the brazing of stainless steel leaving joints of high remelt temperature. The mechanisms by which remalt temperature is increased have been studied for the range of alloys investigated which contained nickel, chronium, germanium, iron, lithium, and phosphorus. The two main mechanisms are (i) volatilization of the filler metal constituents.

It has been shown theoretically and experimentally that in order for remelt temperature to increase there must be solid solubility of the diffusing or volatile element in the filler alloy. Although compositional changes may result from either mechanism, a remelt temperature change does not necessarily occur. The degree to which a compositional change affects remelt temperature is proportional to the solid solubility.

In this program, vacuum pumping was the more efficient vapor-removal method among several discussed and investigated. Furthermore, under vacuum the surface-area-to-volume ratio of the filler becomes less rate-controlling due to boiling. Boiling, however, causes porosity in the joint.

Of the alloys investigated a 61% Ni-39% In alloy exhibited the greatest remelt temperature rise due to volatilization alone, whereas a 94% Ni-6% P responded only to dissolution and diffusion to exhibit the largest rise in remelt temperature due to this mechanism. Ni-Cr-In-Ge alloys exhibited a substantial rise in remelt temperature due to the operation of both mechanisms. This alloy and the binary Ni-In alloy can be considered practical alloys for high-temperature usage.

A direct effect of metallic vapors on wetting and flow of the filler alloy was observed and subsequently discussed with respect to their influence on surface tension relationships. It has been shown that in the presence of metal vapors, discoloration of the base metal, wetting, flow, and skull formation of the filler alloy are functions of the rate and quantity of metal vapor removal.

Avco Corporation. WADC TR 59-695, Part I. BERYLLIUM JOINING RAD SPONSORED PROGRAM. J. B. Cohen. AF 33(616)-5913. April 1960. PB 161 830. Order from OTS $1.25.

New brazing techniques are described for joining beryllium to itself. A Be-20 a/o Ag brazing alloy developed under this program is shown to yield joint strengths at room temperature of 60 percent (30,000 psi) of that of the base metal. In the temperature range of 700 to 1450°F, the joint strength is 80 percent that of the base metal. Twenty-four hour exposures at these temperatures did not affect the room temperature strength.

Similar strengths are achieved by brazing with pure silver, if a continuous interface of silver in the joint is absent. This is readily accomplished by heat treatment because of the rapid intergranular penetration of silver into beryllium. The resulting thick joint is not deleterious, since it is shown that a two phase silver beryllium alloy with a silver network is ductile. As in the above case, long time exposures (24 hrs) at temperatures above 1300°F did not affect the room temperature joint strength.

Spreading of liquid silver on beryllium is not appreciable, probably because of its rapid intergranular penetration. Because of this and the toxicity of beryllium fumes given off during a high-temperature braze, the silver should be preplaced. This can be accomplished by electroplating; thin layers can be plated, reducing the heat treating time to eliminate the interface.

Evidence is presented which suggests that the gamma phase in the silver-beryllium system may extend to a temperature of 1350°F, 50°F lower than has been tentatively assigned as its lower limit of stability in the published phase diagram.

Battelle Memorial Institute. WADC TR 59-531. DETERMINATION OF THE CAUSES OF WELD-METAL CRACKING IN HIGH-STRENGTH STEELS AND THE DEVELOPMENT OF HEAT-TREATABLE LOW-ALLOY-STEEL FILLER WIRES FOR USE WITH THE INERT-GAS-SHIELDED ARC-WELDING PROCESS. Herbert W. Mishler, Robert E. Monroe, Perry J. Rieppel. AF 33 (616)-5878. May 1960. PB 161 981. Order from OTS $1.50.

The work conducted under this contract was divided into four phases: three dealt with the study of the causes of weld-metal cracking in SAE 4340 steel and the fourth was concerned with the study of filler wires for producing welds heat treatable to ultimate-tensile-strength levels greater than 225,000 psi.

Freezing-cycle hot-tension studies were made on various heats of SAE 4340 steel containing various sulfur and phosphorus contents. Results indicated that the combined sulfur and phosphorus contents of SAE 4340 steel should be kept below 0.025 percent to prevent weld-metal hot cracking. An intergranular phase believed to be associated with hot cracking was found during light and electron microscopy of high-phosphorus SAE 4340 steels. This phase was tentatively identified as the iron-iron phosphide eutectic. Two new restrained weld-metal-cracking tests were developed that will give a quantitative measure of weld-metal-cracking resistance and which also will be inexpensive to conduct. Filler wires were developed which can produce weld metals capable of being heat treated to various strength levels in the range of 225,000 to 280,000 psi ultimate tensile strength.

Battelle Memorial Institute. WADD TR 60-607. FUNDAMENTAL STUDIES ON THE MECHANISM OF ULTRASONIC WELDING. W. J. Lewis, J. N. Antonevich, R. E. Monroe, P. J. Rieppel. AF 33(616)-6268. December 1960. ASTIA Document No. AD 257733. Order from OTS $1.50.

The fundamental mechanisms of ultrasonic welding and the application of this process to the joining of various heat-resistant materials were investigated.

Previous fundamental studies which had shown the effects of time, temperature, and clamping force on ultrasonic welds were expanded to include studies of the shear force during welding. The results of these studies exhibited considerable scatter, but several trends appeared

to be indicated.

Ultrasonic spot welds made in various combinations alloys generally confirmed the results of previous work with these alloys. Cracks were found at the edges of the spot welds in most of the material combinations studied. The effects of these cracks on weldment properties varied with the material. The presence of a reaction zone, apparently consisting of intermetallic compounds, was apparent in ultrasonic welds made between titanium and stainless steel. These findings indicate that ultrasonic welding is not suitable for the production of aircraft-quality spot welds in the heat-resistant and dissimilar metal combinations included in this program.

General limitations on the use of this process in its current state of development can be established from the work conducted. It is apparent as a material's hardness increases, and the ratio of tensile strength to yield strength decreases, that weldability by the ultrasonic process decreases.

Mechanical Evaluation

Southern Research Institute. WADC TR 57-649, Part II.
DETERMINATION OF THE MECHANICAL PROPERTIES OF AIRCRAFT-STRUCTURAL MATERIALS AT VERY HIGH TEMPERATURES AFTER RAPID HEATING. James B. Preston, J. Robert Kattus. AF 33(616)-3494. April 1960. PB 161 893. Order from OTS $2.25.

Structural components in high-speed aircraft and in missiles must function for short periods of time at high temperatures and at high stresses, and frequently the heating and loading occur simultaneously. The requirements for reliability without overdesign demands accurate test data obtained under conditions approximating the expected operating conditions. In an effort to fulfill a portion of this need, this program was divided into four independent areas of work as follows: (1) The tensile properties of unalloyed beryllium were determined at test temperatures from ambient through 1500°F. (2) The short-time, elevated-temperature tensile properties were determined for ten combinations of base materials and coating materials (Cr-Ni electroplate on copper sheet, Rokide A on copper sheet, Rokide Z on copper sheet, Rokide ZS on copper sheet, Cr electroplate on A-nickel sheet, Ni-Cr electroplate on molybdenum sheet, Rokide Z on molded graphite, Rokide ZS on molded graphite, Crystalon C on molded graphite and SiC-SiN on molded graphite). (3) The effects of linear thermal gradients up to 1500°F/in on the tensile properties of a typical refractory alloy were investigated. (4) The effects of simultaneous heating and loading on the tensile properties of a typical structural alloy were investigated. This report covers the first three of these four areas of work; a supplementary report will be issued shortly containing all of the results of the investigations involving simultaneously transient temperature and load.

Midwest Research Institute. WADD TR 60-313. RESEARCH ON THE MECHANISMS OF FATIGUE. J. C. Grosskreutz. AF 33(616)-6383. April 1960. ASTIA Document No. AD 247129. PB 171 285. Order from OTS $1.50.

Based on a critical appraisal of existing fatigue theory and experimental data, significant research areas have been outlined for the study of fatigue mechanisms. Polycrystalline aluminum and copper and single crystals of aluminum have been used to investigate dislocation densities and configuration during crack initiation and the mechanism by which cracks propagate, both on the surface of a sample and internally. Back reflection X-ray patterns show that an observable increase in dislocation density occurs within the first 0.1 percent of fatigue life and that a saturation density is reached after only 1 percent of the life. These dislocations accumulate into stable subgrain boundaries provided either that the stressing is done under a tensile preload or that the amplitude of symmetrical stressing is large enough. For small symmetrical loading (strains $\gtrsim$ 0.001 for Al) a random dislocation array results with no subgrain formation. These results can be correlated with fatigue hardening behavior and possibly with the shape of the S-N curve. There is no obvious relation between the bulk behavior of fatigue induced dislocations and the formation of the initial fatigue crack which is a highly localized phenomenon.

Continuous observation of the surface of fatiguing samples utilizing strobe-microphotography has confirmed the accumulating evidence that cracks initiate in persistent slip bands. Tapered sections have shown that these slip bands correspond to deep notches or fissures. The propagation of the crack is observed to occur along well defined slip systems with the crack jumping from one parallel slip band to another by cross slip. Transverse sections cut through the crack have shown that the crack propagates into the volume of the metal from the surface, again via slip planes. The crack is observed to propagate generally perpendicular to the stress axis; with the available slip system, this usually results in a zigzag path. A "cloud" of a slip is observed to precede the crack tip and from this fact and the foregoing data a mechanism of crack propagation is postulated; i.e., crack propagation is a series of reinitiations in persistent slip bands.

Armour Research Foundation. WADD TR 60-191. THE DETERMINATION OF THE EFFECTS OF ELEVATED TEMPERATURES ON THE STRESS CORROSION BEHAVIOR OF STRUCTURAL MATERIALS. Frank A. Crossley, Carl J. Reichel, Charles R. Simcoe. AF 33 (616)-6392. May 1960. PB 161 962. Order from OTS $1.50.

Certain aspects of elevated-temperature chloride salt stress-corrosion of Ti-6Al-4V alloy have been investigated. Experiments were conducted to determine whether the protection of the oxide film is lost by direct chemical reaction, or by dissolution, i.e., oxygen diffusion into the base metal. The dissolution rates of anodized oxide films over the temperature range from 800° to 1000°F were determined. Coefficients for oxygen diffusion in Ti-6Al-4V alloy are given by:

$D, cm^2/sec: 2.87 \times 10^{4} \exp(-67,900 \pm 300/RT)$.

Experiments showed conclusively that film protection is lost by chemical reaction of the chloride salt with TiO_2.

Elevated-temperature creep testing in the presence of chloride salt was found to be detrimental to post-exposure tensile properties of the following materials: 17-7 PH, stainless steel Type 321, Inconel-X, and B120-VCA titanium alloy. Materials which, apparently, were not harmed by such exposure were: 2024T-86 aluminum alloy, Nickel-plated 17-22 AS low alloy steel, Lapelloy 13% chromium steel, and ZK-60 magnesium alloy.

Materials exhibiting premature failure with greatly reduced ductility in tension static fatigue tests at room temperature in the presence of 10% chloride salt aqueous solution were: 2024T-86, ZK-60, 17-7 PH, and B120-VCA.

The presence of JP-4 fuel in tension static fatigue tests at room temperature was not indicated to be detrimental to 2024T-86, Nickel-plated 17-22 AS, 17-7 PH, Lapelloy, Type 321, or B120-VCA. There was some

indication that the presence of fuel residue in elevated-temperature creep tests may be slightly detrimental to 2024T-86 alloy.

University of California. WADD TR 60-326. THE EFFECT OF DECREASES IN STRESS ON THE CREEP BEHAVIOR OF POLYCRYSTALLINE ALUMINUM IN THE DISLOCATION CLIMB REGION. L. Raymond, W. Ludemann, N. Jaffe, J. E. Dorn. AF 33(616)-3860. May 1960. ASTIA Document No. AD 245 309. PB 157 931. Order from OTS $1.60.

The effect of stress on the creep rate in the dislocation climb region was isolated from the effect of substructural changes by determining the effect of decreasing the stress at fixed creep strains on the subsequent course of creep. Two substructures, one produced by precreeping at 1300 psi to a strain early in the primary stage and another produced by precreeping to the secondary stage at the same stress, were studied. It was found that the effect of stress on the creep rate was a function of the substructure; the creep rate increases less rapidly with stress for the substructure present at the secondary creep stage which contains more closely spaced barriers to dislocation motion. Whereas the barriers to slip are introduced during the primary stage of creep in the dislocation climb region, these barriers are thermally recoverable. The secondary stage of creep is reached when the rate of introduction of new barriers equals the rate of removal of existing barriers as a result of recovery. Due to recovery of barriers the same secondary creep rate is eventually obtained for a given final stress regardless of the preceding creep history.

WADD TR 60-245. ELEVATED TEMPERATURE DYNAMIC MODULI OF VANADIUM TITANIUM AND V-TI ALLOYS. W. H. Hill, B. A. Wilcox. May 1960. PB 171014. Order from OTS $0.50.

The dynamic moduli of calcium-reduced vanadium, high grade aluminothermic vanadium, iodide titanium and Ti-75A alloy were determined over the temperature range R. T. to 1250°F. In addition, elevated temperature dynamic modulus data were obtained for five V-Ti alloys, having nominal compositions of V-8Ti, V-17Ti, V-25Ti, V-32Ti, and V-48Ti. The decrease in modulus of vanadium with increasing temperature was found to be relatively small as compared to iodide Ti and Ti-75A. Solid solution additions of Ti were observed to lower the modulus of V over the entire temperature range studied, but the additions did not affect the slope of the modulus versus temperature curve.

An empirical correlation relating modulus to density, absolute melting point, and atomic weight was successfully applied to vanadium and V-Ti alloys, such that good agreement was realized between the ratios of E_v/E alloy (calculated) and E_v/E alloy (measured).

General Electric Company. WADC TR 59-511. GAS ATMOSPHERE EFFECTS ON MATERIALS. R. A. Baughman. AF 33(616)-5667. May 1960. PB 161 980. Order from OTS $3.50.

A three phase program was conducted to determine the effect of a special gas containing 5% H_2-95% N_2 on the properties of several materials. Both low and high temperature materials were included. The gas was about 70% saturated with water vapor. The following items were included:

1. Elevated temperature investigation including 6 high temperature alloys for tensile tests, rupture tests and chemical analysis. Two systems of gas atmosphere testing were included on A-286, L-605, X-40, R-41, Inco 702 and U-500. Limited tests were made on welded, brazed and notched specimens.
2. Low temperature investigation including seven low temperature alloys for tensile tests, strain aging tests and impact tests. The alloys included were 302 SS, 316 SS, 6061, AllOAT, Be-Cu, R-41 and 17-7PH. Limited tests were included on welded and brazed specimens.
3. Fundamental mechanism and coating study was done on Inco 702, which is most sensitive to gas environment. Special oxidation cycles, over-aging cycles, strain aging cycles, specimen geometric changes and coatings were tested by rupture techniques to indicate the nature of the mechanism reducing rupture strength and possibilities for preventing strength loss.

This report includes all data obtained and detailed discussion of results. The high temperature rupture strength of each alloy studied was shown to be reduced below the corresponding air atmosphere strength by the special gas. The low temperature properties were shown to be independent of the environmental effects studied. The basic mechanism effecting the high temperature properties has not been clearly determined.

University of Minnesota. WADD TR 60-60. STEADY STATE RESPONSE OF BEAMS WITH TRANSLATIONAL AND ROTATIONAL DAMPING MOTIONS AT THE SUPPORTS. C. C. Fu, T. J. Mentel. AF 33(616)-5426. May 1960. PB 161 957. Order from OTS $1.50.

Two methods of analysis are presented for the steady state response of beams with translational and rotational damping motions at the supports. The first of these methods uses a continuous model and the second uses a discrete (three-degree-of-freedom) model. Both cases are characterized by non-linear equations and approximate solutions are produced for each. Numerical results are presented, giving comparisons and optimum damping configurations.

American Machine & Foundry Company. WADC TR 59-762, Part II. ULTRA-SHORT-TIME CREEP RUPTURE. Joseph S. Ives, Jr. AF 33(616)-5557. May 1960. PB 161 983. Order from OTS $2.75.

The creep behavior of eight structural sheet metals, (321 stainless, 410 stainless, Inconel-X, PH-15-7 Mo, Rene 41, Udimet-500, B120 VCA Ti, and AM 350), when subjected to temperatures up to 2,000°F within 200 milliseconds is presented in tabular and graphic form. The equipment used for these measurements consisted of a Tatnall Model RL-6 Creep machine modified so that a 2000 microfarad bank of capacitors, charged to 5000 volts could be discharged through the test specimen to bring it to the desired test temperature. This initial discharge was followed by an ac current of sufficient magnitude to maintain the specimen at the test temperature.

Data was taken on each of the materials at four creep rates for each temperature. From this data strain vs time, isochronous stress vs strain, and stress vs time curves were plotted and are presented in the Appendix. Data was not reported when an improper transfer of energy from the capacitors to the specimens occurred. Emissivity data, or data from which the emissivity can be calculated, for each of the materials are presented. In addition, the initial transient behavior, due to thermal expansion, of the specimens is given in tabular form.

University of California. WADD TR 60-53. EFFECT OF TEMPERATURE ON THE CREEP OF POLYCRYSTALLINE ALUMINUM BY THE CROSS-SLIP MECHANISM. N. Jaffe, J. E. Dorn. AF 33(616)-3860. June 1960. ASTIA Document No. AD 234 957. PB 171 360. Order from OTS $0.75.

The apparent activation energy for creep of polycrystalline aluminum was determined over the range of 273° to 350° K by the effect of small abrupt changes in temperature on the creep rate. A constant activation energy of 27,400±1000 cal/mole was obtained over strains of 0.003 to 0.23, stresses ranging from 2250 to 6000 p.s.i. and strain rates varying from 0.1145×10^{-5} to 29.5×10^{-5} per minute. Metallographic studies and comparison with theory suggested that creep in this range is controlled by the rate of cross-slip of dislocations.

Both X-ray diffraction analyses and room temperature tensile stress-strain data following precreep revealed that the substructure produced in this range by creep under a given stress depended only on the total creep strain being independent of the actual test temperature. As a result it was deduced that the total strain during creep under a given stress should be a function of temperature compensated time $= te^{\frac{Q}{RT}}$, energy, R the gas constant and T the absolute temperature. A number of creep tests conducted at two different temperatures verified the validity of this conclusion.

Battelle Memorial Institute. WADD TR 60-254. THE EVALUATION OF THE EFFECTS OF VERY LOW TEMPERATURES ON THE PROPERTIES OF AIRCRAFT AND MISSILE METALS. Leonard P. Rice, James E. Campbell, Ward F. Simmons. AF 33(616)-6345. June 1960. PB 171 016. Order from OTS $1.75.

In the past few years, the use of liquefied gases such as oxygen, nitrogen, hydrogen, and helium has increased enormously in the aircraft and missile industries. There is a great deal of interest in metallic materials to store, transport, or otherwise handle these extremely low-temperature liquids. This investigation presents the tensile and hardness properties of eight different alloys of interest to the aircraft and missile industries at temperatures ranging from -253°C (liquid hydrogen) to room temperature. These alloys are Ti-6Al-4V, Ti-4Al-3Mo-IV, Ti-16V-2.5Al, and B-120VCA (all-beta alloy) titanium alloys; 17-7PH, PH15-7Mo, and Type 301XH stainless steels; and Vascojet 1000 alloy steel.

In general, the values for hardness, elastic modulus, and tensile and yield strengths of these materials tended to increase as the best temperature was reduced to -253°C. However, of the eight alloys investigated, the Ti-6Al-4V and the Ti-4Al-3Mo-IV titanium alloys and the Type 301 XH stainless steel were the only ones that did not reveal a serious loss of ductility or did not fracture before reaching 0.2 percent offset strain at -253°C. In most of the other alloys, brittle behavior, as revealed by notched tensile tests, became evident at temperatures of -78°C and below. The titanium alloys have the highest yield strength-density ratios at low temperatures of any of the materials tested.

University of Minnesota. WADD TR 60-280. RHEOLOGICAL PROPERTIES OF ADHESIVES CONSIDERED FOR INTERFACE DAMPING. J. S. Whittier. AF 33(616)-6828. June 1960. PB 171 023. Order from OTS $1.75.

Machines for testing soft adhesives under static compression and shear and under dynamic shear are described. Static creep data in compression and shear are reported for Minnesota Mining and Manufacturing Company's 3M Tape #466. At a given compressive load this material deforms, after sufficient time has elapsed to an "equilibrium" thickness. Dynamic shear data for 3M Tape #466 are reported for frequencies from 0.1 to 120 cps and for shear strain amplitudes from zero to unity and greater. Stress history and fatigue effects are also mentioned. This material is found to dissipate large amounts of specific damping energy when undergoing safe dynamic shear strains. Spot checks on the dynamic shear properties of three other materials at 11.5 cps are also presented. The terms used for data presentation are explained in the report by giving data reduction formulas.

University of Minnesota. WADD TR 60-360. EFFECT OF VISCOELASTIC FOUNDATION ON FORCED VIBRATION OF LOADED RECTANGULAR PLATES. C. T. Hsu, C. W. Chu, C. C. Chang. AF 33(616)-6828. July 1960. PB 171071. Order from OTS $0.75.

Forced vibration of a loaded rectangular plate with visco-elastic foundation is treated. Two types of visco-elastic material are considered, Kelvin-Voight solid and Boltzmann model. Analytic solutions are given for the plate subject to compression and a periodic transverse pressure. A numerical example is worked out for the case of Kelvin-Voight solid. Some features revealed by the examples are:

a. With visco-elastic effect, the natural vibration damps out rapidly (Fig. 3-5).
b. There exists a critical damping factor γ_c. For $\gamma_1 < \gamma_c$ the damping is oscillatory (Fig. 3); otherwise the damping is monotonic (Figs. 4-5).
c. The closer γ_1 is to γ_c in magnitude, the faster the damping.
d. The time required for damping is almost independent of the frequency of the periodic loading.
e. The greater the viscosity coefficient μ_2, the smaller the amplitude of vibration.

The above statement is obtained from preliminary computations. Further work has to be done before conclusive summary can be made.

University of Minnesota. WADD TR 60-426. FATIGUE AND STRESS RUPTURE PROPERTIES OF INCONEL 713C, V-57C and TITANIUM ALLOYS 7A103Mo-Ti AND MST 821 (8Al-2Cb-1Ta-Ti). A. E. Cers, A. A. Blatherwick. AF 33(616)-6826. July 1960. PB 171 064. Order from OTS $2.25.

Fatigue, rupture and creep data at various temperatures obtained at various alternating and mean stress combinations are presented for the alloys Inconel 713C, V-57C (modified Super A-286), 7Al-3Mo-Ti and MST 821 (8Al-2Cb-1Ta-Ti). The tests were performed on unnotched specimens and for Inconel 713C also on notched specimens having a theoretical stress concentration factor of 2.9. The data are presented as S-N curves and stress range diagrams to show the effect of temperature, ratio of alternating-to-mean stress, stress magnitude and specimen notch on the fatigue and rupture properties.

University of Minnesota. WADD TR 60-188. INFLUENCE OF NATURAL FREQUENCIES AND SOURCE CORRELATION FIELDS ON RANDOM RESPONSE OF PANELS. R. F. Lambert, D. H. Tack. AF 33(616)-6828. July 1960. PB 171090. Order from OTS $2.00.

Theoretical and experimental analysis of random responses of panels with a view toward problems of design are presented. The influence of source correlation fields, damping, and boundary impedance are integrated into the analysis. Modal response is viewed from the complex frequency plane in the form of a pole diagram.

The concepts developed from this point of view have direct application in design for specified mean square response. The statistical independence of modes and its relationship to properties of the source and panel are discussed and interpreted for several situations.

New York University. WADC TR 59-416, Part II. INVESTIGATION OF CREEP BUCKLING OF COLUMNS AND PLATES PART II. Creep Buckling Experiments with Columns of Ti-7Al-4Mo Titanium Alloy. Ralph Papirno, George Gerard. AF 33(616)-5807. July 1960. PB 171 082. Order from OTS $1.50.

Creep buckling and short time buckling experiments have been performed on columns of Titanium Alloy Ti-7Al-4Mo with simulated simple support and fixed end boundary conditions. Both end shortening and central deflection were recorded autographically during loading and during creep. Elevated temperature properties of the test material had previously been determined from 3/16 inch thick stock and these were augmented by tests on 3/8 inch thick stock since the mechanical properties and structure were significantly different for the two thicknesses.

The excellent creep properties of the test material indicate that creep buckling tests must be conducted at 950°F or higher for creep buckling to occur in reasonable times at stress levels below the short time compressive yield strength. Since only a narrow range of test conditions is available for creep buckling experiments with this material, column tests were conducted at L'/ρ of approximately 40 at 950° F.

Curtiss-Wright Corporation. WADD TR 60-42. SOME QUANTITATIVE ASPECTS OF FATIGUE OF MATERIALS. Harold N. Cummings. AF 33(616)-6552. July 1960. PB 171 084. Order from OTS $4.00.

In this report are given not only the fatigue properties of many structural materials but also the "static" properties and such other supplementary information as was given in the references consulted. The data are in general from room temperature tests, but a few data are given on tests at higher temperatures. The data are presented in tables and on curves, supplemented by brief discussions in the text.

Bolt Beranek and Newman, Inc. WADD TR 60-468. HIGH VELOCITY ELECTRIC ACCELERATOR SYSTEMS. Jordan J. Baruch, Denis U. Noiseux, Jay H. Ball, C.M. Gogos. AF 33(616)-5730. August 1960. PB 171138. Order from OTS $2.75.

Accelerators designed to convert electrical energy to particulate kinetic energy have been investigated and their capabilities and limitations ascertained. Experimentally, speeds up to 14,500 ft/sec have been obtained with a 20,000 joule energy input. Four different mechanisms for electrical-kinetic energy conversion have been worked out and tested experimentally.

University of Minnesota. WADD TR 60-308. QUASI-ORTHOGONAL MODES OF DYNAMICAL SYSTEMS. Lawrence E. Goodman, Yellappa C. Das. AF 33(616)-6828. August 1960. PB 171 070. Order from OTS $1.50.

When energy dissipating devices are inserted at the boundaries the analysis of structural vibration problems becomes difficult. The usual classical methods in such cases yield an infinite set of equations for the coefficients of modal expansion of the solution. The method developed in this report, gives a finite equation for each coefficient of the modal expansion and thus makes it possible to obtain solutions for structural vibration problems with boundary conditions involving the time derivatives.

The structural vibration problem treated is identical with that of the transient analysis of electrical circuits having distributed capacity and inductance and terminated by a lumped resistance. Certain problems in heat conduction also fall within the scope of the method developed.

American Machine & Foundry Company. WADC TR 59-762, Part III. ULTRA-SHORT-TIME CREEP RUPTURE. C.W.H. Barnett. AF 33(616)-6798. August 1960. ASTIA Document No. AD251 713. PB 171 947. Order from OTS $2.25.

The creep behavior of three structural sheet materials, namely HM-21, H-11 and 310 stainless steel, was studied to temperatures up to 2,000°F within 200 milliseconds. The recorded results are reported in tabular and graphic form. The testing procedure used in implementation of previous contracts for this same type of work has been modified to impose the stress on the material simultaneously with the heating action. In the previous work, on the other hand, the stress was first applied following which step the specimen was heated.

The equipment used for all tests consists of a Tatnall Model RL-6 creep machine, so modified that a charge in a bank of capacitors can be used to heat the test specimen to the desired temperature by controlling the voltage of the capacitor charge.

Data were taken on each of the materials at four creep rates and at three loading rates. The controlled loading rates are an innovation in this contract. From the data obtained, curves of strain vs time, isochronous stress vs strain, and stress vs time were plotted and are presented in the Appendix.

Syracuse University Research Institute. WADD TR 60-310. THE EFFECT OF SEVERAL GEOMETRICAL VARIABLES ON THE NOTCH TENSILE STRENGTH OF 4340 STEEL SHEET HEAT TREATED TO THREE STRENGTH LEVELS. Volker Weiss, John Sessler, Paul Packman, George Sachs. AF 33(616)-6523. September 1960. PB 171396. Order from OTS $1.25.

A systematic study was conducted to evaluate the effects of initial stress distribution and strength level on the notch tensile behavior of a typical high strength steel sheet alloy (SAE 4340) in the presence of stress concentrations.

The initial stress distribution, characterized by the stress concentration factor (K) and the stress gradient at the root of a notch, was determined analytically from the notch geometry according to elastic theory. As part of the study, K was kept constant and the stress gradient allowed vary for different notch depths and section widths.

The 4340 steel alloy sheet was tested in three different heat treat conditions ranging from ductile (F_{tu} = 150 ksi) to moderately notch brittle (F_{tu} = 260 ksi).

For the material conditions investigated, it was found that the effect of stress gradient on notch strength was fundamentally identical with that of section width. Increasing the section width (or decreasing the stress gradient) resulted in a decrease in notch strength when all other factors were held constant. However, the effect was small when compared to the section size effect observed previously for notched cylindrical bars.

In addition, results of tests on an extremely brittle titanium sheet alloy indicated that the stress gradient is the predominant factor that influences notch strength. For a truly brittle material (which exhibits no

plastic deformation prior to fracture), it appears that the stress concentration factor, the depth of a notch and the specimen width are significant only insofar as they contribute to the magnitude of the stress gradient and the maximum stress at the notch root.

Northrop Corporation. WADC TR 56-585, Part II. EFFECTS OF TEMPERATURE-TIME-STRESS HISTORIES ON THE MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL METALLIC MATERIALS - PART II. Stressed Exposure of 7075-T6. C.D. Brownfield, D.M. Badger. AF 33(616)-5769. September 1960. PB 171 328. Order from OTS $2.50.

A study has been made on the problem of predicting strength of a hardened metal alloy after subjection to variable thermal and stress environments severe enough to cause permanent loss of properties. Methods have been developed for predicting tensile ultimate, tensile yield, and compressive yield strengths of 7075-T6 aluminum alloy after single or multiple exposure to various conditions of temperature and stress. An analytical expression suitable for automatic computing machine use has also been developed.

The results of tensile and compressive tests on alclad 7075-T6 aluminum alloy showed that stresses large enough to produce inelastic creep strain during thermal exposure cause reduction in residual strength after exposure. The test results have been used to establish the usefulness of the Larson-Miller exposure parameter for correlating residual strength after simple and complex, stressed and unstressed exposures.

Battelle Memorial Institute. WADD TR 60-278. NOTCH SENSITIVITY OF REFRACTORY METALS. Albert G. Imgram, Frank C. Holden, Horace R. Ogden, Robert I. Jaffee. AF 33(616)-6291. September 1960. PB 171 198. Order from OTS $2.50.

The tensile and notch tensile properties of four refractory metals, molybdenum, tungsten, columbium, and tantalum, and one alloy, Mo-0.5 Ti, were investigated at five temperatures selected to encompass the brittle-to-ductile transition. All specimen failures were classified according to fracture mechanism. The notch sensitivity of each material was evaluated by analyzing the notch and unnotch tensile strength, the ductility transition, and the fracture transition.

WADD TR 60-523. APPLICABILITY OF PRESENT CREEP PREDICTION TECHNIQUES FOR EXTRAPOLATING VERY LONG TIME CREEP BEHAVIOR. K.D. Shimmin. October 1960. PB 171 487. Order from OTS $0.75.

A number of methods of correlation and extrapolation of creep and stress-rupture data are reviewed and discussed briefly. It is shown that, of the time-temperature parameter correlation methods, the Manson-Haferd parameter method has been most successful in correlating existing data. Of the empirical methods of extrapolation, the graphical method of Grant and Bucklin, and the statistical approach reported by Clauss are shown to hold promise as a means of predicting long-time creep data. Recommendations are given for an experiment to provide verification and evaluation of the methods of extrapolation of long-time data.

Ultrasonic Testing & Research Laboratory. WADD TR 60-553. APPLICATION OF ULTRASONICS TO SOLID ROCKET SYSTEMS. R.E. Kleint, R.D. McKown, J.B. Ramsey. AF 33(616)-6603. October 1960. ASTIA Document No. AD 249103. PB 171499. Order from OTS $2.50.

This report contains the results of investigations to establish the applicability of ultrasonic inspection techniques to solid fuel rocket motors. Special emphasis was placed on the detection of unbonded areas at the interfaces of solid fuel rocket motors having a basic construction of a steel case, rubber liner and solid propellant bonded together in that order. The acoustic properties were obtained for these and other materials used for construction of rocket motors. Commercially available ultrasonic inspection and recording equipment was used to obtain data. Various pulse-echo and through transmission techniques were utilized to determine optimum conditions for detecting defects and displaying them on both cathode ray image and C-Scan (plan view) facsimile paper recordings.

Climax Molybdenum Company of Michigan. WADD TR 60-451. EFFECT OF THERMAL-MECHANICAL VARIABLES ON THE PROPERTIES OF MOLYBDENUM ALLOYS. M. Semchyshen, Robert Q. Barr, Gordon D. McArdle. AF 33(616)-5447. November 1960. PB 171 597. Order from OTS $2.50.

Unalloyed molybdenum and three alloys, Mo-050% Ti, Mo-0.059% Zr, and Mo-0.49% Ti-0-057% Zr, have been studied from the standpoint of effect of fabrication variables on mechanical properties and structural stability. Strain hardening was accomplished either by rolling or by forging. Working temperatures were 2200°and 3000°F.

Molybdenum and the above-mentioned alloys were capable of being strain hardened even at low reductions by rolling or forging at 3000°F. Thus none of the reductions studied fell under the scope of true "hot-working". A previously observed contradiction to classic concepts was again observed in that bars reduced up to about 30% in area at 3000°F exhibited higher hardnesses and higher tensile strengths than bars reduced similar amounts at 2200°F. At high reductions, recrystallization during the reduction schedule entered the picture, with the result that bars rolled at 2200°F displayed the higher hardness and strength values.

For any given set of deformation variables, the highest strength properties and greatest resistance to recrystallization were consistently demonstrated by the ternary alloy, Mo-0.49% Ti-0.057% Zr.

The role of externally applied stresses in influencing the recrystallization behavior of the materials under investigation was found to be quite complex. At least part of the complexity of the problem as approached here resulted from the large number of fabrication variables that were inherent in the test specimens. Depending upon the magnitude of the externally applied stress, it was possible either to lower or to raise the hardness as compared with similarly exposed, unstressed specimens.

Four new experimental alloy compositions are discussed. The extrusion of these complex molybdenum-base alloy castings was greatly facilitated by the use of ceramic (Al_2O_3) coated extrusion dies.

University of Minnesota. WADD TR 60-161, Part I. EXPERIMENTS ON SLIP DAMPING AT ROUNDED CONTACTS. L.E. Goodman and G.E. Bowie. AF 33(616)-6828. November 1960. PB 171 594. Order from OTS $2.00.

This report describes an apparatus for studying slip damping at the contacts of a sphere with two flat parallel plates. A load of constant magnitude is applied normal to the plates and the sphere is driven in a direction

tangential to the plates by an electrostrictive device operated at 60 cycles per second. Experimental results obtained with a one half inch diameter type 316 stainless steel sphere pressed between flats of the same material are presented. They are in fair agreement with theoretical predictions by R.D. Mindlin, et al. Results for pyrex glass differ from those for stainless steel and are not compared with theory. Suggestions are made for future work on contact problems.

Nuclear Metals, Inc. WADD TR 60-403.
DEVELOPMENT OF RANDOMLY ORIENTED WROUGHT BERYLLIUM SHEET. F.M. Yans, A.K. Wolff, A.R. Kaufmann. AF 33(616)-6616. December 1960. ASTIA Document No. AD 258241. Order from OTS $2:50.

Various factors affecting texture development in beryllium were studied in an effort to produce randomly oriented wrought beryllium sheet. An extensive literature survey was performed, the purpose of which was to ascertain the major phenomena affecting textural changes in metals, especially beryllium. Texture analysis techniques applicable to the determination of textures in beryllium sheet were evaluated and subsequently standardized. A combination of the original and modified Schulz techniques was chosen as the best method of evaluating the texture of a beryllium sheet sample.

Rolling experiments indicated that the specimen geometry and rolling sequence affected the textures developed during working. It was determined that, during rolling, the basal plane population parallel to the plane of the sheet increases with reduction in area and, when certain specimens are heat treated at high temperatures for short times, the basal plane population parallel to the plane of the sheet is reduced. Further rolling and annealing studies performed on bi-directionally rolled sheet indicated that, when the sheet is subjected to annealing temperatures in excess of 950°C, the location of the basal plane peak intensity is shifted and the intensity is shifted and the intensity profile changes considerably, yielding a third-dimensional ductility of 1.75%. It should be noted that the same sheet in only the stress-relieved condition possesses only 0.2% third-dimensional ductility. The same textural changes were observed in sheet samples manufactured by compression rolling.

BeO particles were dispersed in a matrix of beryllium prior to working and the turbulent flow caused by the presence of these BeO particles reduced the degree of preferred orientation developed for a given working procedure. However, the embrittling effect of the BeO dispersion offset any improvements in properties resulting from the decrease in preferred orientation.

Single crystal rolling and annealing studies, in combination with hot-stage microscopy, further corroborated the fact that twinning is a primary mode of deformation in beryllium sheet. Twin absorption, recrystallization, and grain formation were also studied in the single crystals. It was noted that recrystallization in beryllium can occur at temperatures as low as 1200°F.

University of Minnesota. WADD TR 60-437.
EFFECT OF STRESS NONLINEARITY ON EXTREMAL STATISTICS AND FATIGUE LIFE OF A SIMPLY SUPPORTED BAR. Richard H. Lyon. AF 33(616)-6828. December 1960. ASTIA Document No. AD 258 347. Order from OTS $0.75.

The statistics of the extrema of the stress at the surface of a supported bar are studied when the lowest mode of the bar is excited by random noise. The stress is a nonlinear function of displacement because the hinged ends have a fixed separation. The effect of this nonlinearity on the distribution of maxima and minima is calculated assuming that the tensile stress does not significantly affect the displacement statistics. The effect of nonlinearity on expected fatigue life is calculated for a linear fatigue law. It is found that nonlinearity may significantly diminish the expected fatigue life.

University of Southampton, Hampshire. WADD TR 61-25.
CRITERIA FOR COMPARING THE EFFECTIVENESS OF DAMPING TREATMENTS. D. J. Mead. AF 61(052)-332. January 1961. PB 171 879. Order from OTS $1.25.

In this report, expressions are derived for the response of simple vibrating systems, from which criteria have been deducted to indicate the effectiveness of a damping treatment in attenuating the response. The criteria include factors by which the treatment increases the mass and stiffness of the system, together with the loss factor increment. The response quantities considered include bending stresses, accelerations, inertia forces and sound transmission associated with simple vibrating plates under harmonic and random excitation. Coincidence sound transmission is also briefly considered. It is shown that whereas the mass and loss factor increase is always advantageous, a stiffness increase in some instances is detrimental.

As an example, three different commercial treatments are compared on the basis of some of the criteria. With low treatment weights, the treatment providing the highest loss factor is superior judged by each criterion, but at higher weights according to some criteria a treatment having lower stiffness, density and loss factor is more effective. The existence of optimum treatment weights for maximum effect upon the response is also shown by some criteria.

Martin Company. WADD TR 60-777.
EXPERIMENTATION, ANALYSIS AND PREDICTION FOR ENVIRONMENTAL CREEP. C.J. Giemza. AF 33(616)-6453. January 1961. ASTIA Document No. AD 258 565. Order from OTS $2.50.

An examination was made of the environmental creep behavior of bare 7075-T6 aluminum alloy sheet with the aim of developing an experimental approach as well as relations for characterizing a material's response to an arbitrary stress-temperature-time environment following a prior history during which a metallurgical change has occurred.

A technique called Random Balance (Ref. 2), partly modified to a multiple balance design, was employed to select from several million possible environment test combinations a representative group. The general employment of associated techniques for analysis, common to the statistical field, was precluded because of the creep scatter which was attributed to inhomogeneity in the aluminum sheet.

It was found that a loss of strength in the order of 5% caused by overaging could result in an increase of total creep strain of several hundred percent in subsequent cycles. However, it was also observed that a recovery mechanism, following overaging, could restore a significant degree of resistance to creep.

In every instance, it was found that a transient stage of creep was manifested in each cycle upon reapplication of load. The magnitude of transient creep strain, as well as duration of the transient stage after the initial cycle, diminished as the number of cycles increased for combinations of high stress and high temperature or low stress and low temperature. At test conditions which could be identified with a service environment, the transient creep stage comprised a relatively large fraction of the total creep strain generated during a given cycle.

A qualitative association of creep behavior of the specimens to their origin in the aluminum sheet revealed a pronounced inhomogeneity which could not be related to static tensile properties. Furthermore, small creep strain differences which specimens exhibited at moderate stresses were magnified during subsequent recycling as the magnitudes of stress and temperature increased; alight overaging further severly aggravated the initial disparities among the specimens.

Excellent reproducibility of an arbitrary test environment was possible when the repeat tests were made with specimens which were obtained from adjoining positions in the sheet. Accordingly, as the distances between specimens in the sheet increased, the differences in creep behavior became greater for identical tests, regardless of nominal identities of static properties.

A method was developed which made it possible to predict the environmental creep behavior for an arbitrary test condition during which overaging at 350°F took place and subsequently, when creep resistance was restored as the result of a recovery mechanism. The technique was based on the strain hardening rule compensated with empirically determined factors to account for creep damage and/or subsequent restoration of creep resistance.

During the examination of compatibility of mathematical forms for the representation of the creep phenomenon, an expression ($E_c = Kt^N$) provided a good fit in the primary creep region and a reasonably good approximation for part of the secondary stage of creep; an apparently unique correspondence between the values of K and N and the test variables, stress and temperature also was revealed.

University of Illinois. WADD TR 60-580, Part II. INELASTIC DESIGN OF LOAD CARRYING MEMBERS - Part II. The Effect of End Conditions on the Collapse Load of Columns. G.A. Costello, O.M. Sidebottom, Eugene Pocs. AF 33(616)-5658. January 1961. ASTIA Document No. AD 257 866. Order from OTS $1.25.

A theory was presented for constructing the load-deflection relation and for determining the collapse load of a column having any known end condition. A trial and error solution was required which used interaction curves and assumed that the inelastic column assumed the shape of a sine curve. For time independent (creep) inelastic deformation arc hyperbolic sine interaction curves were used.

The experimental part of the investigation included tests of rectangular section columns made of 17-7PH stainless steel and tested at room temperature and at 972°F. Several slenderness ratios were considered, and the columns had end conditions which were either fixed, equal and opposite end eccentricities, or unequal end eccentricities. Good agreement was found between theory and experiment.

University of Illinois. WADD TR 60-580, Part III. INELASTIC DESIGN OF LOAD CARRYING MEMBERS - Part III. The Significance of an Inelastic Analysis of Eccentrically-Loaded Members. O.M. Sidebottom. AF 33(616)-5658. January 1961. PB 171 878. Order from OTS $0.75.

The author has worked with others on ten investigations, sponsored by Wright Air Development Division, which have considered the theoretical and experimental inelastic analyses of eccentrically-loaded tension and compression members. In all cases good agreement was found between theory and experiment for members tested at room temperature and at elevated temperatures. This investigation was undertaken to consider the significance of an inelastic analysis of eccentrically-loaded members. If the inelastic deformation can be considered time independent, a choice has to be made between an elastic and an inelastic solution. A study was made of the effect of several variables on the ratio of the load necessary to produce a specified inelastic deformation to the maximum elastic load. If the inelastic deformation is time dependent (creep), the only choice is an inelastic solution.

Columbia Univ. WADD TR 60-752. REDUCTION OF THE ENDURANCE LIMIT AS A RESULT OF STRESS INTERACTION IN FATIGUE. Robert A. Heller. AF 33(616)-7042. February 1961. ASTIA Document No. AD 258 024. Order from OTS $1.00.

This paper presents the results of an investigation of the effects of stress interaction on fatigue life of aircraft structural materials subjected to randomized load spectra. All three materials: 2024 and 7075 aluminum and SAE 4340 steel exhibit fatigue lives shorter than those predicted on the basis of the linear (Miner) damage rule. A quasi-linear rule is proposed with a variable, spectrum dependent, endurance limit producing safe life estimates; the dependence of the endurance limit on the stress spectrum and its resulting design inadequacy is shown.

Tests were performed on high speed, programmed rotating bending fatigue machines of special design.

Univ. of Southampton, United Kingdom. WADD TR 61-97. ON INFLUENCE FUNCTIONS IN THE THEORY OF FORCED VIBRATIONS OF MEMBRANES. I. Torbe, D.I.G. Jones. AF 61(052)-332. March 1961. ASTIA Document No. AD 257 818. Order from OTS $0.75.

In this report a general method is outlined for the calculation of the response of membranes with arbitrary boundaries to arbitrary loadings. It is assumed that, by projecting the area of the given membrane on the surface of an unbounded membrane and then applying the given loading to this projection, the application of a suitable load distribution around the boundary of the projection will enable us to satisfy the boundary conditions appropriate to the given membrane. An attempt to find the distribution in question leads to a logarithmically singular integral equation of an unusual type. A few solutions are outlined.

Non-Destructive Testing

TR 5598. COMPARISON OF FLUORESCENT PENETRANT AND MAGNETIC PARTICLE INSPECTION METHODS. Edward Dugger. June 1947.

TR 5898. THE APPLICATION OF STATIFLUX FOR NONDESTRUCTIVE INSPECTION OF NONCONDUCTING AIRCRAFT MATERIALS. Edward Dugger. July 1949.

Stanford Research Institute. WADC TR 54-231, (Part 1). DEVELOPMENT OF NONDESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS. J. S. Arnold. AF 33(616)-2035. October 1954.

Research has been done on the use of sonic methods for the non-destructive evaluation of metal-to-metal adhesive bonds. No aspect of the low frequency behavior (less than 15,000 cps) has been found to be indicative of joint strength. Techniques utilizing high frequency impedance measurements as provided by mechanical driving systems, and steady displacements produced by the application of vacuum cups, have been developed. These techniques show sufficient correlation between bond strength and measurable parameters to justify further investigation, and one or more of them may ultimately provide a satisfactory solution to the non-destructive test problem for adhesive bonds.

Stanford Research Institute. WADC TR 54-231, (Part 3).
DEVELOPMENT OF NONDESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS. J. S. Arnold. AF 33(616)2035. April 1955. PB 111678. Order from OTS $1.00.

An ultrasonic technique is being developed for the non-destructive evaluation of structural adhesive bonds. A ferroelectric transducer is excited by a variable frequency driving current of constant amplitude, and the voltage developed across the transducer is displayed on an oscilloscope as a function of frequency. The voltage-frequency curve conveys information regarding the motional impedance of the system and hence the constraints that limit the vibration of the transducer. These constraints are imposed partly by the mechanical properties of the transducer and partly by the mechanical properties of the load (test specimen) to which it is coupled. The variation in the voltage-frequency curves that occur when the transducer is applied to satisfactory and defective test specimens can usually be correlated with the strength of the adhesive bonds, as indicated by subsequent destructive tests performed on the specimens. Block circuitry, typical oscilloscope patterns, and data on the correlation between destructive and non-destructive tests are presented.

Stanford Research Institute. WADC TR 54-231. (Part 2).
DEVELOPMENT OF NON-DESTRUCTUVE TESTS FOR STRUCTURAL ADHESIVE BONDS. PART 2, MECHANICAL INTERFERENCE TECHNIQUE. J. S. Arnold. AF 33(616)-2035. May 1955.

A mechanical interference technique is described for use in the evaluation of adhesive bonds. This technique uses an optical-mechanical method for measuring small displacements of the skin of a honeycomb sandwich panel as a vacuum-cup induced load is applied. The indication of defects is presented visually in terms of a changing pattern of light and dark "mechanical interference fringes." The technique is a comparatively simple one, but its range of applicability seems to be limited to certain classes of panel defects.

Univ. of Alaska. WADC TR 55-175, Part 1.
METEOROLOGICAL AND SOLAR RADIATION DATA. Wm. S. Wilson. AF 18(600)-40. January 1956. PB 121207. Order from OTS $2.25.

The daily observations of maximum and minimum temperatures, maximum, minimum, and mean of the relative humidity with the approximate time (Alaskan Standard Time) of the occurence of each maximum and minimum, maximum intensity of solar radiation and total radiation received on a horisontal surface and a surface slanted 45° to the south are tabulated along with the monthly averages and totals of each of these factors as obtained in connection with the project on the "Exposure of USAF Materials near College, Alaska." The temperature data for a given year are represented on a composite graph. The monthly values of all data are accumulated in separate tables.

Univ. of Alaska. WADC TR 55-175, Part 2.
METEOROLOGICAL AND SOLAR RADIATION DATA. Wm. S. Wilson. AF 18(600)-40. January 1956. PB 121185. Order from OTS $1.25.

The daily observations of maximum and minimum temperatures, maximum, minimum, and mean of the relative humidity with the approximate time (Alaska Standard Time) of the occurrence of each maximum and minimum, maximum intensity of solar radiation and total radiation received on a horizontal surface and a surface slanted 45° to the south are tabulated along with the monthly averages and totals of each of these factors as obtained in connection with the project on the "Exposure of USAF Materials near College, Alaska." The temperature data in each year are represented on a composite graph. The monthly values of all data are accumulated in separate tables. The period covered is from 1 September 1954 through 31 August 1955.

Goodyear Aircraft Corporation. WADC TR 55-322.
EVALUATION OF TESTERS FOR THE DETERIORATION OF DOPED-FABRIC SURFACES ON AIRCRAFT. Bruce K. Bain, Lawrence P. Slivka. AF 33(600)-27305. February 1956.

Three types of portable doped-fabric testers, the Mobile AMA (Hughes) tester, Seyboth fabric tester, and CAA impact tester, are evaluated as methods of determining deterioration of doped-fabric surfaces on aircraft. Correlation of these testers with the Mullen burst tester is made, and the effects of different temperatures and relative humidities on the tester readings are determined. It is found that the Mobile AMA (Hughes) tester is useful as a survey instrument for determining deterioration of doped-fabric surfaces on aircraft. In their present forms, the CAA and Seyboth testers are found to be useful only for weaker fabrics than those specified by Air Force standards.

Battelle Memorial Institute. WADC TR 56-607.
SURVEY TO DETERMINE THE AIR FORCE RESEARCH AND DEVELOPMENT NEEDS IN THE FIELD OF NON-DESTRUCTIVE TESTING. S. A. Wenk, A. J. Schwarber, Jr., R. R. Showalter. AF 33(616)-3475. June 1957. ASTIA Document No. AD 130848.

The increasing complexity of air weapons systems has resulted in increased dependence on nondestructive testing as a means of assuring the conformance of aircraft materials and components to design criteria. This survey reviews the problems inherent in existing non-destructive testing methods, as well as those inspection problems for which no present nondestructive test is adequate. In addition, the shortage of skilled personnel, and the necessity of establishing training programs, is cited.

Specific problems which require a research and development effort are enumerated and, where applicable, recommendations for action have been made, based on Air Force need and chances for technological success.

Ultrasonic Testing & Research Laboratory. WADC TR 57-268.
RESEARCH AND DEVELOPMENT LEADING TO THE ESTABLISHMENT OF ULTRASONIC TEST STANDARDS FOR AIRCRAFT MATERIALS. A. D. Guinn, J. C. Fols,

R. D. McKown, W. C. Hitt. AF 33(616)-3363. October 1957. ASTIA Document No. AD 142034. PB 131564.

Very little information has been available for use by engineering personnel interested in setting up ultrasonic inspection and testing reference standards for use in determining the acceptability of aircraft materials. Since the ultrasonic method of inspection and testing of aircraft materials is now widely employed, a very definite need exists for test standards for use in determining the acceptability of aircraft materials undergoing this method of inspection and testing. While this research program was somewhat limited in its scope, it is believed that the data collected will provide the basis for the establishment of ultrasonic test standards for the aircraft materials studied.

Franklin Institute. WADC TR 58-35.
EARLY DETECTION OF FATIGUE IN ALUMINUM ALLOYS BY ULTRASONICS. Karl Sittel, Marvin Herman, Robert C. Good, Jr. AF 33(616)-3930. May 1958. ASTIA Document No. AD 155558. PB 151019. Order from OTS $1.25.

The literature concerning ultrasonic wave propagation and fatigue-induced damage in aluminum has been surveyed to determine the possible interactions. The causes of wave attenuation have been compared with the fatigue effects in metals predicted by various theories to determine the experimental conditions that would maximize the measurements of those effects. Particular attention is paid to the dislocations present in metals because they form and move throughout the fatigue life of a sample. The problem at hand is the detection of incipient fatigue which implies early detection and measurement.

The experimental work has included the design and construction of a resonant type fatiguing machine and an electro-acoustic driving system for ultrasonic waves. Enough tests were made to show the operability of the individual parts of the system, but insufficient time was available after the literature search to record full-scale tests. The specimen shape to be recommended for further tests is a torsion bar with the ultrasonic waves propagated along its axis.

Stanford Research Institute. WADC TR 54-231, Part 6.
DEVELOPMENT OF NONDESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS. J. S. Arnold. AF 33(616)-3833. June 1958. ASTIA Document No. AD 155632.

An ultrasonic technique for evaluation of structural adhesive bonds (the STUB-meter) is being developed and tested. The operation of the instrument is based on the empirically observed fact that the behavior of a ferroelectric transducer, when mechanically coupled to a test specimen, is affected by the structural properties of the test specimen. An evaluation program has been carried out in cooperation with organizations in the aircraft industry. The results of the program prove the technique to be useful, and do much to define the proper ranges of parameters for its application.

Laboratory work has produced a significant improvement in the description of the electromechanical phenomena of the method, and experimental apparatus has been constructed to take advantage of this information.

Univ. of Illinois. WADC TR 59-412.
NONDESTRUCTIVE TESTS FOR CERAMIC, CERMET AND GRAPHITE MATERIALS. Julian H. Lauchner, Dwight G. Bennett, George L. Morgan. AF 33(616)-5687. November 1959. PB 161815. Order from OTS $1.75.

As a direct result of the stringent requirements placed upon ceramic, cermet and graphite materials when contemplated for use as components in flight and space travel, the feasibility portion of this study was divided into three areas. External and internal defects, residual stress, and particle size measurements in polycrystalline bodies were investigated and their subsequent effect on static strength indicated.

Radiographic studies of polycrystalline ceramic bodies resulted in internal flaw detection of one or more percent of the total body cross section. Surface flaws were more reliably detected in nonporous bodies by fluorescent penetrant inspection, however, neither method was found to be applicable to relatively porous bodies. Static loading of specimens, non-destructively tested, indicated the extreme importance of surface structure and texture, whereas internal flaws were in many cases negligible in strength considerations.

The state of residual stress existent in a body was found to be a first order factor in predicting performance. Controlled residual stress developments in composite bodies formed the basis of the second phase of the study. Thermal expansion, elastic moduli, shape factors and heat treatment were employed in development and analysis of residual stress levels. Sandwich and concentric cylindrical type specimens were prepared and subsequently subjected to transverse loads. Measured apparent specimen strengths were analyzed in terms of residual body stress. Change in apparent specimen strength was found to be a direct function of the residual stress in the surface layer; however, certain limiting factors were found. In the balanced stress system, specimen strength decreased when internal stress, either compressive or tensile, exceeded some critical value characteristic of the body.

Thermal treatment studies indicated that body density resultant from body forming techniques or firing schedules, was a measure of body maturity and general freedom from macroscopic defects. Within limited density ranges, increased density values were observed to result in increased flexural strengths.

Relative particle size analysis by X-ray back reflection techniques correlated with flexural strength of alumina bodies for the average particle ranges between one and ten microns.

Stanford Research Institute. WADC TR 54-231, Part VII.
DEVELOPMENT OF NONDESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS. J. S. Arnold, C. T. Vincent. AF 33(616)-5508. December 1959.

An ultrasonic technique for the evaluation of structural adhesive bonds has been developed and tested. Several fabricators of such adhesive bonds have participated in the testing program, and the results of these tests contribute to the extensive body of information now available relative to the technique.

Laboratory work with the mechanical loading of the transducers has led to a better understanding of the relationship between bond quality and the indications thereof that are provided by ultrasonic instrumentation. As a result of this work it has been possible to construct simplified apparatus that appears to be suitable for production testing.

Automation Instruments, Inc. WADC TR 59-302.
INVESTIGATION OF METHODS FOR DETERMINING ACTUAL FLAW SIZE IN MATERIALS BY NON-DESTRUCTIVE ULTRASONIC TECHNIQUES. Gerald J. Posakony, Merle R. Loetz. AF 33(616)-5714. December 1959. PB 161530. Order from OTS $1.75.

The investigation of methods to determine actual flaw size in materials by ultrasonic non-destructive techniques has led to the development of breadboard equipment capable of displaying additional flaw information. By electronic networks, the ultrasonic reflections are displayed into a three-dimensional view of the internal structure of a flat metal part. Three dimensional pictures are included to show the resultant of the single and multiple transducer techniques. Various frequencies and materials are evaluated to establish preliminary applicability of the processes.

Ultrasonic Testing & Research Laboratory.
WADC TR 59-466.
RESEARCH AND DEVELOPMENT LEADING TO THE ESTABLISHMENT OF ULTRASONIC TEST STANDARDS FOR AIRCRAFT MATERIALS. G. L. Cross, R. E. Kleint, R. D. McKown. AF 33(616)-5877. February 1960. PB 161753. Order from OTS $2.75.

This report contains the results of a four phase investigation of materials and techniques having to do with the detection and evaluation of internal flaws by ultrasonic means. The four questions studied were:

1. What material variables affect the transmission of ultrasonic compressional waves in certain steel, aluminum, and titanium alloys?
2. What material and geometrical variables affect the transmission of ultrasonic shear and surface waves in thin sheet materials?
3. What are some of the quantities involved in altering the ultrasonic beam diameter by collimation?
4. What correlation exists between the ultrasonic characteristics and mechanical properties of certain aluminum and steel materials which contain large quantities of minute porosity?

To obtain complete answers to the above questions would, obviously, require more effort than the twelve (12) month investigation concluded. However, certain findings were considered of note in that they partially answer the questions and at the same time give rise to more interesting questions for further research. Briefly, these findings were as follows:

1. Most significant of the material variables affecting compressional wave energy is the amount of working to which the material is subjected, mainly a function of material production methods and the as wrought dimensions.
2. The more important factors in determining the quantities of shear and surface wave energy are the sheet thickness and the flaw size to sheet thickness ratio.
3. A very useful system for selecting collimator orifice diameters is that described by the function $D = 2\sqrt{R\lambda}$, where D is the diameter, R is the flaw depth, and λ the wavelength. Collimator design should be such that the collimator does not adversely interfere with the incident or reflected beam.
4. Ultrasonic and mechanical properties show good correlation as far as fatigue life and ductility are concerned. The results indicate that the transverse properties are more severely affected by small discontinuities.

Nuclear Measurements

WADC TR 59-119.
REMOVAL DOSE AS AN ENVIRONMENTAL MEASUREMENT OF X-RAYS AND GAMMA RAYS. R. L. Hickmott. April 1959. ASTIA Document No. AD 211918. PB 151838. Order from OTS $1.00.

The removal dose of any material is the net energy per unit mass that a limiting small mass of the material transfers from the incident photon flux to the kinetic energy of the associated flux of charged particles. Removal dose is compared to both absorbed dose and exposure dose (roentgen) in terms of cross sections and spectra. Its relation to radiation effects is compared to the similar relation of absorbed dose.

WADC TN 59-167.
RADIATION INDUCED ULTRA-VIOLET ABSORPTION IN METHYL METHACRYLATE AS A METHOD OF DOSIMETRY. D. R. Johnson. June 1959.

Most current methods of radiation dosimetry are very limited in range and become useless after relatively low integrated dosages; i.e., 10^8 to 10^9 ergs gm.$^{-1}$ (C). Hence, the dosimetry problem becomes quite significant when using the higher dose levels and integrated dosages which are needed to simulate the radiation environments of high performance nuclear systems. This technical note summarizes a series of experiments concerned with the development of an accurate method of dosimetry for measuring the dosages encountered when radiation effects are studied at higher levels. The method investigated utilizes the radiation induced ultra-violet absorption in methyl methacrylate for correlation with total dosages.

The radiation induced ultra-violet absorption in methyl methacrylate has been investigated in detail using gamma radiation, and found to provide a convenient, inexpensive and accurate method of measuring radiation dosages from 10^7 ergs gm.$^{-1}$ (C) to about 2×10^{10} ergs gm.$^{-1}$ (C). However, the ultimate accuracy of the method depends entirely on its calibration by a standard source of radiation for it is entirely an empirical method.

WADC TN 59-401.
ATTEMPT TO EVALUATE CHLORINATED HYDROCARBON DOSIMETERS BY CROSS CALIBRATING ELEVEN GAMMA RAY SOURCES. Robert L. Hickmott, Maurice J. Cote, Philip B. Hemmig. November 1959.

An evaluation of the chlorinated hydrocarbon dosimetry system of Convair, Fort Worth, was attempted by the Materials Laboratory, Wright Air Development Center, with the cooperation of a number of high intensity irradiation facilities throughout the country. The evaluation consisted of two phases. The first phase tested the precision of the dosimetry system. It involved the irradiation of thirty five single dosimeters at Wright Air Development Center. The second phase was intended to field test the dosimetry system. It was also hoped that data from this phase would permit the cross calibration of the participating facilities.

Data obtained in this program is presented with an analysis of the results. Variations from facility to facility, and within the same facility, were noted which cannot be explained by the available information, however, the program was not specifically designed to separate rate effects, spectrum effects, and temperature, storage, and handling effects which could have influenced the results.

Although the data appear inconclusive they illustrate the problems that may arise in applying such dosimetry systems, and demonstrate the desirability of further work to cross calibrate the many irradiation facilities.

WADC TR 59-4.
MEASUREMENT OF SCATTERED GAMMA RAYS BY INFOIL ACTIVATION. Michael P. Charles. December 1959. ASTIA Document No. AD 209388. PB 151707. Order from OTS $4.00.

It is the purpose of this report to determine the scattered radiation component of pure gamma-ray sources used in radiation effects studies.

The method to be developed involves the excitation of metastable levels in such materials as In, Cd, Ag, Lu, Hg, Pt, Hf, Sr, and Rh (Table I) by gamma-ray inelastic scattering processes. The direct excitation of the metastable states is forbidden, but these levels can be excited indirectly if the Co^{60} gamma rays have been degraded in energy-so that the remaining energy corresponds to that of a level higher than the metastable levels but which decays immediately to the metastable level.

Although the cross-section for gamma-gamma activation at these levels has not been measured, the relative activation of such foils throughout the irradiation volume was found to provide considerable information on the energy distribution of scattered radiation in the WADD kilocurie gamma ray sources.

WADC TN 59-372.
CHEMICAL DOSIMETRY. Dennis R. Johnson, Lowell A. King, Gordon Wepfer. January 1960.

In radiation effects and radiation chemistry studies, an accurate specification of the radiation dose is absolutely essential. Chemical dosimetry is one area which has received a considerable amount of attention for both low and high level radiation doses. This technical note summarizes the work initiated to study systems which would be usable to higher total doses than those currently available and still maintain a certain degree of ease of handling and read-out methods. The ideas generated during this work and the experiments and evaluations performed depend on a rather broad definition of the term, chemical dosimetry. Several unrelated methods and systems have been studied with varying degrees of completeness.

WADC TR 59-711.
OPTICALLY ACTIVE ORGANIC COMPOUNDS AS HIGH LEVEL GAMMA DOSIMETERS. Stanley M. Dec. February 1960. PB 161881. Order from OTS $1.00.

Materials which are of necessity located in high level radiation fields frequently undergo degrading effects. A measurement for high level radiation is an important factor in the practical application of materials in nuclear environments.

This project was initiated in order to develop a dosimeter system which could extend the range of current dosimeter methods. A new technique was used, wherein the effects on the rotation of optically active organic compounds were determined with gamma radiation. The program was directed toward obtaining characteristics of a number of probable dosimeter systems. Linear or near linear response was observed for several systems to a range of at least 10^{10} ergs $gram^{-1}$ carbon. Maximum range levels were not determined since a considerable length of time is needed for irradiations at higher dose..

WADC TN 60-48.
DESIGN OF A DUAL SENSOR GAMMA RAY CALORIMETER. James R. Coss, Philip B. Hemmig. March 1960.

Design for a small, dimple, dual element calorimeter for use in measuring gamma dose rates in the range above 10^7 ergs/gm(C) is presented. Experimental data, design, and theoretical considerations of the calorimeter are presented.

WADD TN 60-17
INCREASING THE DOSE RATE OF THE WADD KILOCURIE COBALT-60 FACILITY BY ALTERING ITS GEOMETRY. D. J. Dunbar, D. R. Johnson, R. E. Rondeau. March 1960.

On 16 June 1959, the configuration of the WADD 20,000 curie Cobalt-60 source was changed in order to increase the dose rate inside the cylindrical irradiation volume. The original source basket with a diameter was 11.6 inches was replaced with a new basket with a diameter of approximately 6.0 inches, thus clustering the aluminum encased Cobalt-60 rods nearer the axis of the source.

The dose rate in the center of the new configuration has been found to be 6.16×10^7 ergs gm^{-1} (C) hr^{-1} which represents a boost of nearly 100% over the former rate of 3.15×10^7 ergs gm^{-1} (C) hr^{-1}. A schematic map of currently available dose rates in the facility is presented in the form of isodose lines representing dose rates of 6.0, 5.5, 5.0, 4.5, 4.0 x 10^7 ergs gm^{-1} (C) hr^{-1}.

Airborne Instruments Laboratory. WADD TR 60-98. MICROWAVE GAMMA-RAY ION CHAMBER. Donald A. Brown, Karl C. Speh, Jesse J. Taub. AF 33(616)-5758. May 1960. PB 161986. Order from OTS $1.75.

A microwave gamma-ray ion chamber and its associated equipment has been developed that is capable of measuring gamma radiation in the range from 10^6 to 10^{10} ergs per gm-hr (carbon). This equipment operates at a microwave frequency of about 9 kmc and measures radiation by using the changes in the reflection coefficient looking into a gas-filled microwave resonant cavity caused by ionization of the gas due to radiation. Data on gas conductivity and resonant frequency shift have been taken for different gas mixtures.

Aerojet-General Nucleonics. WADC TR 59-607. STANDARDIZATION OF FABRICATION TECHNIQUES (THRESHOLD FOILS). I. E. Lamb, R. R. Tsukimura. AF 33(600)-35917. May 1960. PB 161982. Order from OTS $2.50.

The techniques used to develop and standardize a series of neutron detection foils for measurement of neutron spectra are described. The series includes gold and cobalt foils, and plutonium-239, uranium-238, and sulfur threshold foils. A method of calibrating the foils with a low-flux research reactor is discussed. The instrumentation required to obtain data from the foils is presented, together with the neutron intensities that may be explored with these detectors.

Texas Nuclear Corporation. WADD TR 60-293. A PROTON-RECOIL ORGANIC SCINTILLATION SPECTROMETER AND INVESTIGATION INTO A VARIABLE THRESHOLD SPARK COUNTER. John B. Ashe, William E. Tucker, O. M. Hudson, Jr., J. T. Prud'homme. AF 33(616)-6362. July 1960. PB 171120. Order from OTS $1.00.

This report describes the research which was conducted by Texas Nuclear Corporation on two possible neutron spectral measuring devices based on the detection of recoil protons.

A scintillation detector is described which has the ability to differentiate between neutrons and gamma rays by means of the different decay times of recoil proton and Compton electron scintillations. This detector has been incorporated into a neutron spectrometer which is highly insensitive to gamma rays over a dectable neutron energy region of approximately 0.8 Mev to greater than 17 Mev. Data obtained with the spectrometer are presented.

In addition to the scintillation detector, investigations into the feasibility of using a spark counter as an energy sensitive neutron detector are also described. The results of these investigations are discussed and representative data are presented.

WADD TR 60-544.
RADIOCHEMICAL PRODUCTION AND FATE OF FREE RADICALS. Roger E. Rondeau. August 1960. PB 171 310. Order from OTS $0.75.

This report represents an effort to interpret and correlate to a limited extent, the many physico-chemical phenomena of radiation chemistry in terms of elementary free radical processes.

TRG, Incorporated. WADC TR 59-443.
FANTASIA AND TRIPROD - SHIELDING CODES FOR THE 1103A UNIVAC. Herbert Steinberg, Jerome Heitner, Raphael Aronson. AF 33(616)-5186. September 1960. ASTIA Document No. AD 249258. PB 171 945. Order from OTS $2.50.

Two codes have been developed for the 1103A Univac. FANTASIA computes neutron transmission through laminated slab shields and slowing down density within the shields by Monte Carlo methods. TRIPROD is a slowing down code suitable for shielding problems. It is based on the General Electric reactor code VALPROD. Operating instructions are included.

Hebrew University, Jerusalem, Israel. WADD TR 60-668.
THE RADIATION CHEMISTRY OF CHLOROFORM SYSTEMS. Gabriel Stein, M. Ottolenghi, I. Eliezer. AF 61(052)-224. September 1960. PB 171 497. Order from OTS $0.75.

A dosimeter based on solutions of biallyl in chloroform is developed. The radiation chemistry of pure H_2O and O_2 free chloroform is investigated and all products identified. A reaction mechanism is derived.

TRG, Incorporated. WADD TR 60-312.
THE SPM (STOCHASTIC PROCESS METHOD) EQUATION FOR CALCULATING THE SPATIAL VARIATION OF NEUTRON SPECTRA. Carl N. Klahr, H. Julius Zell. AF 33(616)-5187. September 1960. PB 171 397. Order from OTS $3.00.

The SPM equation is a second order partial differential equation in lethargy (or energy) and spatial coordinates. It promises to describe the slowing down and spatial diffusion of neutrons with sufficient accuracy for shielding problems and for other applications where neutron flux spectra are critical. A primitive SPM with infinite orders of derivatives is first obtained from either the Smoluchowski or Boltzmann equations. It is then truncated after the second order derivatives. The coefficients of two forms of this equation are obtained and suitable boundary conditions are given. A code has been written to solve this equation on the 1103A Univac. Various solutions are given.

U. S. Naval Research Laboratory. WADD TR 60-563.
DOSIMETRY BY LUMINESCENCE DEGRADATION IN ORGANICS. Frank H. Attix. MIPR (33-616)58-21. October 1960. PB 171 491. Order from OTS $1.75.

This report covers an investigation of the phenomenon of photoluminescence degradation in organic materials due to damage by ionizing radiations.

A survey of previous studies of this effect is presented.

The present work stresses reduction to a practical dosimetry system for the 5×10^5 to 5×10^9 rad absorbed dose range. It is demonstrated that pressed wafers of anthracene and of p-quaterphenyl can be employed together to cover the required dose range. Ultraviolet light (3650 angstrom) is used to excite the photoluminescense, which is observed in a narrow band at 4420 angstrom.

Some recovery of photoluminescense with time after X-ray irradiations is observed, and heat treatment is proposed as a method of reducing this effect.

Degradation of luminescence in biphenyl, p-terphenyl, fluorene, naphthalene, and tetracene have also been studied and are reported upon.

U. S. Naval Research Laboratory. WADD TR 60-562.
A SOLID STATE BRAGG-GRAY CAVITY CHAMBER. Victor H. Ritz, Frank H. Attix. MIPR (33-616)-59-27. October 1960. PB 171 490. Order from OTS $0.75.

An attempt has been made to construct the solid state analogue of a Bragg-Gray cavity ionization chamber. Thin layers of materials sensitive to radiation have been placed between walls of C, Al, Cu, Sn or Pb. Two types of sensing agents have been employed; thin layers of anthracene whose ultraviolet induced luminescence degrades upon irradiation, and 6 micron thick polyethylene terephthalate (Mylar) films whose optical density at 3250A increases upon irradiation. Results in a Co^{60} γ-ray field with the anthracene disagree sharply with theory, while the Mylar film yields excellent agreement (±5%) with theory. A preliminary attempt to discriminate between the fast neutron and gamma ray dose in a nuclear reactor has been unsuccessful.

WADD TR 60-645.
RADIATION EFFECTS ON ELECTRONIC COMPONENTS: A COMPARISON OF PAPER CAPACITORS IMPREGNATED WITH VITAMIN "Q" MINERAL OIL AND BIS-(P-PHENOXPHENYL) ETHER. Dennis R. Johnson, Milton E. L. Zellmer. November 1960. PB 171 783. Order from OTS $1.00.

Bis-(p-phenoxyphenyl) ether (BPPE) when irradiated proves to be highly resistant to radiation damage. Paper capacitors impregnated with the ether were manufactured by the Sprague Electric Company, North Adams, Massachusetts in an effort to develop radiation resistant electronic components. The capacitors were irradiated in the kilo-curie Cobalt-60 gamma facility of the Materials Central, Wright Air Development Division. Simultaneously, Sprague standard Vitamin "Q" mineral oil impregnated capacitors were irradiated for purposes of comparison.

Capacitance, dissipation factor and insulation resistance were measured throughout the tests lasting from 200 to 700 hours, depending upon the type of test employed. In most cases, the effects were an initial decrease and then an increase in capacitance, an increase in dissipation factor, and a large decrease in insulation resistance. The most dramatic effect was a marked decrease in capacitor lifetime in the gamma environment. Most failures were attributed to catastrophic breakup of the units due to internal gassing of the dielectric; it was this property which was significantly improved by the substitution of the ether for Vitamin "Q". In most instances, the BPPE units lasted three to four times as long as their Vitamin "Q" counterparts. However, the paper used in the capacitors was found to be a major contributor to the generated gases; therefore, the problem of catastrophic failure of the capacitors was only partially solved. In separate tests, both the Vitamin "Q" and the paper contributed enough gas individually to cause failure, while the BPPE essentially did not gas, thus, little was gained by the use of BPPE as the dielectric since the major

culprit was the paper.

Armour Research Foundation. WADD TR 60-344.

A STUDY OF THE NATURE OF FREE RADICALS IN IRRADIATED CHEMICAL SYSTEMS. P. Y. Feng, W. A. Glasson, S. A. Marshall. AF 33(616)-6141. November 1960. PB 171 596. Order from OTS $1.75.

In order to better evaluate the factors which dictate the susceptibility of organic compounds to form free radicals on exposure to ionizing radiation and the nature and fate of said radicals, a study has been made of the radiation chemistry of various organic compounds. The compounds studied were chosen in order to ascertain the effect of structure and presence of functional groups within the basic carbon skeleton on the radiation damaging of the various substrates. Compounds studied were n-, sec-, and tert-butyl iodides, tert-butyl amine, n-butanol, n-butyric acid, and ethyl acetate. The alkyl iodides were studied in order to ascertain the effect of structural isomerism on a system whose primary dissociative step is known, i.e., carbon-iodine bond fission. The other compounds were studied in order to evaluate the effect of changing the functional group (alcohol, amine, acid, etc.) and the over-all structure (ester) in a four carbon organic system.

The systems were studied using product analysis as the main tool. Gas-liquid partition chromatography was the analytical method used for the liquid products while manometric and mass spectrometric means served for the gaseous products. In addition electron spin resonance techniques were used as a means for determining directly the radical(s) present.

The results indicate that over-all yields of products increase on changing from a primary to a secondary to a tertiary bond to the dissociable entity. Further, on changing the functional group the character of the products change as the bond energy in question approaches that of the carbon-hydrogen bond. The results are in accordance with the view that the products can be predicted on the basis of the relative bond strengths within the molecule and classical theories of free radical chemistry.

Packaging

TR 53-43.

THE SELECTION OF CUSHION AREA IN THE DESIGN OF PACKAGE CUSHIONING. Roger B. Orensteen, 2/Lt. March 1953. PB 111227. Order from OTS $0.50.

Criteria of package cushion area selection are discussed for minimizing cushioning volume and container cubage. Stress-strain, energy-stress, and ratio of stress to energy are shown for a typical cushioning material. Procedures are outlined for using the stress value corresponding to the minimum ratio stress to energy as a criterion in cushion area selection for optimum package design.

TR 53-68.

A TECHNIQUE FOR THE DESIGN OF GLASS FIBER PACKAGE CUSHIONING. Roger B. Orensteen, 2/Lt. March 1953. PB 111228. Order from OTS $0.50.

A technique is demonstrated by which static cushioning data can be used in the design of glass fiber package cushioning. Design curves are provided for selecting density, thickness, and cushion area for economical cushion design. Consideration is given to the advantages of glass fiber cushioning of small area and high density.

Foster D. Snell, Inc. TR 53-22, Part 1.

PACKAGING STUDY OF AIR FORCE INSTRUMENTS. W. L. Hardy and Marvin Masel. AF 33(616)-211. April 1953.

Packaged instrument accelerations resulting from a drop are calculated for linear cushions. An evaluation is made of the effects of non-linearity. Element damage due to instrument acceleration is described. This is done for impact due to a drop and for forced vehicular vibration. A procedure is developed for finding an optimum cushioning material and thickness for an instrument of given unit loading and fragility rating. Methods of specifying the fragility of an instrument are discussed.

U. S. Forest Products Laboratory. TR 53-38, Part 20.

PACKAGING REQUIREMENTS FOR BEARINGS. PART 20 - ANTIFRICTION BEARING CAN PACKAGES SUBJECTED TO CYCLIC EXPOSURE AND 120 DAYS OF STORAGE. R. K. Stern. AF 18(600)-103. July 1953. ASTIA Document No. AD 22063. PB 122339. Order from LC Mi $2.40, Ph $3.30.

Tin cans containing single antifriction bearings with various inner packaging were subjected to a 3-week cyclic exposure plus storage for 120 days at 160°F. and 92 percent relative humidity.

Separable-type ball bearings with steel, fiber, or brass retainers were coated with either a synthetic grease or a petroleum-base preservative, wrapped with 1 of 13 different intimate wraps, and sealed in cans. In addition, coated bearings of each type were packaged without intimate wraps and sealed in cans, and others were sealed in cans that were filled with preservative or grease. The can exteriors were spray-coated to varied thicknesses with one of two kinds of paint.

All bearings packaged without intimate wraps and sealed in cans filled with grease or preservative were free from corrosion and stain after testing. Good results were also obtained when intimate wraps of nylon, cellulose acetate-kraft, or polyethylene-kraft were used with synthetic grease, Specification MIL-G-3278, Grease; Aircraft and Instruments (For Low and High Temperatures). In general, erratic results were obtained when the preservative conforming to Specification AN-C-121, Compound; Soft Film, Corrosion Preventive, was used.

The best over-all results were obtained with the procoating, when paint that conformed to Specification MIL-C-10506, Coatings, Exterior, for Tinned Food Cans, was sprayed on the cans to an average dry film thickness of 1.3 mils.

U. S. Forest Products Laboratory. TR 53-38, Part 19.

PACKAGING REQUIREMENTS FOR BEARINGS. PART 19 - PERFORMANCE OF TIN CANS CONTAINING ANTIFRICTION BEARINGS AND VARIOUS GREASES AND PRESERVATIVES. R. K. Stern. AF 18(600)-103. July 1953. ASTIA Document No. AD 21968. PB 122338. Order from LC Mi $2.40, Ph $3.30.

Tin cans containing antifriction bearings prepared with various amounts of grease or preservative were subjected to a 3-week cyclic exposure plus storage for 60 days at 160°F, and 92 percent relative humidity.

Two of the preservatives complied with Specification MIL-C-11796 Corrosion Preventive, Petrolatum, Hot Application and three with Specification AN-C-124, Compound; Soft Film, Corrosion Preventive; the two

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greases complied with Specification MIL-G-3278, Grease; Aircraft and Instruments (For Low and High Temperatures). Two proprietary compounds of the petrolatum type were also tested.

The bearings were either dip-coated with grease or preservative before they were inserted in the cans or they were inserted in cans filled or half-filled with grease or preservative. The preservatives were applied at 165°F. (±5°F.) or at 225°F. (±5°F.). The greases were applied at approximately 80°F.

Immersion of the bearings in greases or preservatives was more effective than dip-coating for preventing corrosion and staining. Cans, whether full or half-full, performed equally, except that more satisfactory results were obtained in the case of grease, Specification MIL-G-3278, when the cans were full. Equally satisfactory results were obtained with preservative, Specification MIL-C-11796, regardless of the temperature of application. However, where preservative, Specification AN-C-124 was used, better results were obtained with the 225°F temperature of application. All bearings preserved with the proprietary compound by any method were free of stain and corrosion.

Forest Products Laboratory, United States Department of Agriculture. TR 53-38, Part 17.

PACKAGING REQUIREMENTS FOR BEARINGS. PART 17 - THE PERFORMANCE OF TIN CANS CONTAINING TWO ANTIFRICTION BEARINGS PER CAN WITH VARIOUS INNER PACKAGING. R. K. Stern. AF 18(600)-103. October 1953. PB 122336. Order from LC Mi $2.40, Ph $3.30.

Tin cans, each containing two antifriction bearings, were subjected to a three week cyclic exposure plus 120 days storage at 160°F and 92 percent relative humidity.

Ball bearings with steel, fiber, or brass retainers were coated either with a synthetic grease, Specification MIL-G-3278 (Grease; Aircraft and Instruments, for High and Low Temperatures) or petroleum preservative, Specification AN-C-124 (Compound; Soft Film, Corrosion-Preventive). The bearings were then sealed in cans; or, after being coated, wrapped with each of 10 different intimate wrapping materials and sealed in cans; or, after being coated, sealed in cans which were filled with the petroleum preservative or synthetic grease.

Results indicate that the preservation of two bearings within a can with proper packaging materials is practical and compares favorably with results obtained where one bearing is preserved in a can with similar packaging materials.

The synthetic grease performed more effectively as a preservative than the petroleum preservative. Very good results were obtained from the cans filled with the preservative or grease. The most effective intimate wraps used with the synthetic grease were: cellulose acetate-kraft (plain or creped), waxed kraft, polyester film, cellulose acetate, and polyethylene. Wraps that produced the best results, when used with the petroleum preservative were: creped cellulose acetate-kraft, polyester film, and polyethylene.

Forest Products Laboratory, United States Department of Agriculture. TR 53-334.

CALCULATING CUSHION THICKNESS BY ANALYSIS OF STRESS-STRAIN CURVES. R. E. Jones and D. L. Hunsicker. AF 33(038) 51-4065. January 1954. PB 111520. Order from OTS $4.75.

A method for designing package cushioning to protect an article against shock is developed from physical and mathematical concepts. Design curves for calculating cushion thickness are included for many currently available cushioning materials. Methods for selecting the proper cushion are discussed.

TR 53-132.

THE DESIGN OF RECLAIMED LATEX FOAM PACKAGE CUSHIONING. THE THIRD OF A SERIES OF REPORTS ON PACKAGE CUSHIONING. Roger B. Orensteen, 1st Lt, USAF. RDO No. 618-11. January 1954. PB 111518. Order from OTS $0.75.

A method of designing reclaimed latex foam cushioning from static data is presented. Design curves are provided for selecting density, thickness, and cushion area for economical cushion design. Recommended procedures are discussed for designing to optimum density, stress, and ratio/stress energy in a given situation.

Forest Products Laboratory. TR 53-216.

EVALUATION OF CONTAINER-GRADE PAPER-OVERLAID VENEER. E. H. Clarke. AF 33(038)51-4065. January 1954. PB 111519. Order from OTS $1.50.

By conducting over 1,500 tests on specimens of paper-overlaid veneer and container-grade plywood along with tests of 165 containers, information was obtained that indicates a correlation exists between some of the mechanical properties of paper-overlaid veneer materials and the expected rough-handling performance of fully cleated domestic boxes using such material for panels. The results also indicate that all of the tested 19 different paper and veneer combinations, each varying from the other by differences in core, overlays, or manufacturing process, are suitable alternates for 3/20-inch (Groups I and II) container-grade plywood in panel boxes. Some combinations appeared to be suitable as alternates for thicker plywood.

Forest Products Laboratory. United States Department of Agriculture. TR 53-38, Part 18.

PACKAGING REQUIREMENTS FOR BEARINGS. PART 18-THE EFFECT OF RELATIVE HUMIDITY AT TIME OF PACKAGING ON THE CONDITION OF BEARINGS AFTER A CYCLIC EXPOSURE (SERIES G-G). R. A. Aubey. AF 18(600)-103. January 1954. PB 122337. Order from LC Mi $2.70, Ph $4.80.

Bearings were cleaned and packaged in metal cans at each of 6 humidity conditions ranging from 30 to 97 percent relative humidity. The intimate wraps, 1 hygroscopic (polyethylene to kraft) and the other nonhygroscopic (a polyester film), 3 preservative compounds, and 1 grease were conditioned for 18 hours prior to packaging at the humidity in which they were to be used.

The packages were exposed to a 3-week cycle plus 60-days exposure at 160°F. and 92 percent relative humidity.

The condition of the bearings after exposure became increasingly worse as the humidity condition at time of packaging increased. When packaged at relative humidities of 50 percent and higher, bearings wrapped with the nonhygroscopic intimate wrap were in better condition than those with the hygroscopic wrap.

One of the petroleum-base preservative materials, when packaged with a polyester film as an intimate wrap, gave complete protection through all of the humidity conditions.

Forest Products Laboratory, United States Department of Agriculture. TR 53-38, Part 14.

PACKAGING REQUIREMENTS FOR BEARINGS. PART 14-

PERFORMANCE OF TIN CANS CONTAINING ANTI-FRICTION BEARINGS AND COMMERCIAL MOTOR OILS (SERIES E-E). A. A. Mohaupt. AF 18(600)-103. January 1954. ASTIA Document No. AD 27592. PB 122333. Order from LC Mi $2.40, Ph $4.80.

To determine if commercial motor oils could be used for packaging antifriction bearings, tin cans containing separable-type ball bearings were filled or half filled with oil and sealed. Three motor oils, each of various viscosities, and an inhibited oil were used for these tests. The oils were applied at either 230°F. or room temperature. The cans were procoated with an olive-drab paint, and the packages were subjected to a 3-week cyclic exposure plus 60 days storage at 160°F. and 92 percent relative humidity.

Bearings packaged in an inhibited oil were in better condition after exposure than those packaged in motor oils. The different viscosities of the motor oils, filling or partially filling the tin cans, and the temperature at which the oil was applied to the bearings at the time of packaging had little or no effect on the condition of the bearings after exposure.

Forest Products Laboratory, United States Department of Agriculture. TR 53-38, Part 8. PACKAGING REQUIREMENTS FOR BEARINGS. PART 8 - PERFORMANCE OF TIN CANS CONTAINING ANTI-FRICTION BEARINGS AND VARIOUS TYPES OF OILS (SERIES P AND Q). L. O. Anderson, M. A. Taras and R. K. Stern. AF 18(600)-103. January 1954. PB 122327. Order from LC Mi $2.40, Ph $4.80.

An investigation to determine the effectiveness of procoated tin cans for packaging antifriction bearings with various oils was conducted by the U. S. Forest Products Laboratory.

Three types of oils together with three bearing types were investigated. Cans, each containing 1 bearing and a maximum amount of oil, were sealed and subjected to a 3-week cyclic exposure test plus 30, 60, or 120 days of storage at 160°F, and 92 percent relative humidity. In addition, cans containing 2 bearings and a maximum amount of oil were subjected to the 3-week cyclic exposure and 120 days of exposure.

No corrosion was evident on any of the bearings in packages subjected to the 30-day exposure period. Bearings packaged in tin cans with the inhibited oils were in excellent condition after storage for 120 days. Corrosion of a minor extent was present on 50 percent of the bearings packaged in tin cans with the uninhibited oil that were exposed to a 3-week cycle plus 120 days of storage.

No significant difference was evident when 2 bearings, instead of the customary 1, were packaged in a can.

Forest Products Laboratory, United States Department of Agriculture. TR 53-38, Part 1. PACKAGING REQUIREMENTS FOR BEARINGS. PART 1 - STUDY OF GREASEPROOF BARRIER REQUIREMENTS FOR AN-G-25 GREASE. R. S. Kurtenacker. AF 18(600)-103. January 1954. PB 122320. Order from LC Mi $3.00, Ph $7.80.

Various greaseproof barrier materials were used in this investigation. Twenty-three materials were tested for compliance with Specification JAN-B-121, Barrier-Materials, Greaseproof. Four materials were used as conforming wraps for Method IA-1 packs of bearings, prepared with preservative, Specification AN-C-124, Compound, Soft-Film, Corrosion-Preventive or with grease, Specification AN-G-25, Grease, Low Temperature Aircraft, Lubricating and subjected to exposure tests.

Of the twenty-three materials tested, three met all requirements of Specification JAN-B-121, and five met all requirements except for high acidity.

Tests of Method IA-1 packs indicate that the greaseproofness, as determined by Specification JAN -B-121, is not an indication of satisfactory use with grease, Specification AN-G-25. None of the barrier materials prevented dispersion of the oil base from the grease. Metal surfaces were stained and pitted in all packs subjected to the 8-day cycle plus 90 days' storage, except when material SN was used as the barrier material.

In tests of Method IA-8 packs, the metal-foil pouches appeared to prevent dispersion of the oil base from grease, Specification AN-G-25. Bearings in these pouches were free from stain, while bearings in pouches made of barrier materials, SN, PF, or PL were stained or pitted.

Method IA-8 packs appear suitable for packaging bearings prepared with AN-G-25 grease. It is recommended, however, that additional work be done and that the make-up of the AN-G-25 grease be investigated.

TR 53-38, Part 13. PACKAGING REQUIREMENTS FOR BEARINGS. PART 13 - PERFORMANCE OF TIN CANS CONTAINING ANTI-FRICTION BEARINGS SUBJECTED TO CYCLIC EXPOSURE AND ROUGH HANDLING (SERIES F-F). R. K. Stern. AF 18(600)-103. January 1954. ASTIA Document No. AD 27642. PB 122332. Order from LC Mi $2.40, Ph $3.30.

The principal objective of this study was to determine the performance of procoated tin cans containing antifriction bearings and inner packaging of varied cushioning value when the packages were subjected to rough handling during 3 weeks of cyclic exposure. The can packages in groups of 48 were packed by 2 methods in fiberboard inner boxes with outer boxes of wood.

No significant mechanical damage to the cans was caused by rough handling. All bearings were unaffected by cyclic exposure and rough handling. Immediately after testing, however, the procoating on all cans contained a large number of water-filled blisters, most of which disappeared after a few days of exposure to 30 percent relative humidity. This blistered condition could be a principal factor in causing severe corrosion to the cans in the case of a longer period of exposure.

Forest Products Laboratory. TR 53-38, Part 12. PACKAGING REQUIREMENTS FOR BEARINGS. PART 12 - THE EFFECT OF FIVE CLEANING PROCEDURES ON ANTIFRICTION BEARINGS PACKAGED WITH MIL-G-3278 GREASES. R. A. Aubey. AF 18(600)-103. February 1954. PB 122331. Order from LC Mi $2.70, Ph $4.80.

A study was made by the Forest Products Laboratory to determine the effectiveness of five cleaning methods in removing contamination and preventing recontamination of antifriction bearings packaged with greases that complied with Specification MIL-G-3278. The grease-coated specimens and a limited number of controls without grease were exposed in two manners. A group of pouch packages was exposed to a 3-week cyclic exposure plus 60 days of storage at 160°F. and 92 percent relative humidity, and another group of grease-coated rollers was placed on open slings and subjected to an exposure of 80°F and 80 percent relative humidity for 88 days.

The results indicated that cleaning Method C-5 of Specification MIL-P-116A, consisting of the use of a specification fingerprint remover followed by a solvent wash, is the most satisfactory. Other cleaning methods were

accompanied by staining and discoloration of the specimens. None of the methods prevented recontamination. A thin film of oil did not prevent recontamination.

Forest Products Laboratory. TR 53-38, Part 10.
PACKAGING REQUIREMENTS FOR BEARINGS. PART 10 - PERFORMANCE OF PLASTIC-COATED (EXTRUDED) ALUMINUM-FOIL BARRIERS AS INTIMATE WRAPS FOR PACKAGING BEARING PARTS COATED WITH SYNTHETIC GREASE. M. A. Taras and J. O. Boxby. AF 18(600)-103. February 1954. PB 122 329. Order from LC Mi $2.70, Ph $4.80.

This investigation was conducted primarily to determine the performance of aluminum foil with an extruded plastic coating as an intimate wrap in packaging bearing parts coated with MIL-G-3278 synthetic grease. Three hundred pouch-type packages containing the outer rings of ball bearings were prepared with various combinations of grease, coated and uncoated intimate wraps, and methods of closure of the wrap. The test packages were exposed to a 3-week cyclic exposure with temperature extremes of -65°F. and 160°F., followed by 60 days of storage at 160°F. and 92 percent relative humidity.

Severe delamination occurred with polyethylene-foil wraps regardless of the kind of grease or method of closure used. Comparable packages using nylon extruded to foil were not delaminated. Delamination, varying in degrees from none to moderate, was evident in polyethylene-tissue-foil wraps with all greases.

Pinholing (fracturing) of the aluminum foil usually accompanied the use of a fold method of sealing the intimate wrap and was not evident in wraps closed with a heat seal. There was no significant difference, however, in the amount and degree of corrosion in comparable packs that could be attributed to method of closure. Coated foil wraps were usually more effective when the coated surface of the wrap was positioned in contact with the grease-coated bearing part.

Although all four synthetic greases used in this experiment complied with Specification MIL-G-3278, a wide range of effectiveness in corrosion prevention existed among them.

Forest Products Laboratory. TR 53-38, Part 7.
PACKAGING REQUIREMENTS FOR BEARINGS. PART 7 - PLASTIC-COATED, ALUMINUM-FOIL BARRIERS AS INTIMATE WRAPS FOR PACKAGING BEARINGS. M. A. Taras. AF 18(600)-103. February 1954. PB 122326. Order from LC Mi $2.70, Ph $4.80.

A study of the performance of plastic-coated aluminum foils as intimate wraps for packaging bearing parts coated with grease or preservative was conducted by the Forest Products Laboratory. Eight different intimate wrap materials, 2 greases, and 2 methods of sealing the intimate wraps were investigated. The specimens were enclosed in water-vaporproof pouches and exposed to a 3-week cycle of temperatures ranging from -65° to 160°F. Following the 3-week exposure period, 1 group of packages was stored for 30 days and another group for 60 days at 160°F. and 92 percent relative humidity.

Results of this study indicated that the heat-seal method of closing laminated plastic-coated aluminum-foil barrier intimate wraps was more effective in regard to corrosion prevention than the drugstore method of closure. The only barrier that offered complete protection to the synthetic grease-coated bearings was a heat-sealed polyethylene-foil-polyethylene-scrim intimate wrap. Vinyl-foil, cellulose acetate-foil and plain aluminum foil gave the poorest performance with the synthetic grease. The cellulose acetate-foil, vinyl-foil, and polyethylene-kraft gave the poorest performance with the AN-C-124 preservative. The plastic coatings on the foil reduced the amount of fracturing of the aluminum foil but did not reduce it sufficiently to keep the grease or preservative within the confines of the intimate wrap. The synthetic grease had a tendency to deteriorate all the plastic laminated foils severely except the polystyrene-foil combinations.

Forest Products Laboratory. TR 53-38, Part 11.
PACKAGING REQUIREMENTS FOR BEARINGS. PART 11-PERFORMANCE OF PROCOATED BLACK IRON CAN PACKAGES CONTAINING VOLATILE CORROSION INHIBITOR AND ANTIFRICTION BEARINGS (SERIES T). V. C. Setterholm. AF 18(600)-103. February 1954. PB 122 330. Order from LC Mi $2.40, Ph $3.30.

This study was initiated to investigate the performance of volatile corrosion inhibitors as protection for two types of anti-friction bearings in procoated black iron cans.

Interior protection was provided by dicyclohexylammonium nitrite-impregnated paper or crystals. Procoated black iron cans were used to contain antifriction bearings with steel or brass retainers.

The packages were exposed at a 3-week cyclic exposure with temperature extremes of -65° to 160°F. One group of bearings with brass retainers were examined after this exposure. Following the 3-week cyclic exposure period, both brass and steel antifriction bearing packages were stored for 60 days at 160°F. and 92 percent relative humidity.

Although the dicyclohexylammonium nitrite was successful in minimizing corrosion of ferrous parts, the tendency of crystals to undergo sublimation resulted in a dense formation of crystals on all surfaces of the bearing. The presence of these crystals on functional surfaces acted to freeze the rolling parts.

The use of MIL-C-10506 procoating material as a protective coating on the black iron cans was unsatisfactory in this test.

Forest Products Laboratory, United States Department of Agriculture. TR 53-38, Part 5.
PACKAGING REQUIREMENTS FOR BEARINGS. PART 5-THE PERFORMANCE OF VARIOUS TYPES OF WATER-VAPORPROOF BARRIERS CONTAINING GREASE-COATED PARTS. M. A. Taras. AF 18(600)-103. February 1954. PB 122324. Order from LC Mi $2.40, Ph $4.80.

An investigation of the performance of various water-vaporproof barrier materials used as pouches for packaging grease- or preservative-coated parts was conducted by the Forest Products Laboratory.

Five different pouch materials, a synthetic grease, and a petroleum-base preservative were investigated. Twelve packages were made with each pouch material, 6 with synthetic grease and 6 with petroleum-base preservative. Each preservative- or grease-coated ring was wrapped in a greaseproof barrier of polyethylene-kraft. These packages were exposed to a 3-week cycle of temperatures ranging from -65° to 160°F. Following the 3-week exposure period, 1 group of 30 packages was stored for 30 days, and the other group of 30 packages was stored for 60 days at 160°F. and 92 percent relative humidity.

Results indicated that pouches of barrier Nos. 60 and 62 do not deteriorate upon exposure with either the grease or preservative used in this experiment. Pouches of barrier No. 63 when used with synthetic grease gave good protection to inner rings of bearings, despite the fact that the pouches deteriorated severely and water droplets were found on the grease. Although two of the barrier materials used for pouches did not deteriorate upon exposure, none of the barrier materials gave good protection to the rings when used with preservative. The exposure condition

of 160°F. is too severe for this preservative, since it flows readily from the ferrous parts at this temperature.

Forest Products Laboratory. TR 53-38, Part 3. PACKAGING REQUIREMENTS FOR BEARINGS. PART 3 - COMPATIBILITY OF MIL-G-3278 SYNTHETIC GREASE WITH VARIOUS INTIMATE WRAPS AND AN-B-20 POUCHES (SERIES A). M. A. Taras. AF 18(600)-103. March 1954. PB 122 322. Order from LC Mi $2.70, Ph $4.80.

One hundred and twenty-six pouch packages containing the outer ring of standard ball bearings assemblies were subjected to a cyclic exposure to evaluate the compatibility of pouch materials, synthetic greases, and intimate wraps. Various combinations of 2 pouches, 2 greases, and 6 intimate wraps were used in the test packs.

Based on the results of the exposure test, the AN-B-20 pouch materials were about equally effective in protecting the rings from moisture vapor and other agents of corrosion. Both MIL-G-3278 greases caused pouch deterioration; however, one of the greases appeared to be less deleterious than the other. None of the combinations of pouches or intimate wraps prevented hardening of the greases. The polyster film was the only intimate wrap material that produced consistently good results with both greases and pouch materials.

Forest Products Laboratory. TR 53-38, Part 2. PACKAGING REQUIREMENTS FOR BEARINGS. PART 2 - INVESTIGATION OF ALUMINUM-FOIL BARRIERS FOR PACKAGING. Dr. V. W. Meloche and D. R. Johnson. AF 18(600)-103. March 1954. PB 122 321. Order from LC Mi $3.00, Ph $6.30.

An investigation of the use of aluminum foil as an intimate wrap for bearings coated with either petrolatum-base preservatives or synthetic greases and packaged in water-vaporproof pouches was conducted by Forest Products Laboratory. The aluminum foil in each pack was either oil-free foil, foil with rolling oil employed in manufacturing the foil, or foil with an excess of rolling oil. The exposure consisted of either three 5-day cyclic tests at temperatures ranging from -65° to 160°F. or this cyclic test plus 60 days at 160°F. and 92 percent relative humidity.

Results indicate that the aluminum foil is subject to pinholding and breaking at the folds, does not retain the grease or preservative with the type of wrap employed, and does not prevent changes in the composition of the synthetic grease. The results also indicate that it is more desirable to employ oil-free foil than the foil with rolling oil present. As a basis for comparison, a polyethylene-coated kraft barrier gave better performance than did the aluminum foil with the synthetic grease.

Forest Products Laboratory. TR 53-38, Part 9. PACKAGING REQUIREMENTS FOR BEARINGS. PART 9 - PERFORMANCE OF FOUR MIL-G-3278 GREASES WITH VARIOUS GREASE-PROOF BARRIERS IN ANTI-FRICTION BEARING-POUCH PACKAGES (SERIES S). Michael A. Taras. AF 18(600)-103. March 1954. PB 122328. Order from LC Mi $2.40, Ph $3.30.

This experiment to determine the effectiveness of four MIL-G-3278 greases used in combination with various types of intimate wraps in pouch-type packages in preventing corrosion of bearing parts was conducted by the Forest Products Laboratory. Four greases and six intimate wraps were investigated. The specimens were enclosed in water-vaporproof pouches and exposed to a 3-week cycle of temperatures ranging from -65° to 160°F. Following the 3 weeks of cyclic exposure, one group of packages was stored for 30 days and another group of packages was stored for 60 days at 160°F. and 92 percent relative humidity.

Results of this study showed that various synthetic greases that comply with Specification MIL-G 3278 perform differently at the same storage conditions. It was likely that variation in grease composition was responsible for this reaction.

Nylon (3 mils) was the only barrier that performed satisfactorily with each of the greases.

Forest Products Laboratory. TR 53-38, Part 6. PACKAGING REQUIREMENTS FOR BEARINGS. PART 6- THE PERFORMANCE OF THREE TYPES OF SEALS FOR VARIOUS INTIMATE WRAPS WITH MIL-G-3278 GREASES (SERIES E). M. A. Taras. AF 18(600)-103. March 1954. PB 122 325. Order from LC Mi $2.70, Ph $4.80.

A study of the performance of three methods of closing various intimate wraps containing ferrous bearing parts coated with grease complying with Specification MIL-G-3278 was conducted by the Forest Products Laboratory. The three methods for closing the intimate wraps included the drugstore fold, heat sealing, and the use of an aluminium band. All specimens were enclosed in water-vaporproof pouches complying with Specification MIL-B-131A and were exposed to a 3-week cycle of temperatures ranging from -65° to 160°F. Following the cyclic exposure, the specimens were stored for 60 days at 160°F. and 92 percent relative humidity.

Results indicate that the effectiveness of the three sealing methods investigated is largely dependent upon the compatibility of the intimate wraps and the greases used. When both greases are considered, the three sealing methods appear to be equally effective. Corrosion when present, however, was more severe in packages using the drugstore fold method.

In general, the greases used hardened upon exposure. Nylon, however, was the only intimate wrap that prevented any of the greases from hardening. No reason for this reaction was apparent. The data also indicate that one of the greases was less deleterious to the vinyl coating of the pouch material used in this test than the other.

Plax Corporation. TR 53-133, Part 1. INVESTIGATION OF THE SHELF LIFE OF LIQUIDS IN POLYETHYLENE BOTTLES. Jules Pinsky, A. R. Nielsen and James H. Parliman. AF 33(616)-112. June 1954. PB 111546. Order from OTS $3.25.

The shelf life of 80 especially selected liquids and solids in blown Plax 4 ounce polyethylene bottles was investigated at temperatures from 32-165°F. Permeability factors for these materials are presented as well as packageability suitability. The suitability of packaging these 80 materials in blown polyethylene bottles of capacities from 32 ounces to 13 gallons is tabulated. Physical effects on the polyethylene and chemical changes in the materials are described. The effect of liquid and vapor phases, as well as the molecular weight of the chemicals, on permeability is discussed.

Forest Products Laboratory, United States Department of Agriculture. TR 53-38, Part 4. PACKAGING REQUIREMENTS FOR BEARINGS. PART 4- THE PERFORMANCE OF FIVE METHODS OF PACKAGING BEARINGS IN METAL CONTAINERS (SERIES I). L. O. Anderson. AF 18(600)-103. June 1954. PB 122323. Order from LC Mi $2.40, Ph $3.30.

This investigation was conducted to determine the relative performance of five methods of packaging bearings as prepared by a roller bearing manufacturer. These

cans were subjected to a 3-week cycle with temperature extremes of -65°F. and 160°F., followed by 60 days of exposure at 160°F. and 92 percent relative humidity.

The best results were produced by a preservative of petrolatum and an intimate wrap of neutral kraft waxed paper. The use of silica gel with this combination of preservative and wrap was also satisfactory. The use of AN-C-124, type II preservative resulted in corroded bearings. The use of MIL-G-10924 grease over a petrolatum film with an aluminum wrap did not give fully satisfactory results. The package using dicyclohexylammonium nitrite as a volatile corrosion inhibitor was also unsatisfactory under the conditions of this test.

These results are not fully conclusive, since all of the test specimens were prepared at the adverse condition of 90 percent relative humidity.

Forest Products Laboratory. WADC TR 53-38 (Part 15).
PACKAGING REQUIREMENTS FOR BEARINGS. PART 15-THE PERFORMANCE OF VOLATILE CORROSION INHIBITORS FOR PACKAGING ANTIFRICTION BEARINGS (SERIES N-N). A. A. Mohaupt, R. K. Stern. AF 18(600)-103. July 1954. PB 111650. Order from OTS $0.75.

An experiment was conducted at the Forest Products Laboratory to evaluate the use of volatile corrosion inhibitors (VCI) for packaging antifriction bearings. Bearings with or without an oil film were sealed in tin cans or flexible, water vaporproof pouches with 1 of 6 VCI materials and subjected to a 3-week cyclic exposure plus 60 days of storage at 120° or 160°F. and 92 percent relative humidity.

Results indicate that some VCI materials can be used advantageously for packaging antifriction bearings.

Under all test conditions, the inhibitor consisting of a kraft paper impregnated with sodium nitrite, urea, and monoethanolamine benzoate gave the best results. A kraft paper impregnated with an amine salt also protected the bearings from corrosion under all conditions, except when used in a pouch exposed to 160°F.

Kraft papers coated or impregnated with VCI compounds produced more desirable results for packaging antifriction bearings than inhibitors in crystalline or powdered form.

In general, the use of an oil film in conjunction with a VCI material did not reduce corrosion under the conditions of this test.

Better results were obtained with certain inhibitors in tin cans than with the same inhibitors in pouch packages.

Forest Products Laboratory. WADC TR 53-38 (Part 16).
PACKAGING REQUIREMENTS FOR BEARINGS PART 16. THE EFFECT OF EXPOSURE ON GLASS PACKS CONTAINING STEEL ROLLERS IN VARIOUS PRESERVATIVES OR GREASES AND IN ATMOSPHERES OF AIR OR NITROGEN (SERIES V). V. W. Meloche, A. Frisque. AF 18(600)-103. February 1955. PB 122335. Order from LC Mi $2.40, Ph $3.30.

This experiment was conducted primarily to determine the compatibility of various materials used for packaging antifriction bearings under controlled conditions. Test specimens consisted of sealed glass tubes each containing a steel roller, a synthetic grease or petroleum-base preservative with various amounts of moisture, and an atmosphere of air or nitrogen. These specimens were subjected to a 3-week cycle with temperatures ranging from -65° to 160°F. followed by 60 days of storage at 160°F.

When no water was added to the test specimens, practically no corrosion or stain occurred on the rollers with any of the packaging combinations. When water was added, stain and corrosion were found on the rollers. This test also demonstrated that air or oxygen was a protective passivating agent. In general, the degree of stain and corrosion on rollers in the oxygen containing atmosphere was less than the stain and corrosion on rollers in a nitrogen atmosphere. There was a definite variation in the performance of the various preservatives and greases.

University of Connecticut and Plax Corporation. WADC TR 53-133 Part 2 (Corrected Abstract).
THEORETICAL INVESTIGATION OF THE MECHANISM OF TRANSFER OF MATERIALS THROUGH POLYETHYLENE. Dr. Henry A. Bent, Jules Pinsky. AF 33(616)-112. August 1955. PB 121194. Order from OTS $2.50.

The effect of temperature on the permeability P has been found to be accurately represented by the two parameter equation

$$P = P_o e^{-E_p / RT}$$

in which E_p and P_o are constants for the particular permeant polymer system. These parameters are calculated from carefully constructed log P vs. 1/T plots.

The size, shape, and polarity of the penetrant molecule are factors influencing E_p and for substances no more polar than acetone the expression

$$E_p = 0.0348V + 0.75\ V/L + 2.4\ H$$

reproduces the data to within about 0.5 kcal/mol. where polyethylene is the permeable film.

It is established that for four homologous series that

$$\log P_o = mEp + b$$

where m and b are constants peculiar to each series.

Master plots for interpolating homologous materials and extrapolating for temperature changes are included.

Plax Corporation. WADC TR 53-133, Pt 3.
INVESTIGATION OF THE SHELF LIFE OF LIQUIDS IN POLYETHYLENE BOTTLES. Part 3-Investigation of the Effects of Molecular Weight, Chain Branching, and Irradiation on Polyethylene With Regard to Shelf Life in Bottles. Jules Pinsky, A. R. Nielsen, J. H. Parliman. AF 33(616)-112. September 1956. ASTIA Document No. AD 97334. PB 121696.

Part I of this contract was concerned with the Shelf Life of Liquids and Solids in blown Plax 4-ounce bottles made from Bakelite's DE-2400. For this investigation, Part 3, the effects of higher molecular weight (Bakelite's De-2450), side chain branching (Alathon 10, Alathon S-1439, and Alathon S-1447), irradiation by electron bombardment (High Voltage Irradiated De-2450) were studied. The changes in permeability factors occasioned by the above variations are presented and are tabulated in comparison with the previous results on Bakelite's DE-2400. It can be seen from this report that, while there are considerable differences in permeability, the packageability status is not generally changed.

University of Connecticut. Plax Corporation. WADC TR 53-133 Part 3, Sup. 1.
INVESTIGATION OF THE SHELF LIFE OF LIQUIDS IN POLYETHYLENE BOTTLES Part 3, Sup. 1. Theoretical Investigation of the Effects of Molecular Weight, Side Change Branching, and Irradiation on the Mechanism of Transfer of Materials Through Polyethylene. Dr. Henry A. Bent, Jules Pinsky. AF 33(616)-112. September 1956. ASTIA Document No. AD 97335. PB 121 696s.

In this study P-Factors and absorption tests

reported in Part 3 are analyzed and discussed, together with some data on aqueous solutions from Part 1. The theory of mass transfer through polyethylene is extended, particularly with regard to the $\log P_o$ vs E_p plot, and the results applied to recent data on carbon tetrachloride.

It is found that chain branching and irradiation increase swelling coefficients and diffusion constants at low temperatures, but decrease them at very high temperatures. The importance of placing room temperature P-Factors in this perspective is stressed. It is suggested that activated diffusion through polyethylene induces structural changes in the polymer not unlike local melting. Thermodynamical reasoning also indicates that frequency and P-Factors increase with increasing rigidity and mass of penetrant. In both respects carbon tetrachloride shows this increase with polyethylene.

Forest Products Laboratory. WADC TN 55-216. THE PERFORMANCE OF STRAPPED, NAILED, WOODEN BOXES HAVING SIDES, TOPS, AND BOTTOMS OF 7/16- OR 9/16-INCH RESAWN LUMBER. R. K. Stern, D. O. Yandle. PO(33-600)-53-4023. September 1956. ASTIA Document No. AD 97252.

The strength of 7/16- and 9/16-inch resawn lumber when used as box sides, tops, and bottoms was compared by rough-handling tests of 112 strapped, nailed, wooden boxes.

Boxes made of 9/16-inch lumber were much more resistant to rough-handling than boxes made of 7/16-inch lumber.

WADC TR 56-342.
THE THEORY AND OPERATION OF A DYNAMIC TESTER FOR EVALUATING PACKAGE CUSHIONING MATERIAL. A. D. Klingenberg, 1/Lt. September 1956. ASTIA Document No. AD 97327. PB 121692.

The principles of cushioning design are presented. The characteristics of several possible types of dynamic cushion testers are discussed, and the reasons shown for selection of the free fall platform tester. A description of the tester and the dynamic cushion test procedure is given. A method for data reduction of test results is shown. The reliability of the cushion tester is proven by statistical techniques. Sample drop test results are given. It is shown how deflections caused by dropping a series of weights can be computed on the basis of a measured deflection occurring in the first drop.

Forest Products Laboratory. WADC TN 55-706. EVALUATION OF FOUR DESIGNS OF EXTERIOR SHIPPING CONTAINER FOR HUMAN REMAINS TRANSFER CASE. T. B. Heebink. PO(33-600)-53-4023. November 1956. ASTIA Document No. AD 110525. PB 121894.

Four boxes, each of different design, were evaluated as exterior containers for the human remains transfer case. The evaluation tests consisted of a high-moisture-content conditioning test and the following rough-handling tests: superimposed load; handle; flat, edgewise- and cornerwise-drop; and pendulum impact.

Three of the boxes swelled objectionably when the moisture content was increased from 7-1/2 to 19 percent. The same three boxes showed either racking distortion or residual deflection in the rough-handling tests. The fourth box, of cleated plywood construction, was superior in every way. It showed very little weakness in the moisture test or the rough-handling tests, was 19 percent lighter, and is possibly cheaper to build.

WADC TR 56-464.
MEASURING FIELD HANDLING AND TRANSPORTATION CONDITIONS. Donald S. Martin, 1/Lt. November 1956. ASTIA Document No. AD 110586. PB 121866.

The proof that an item is properly packaged for shipment is its ability to withstand a drop test. The height from which drop tests are conducted is arbitrarily chosen and may overstate the facts. A more realistic height of drop could be chosen if recording instruments were concealed inside ordinary shipping containers and shipped, where the instrument would record the impacts due to rough handling of packages by handling personnel.

The bottleneck to an instrumented shipping program is instrumentation. Reported, herein, is the development of an instrument of sufficient accuracy to measure rough handling in terms of inches of drop height. Cost of the instrument is $200 each.

The statistical shipping plan for using the instrument and a method of relating the data obtained to container design is also discussed.

Forest Products Laboratory. WADC TN 55-407. MAGNETIC SHIELDING OF MAGNETRON TUBES AND MAGNETS FOR SHIPMENT BY AIR. A. W. Voss. PO (33-600)53-4023. January 1957. ASTIA Document No. AD 110750.

A method is presented for shielding magnets and magnetron tubes containing magnets to protect sensitive aircraft instruments from being affected by stray fields of the magnets. A survey showed that current military practice is to stow magnets at least 7 feet from the compass-sensing device. To meet the criterion established by WADC, the stray fields have to be limited to not more than 5.25 milligauss at a distance of 7 feet from the magnets. Various methods and materials were tested, and it was found that shields of low-carbon steel would confine the stray fields and prevent adverse effects on aircraft instruments. A method of forming these shields is described and illustrated. The minimum weight and number of layers required for various tubes and magnets is tabulated, and methods are presented by which the shielding required for similar tubes and magnets can be established.

WADC TR 57-51.
EVALUATION OF THE TWIST LOCK CONTAINER. Warren D. Hypes. March 1957. ASTIA Document No. 118166. PB 131033.

The twist-lock type rigid metal container qualifying as a Method IId unit protection in accordance with Specification MIL-P-116 was evaluated. The Forest Products Laboratory conducted a field service test, the results of which indicate acceptance of the twist-lock container by the majority of depot personnel. The field service test also concluded that the quality of the gasket, in conjunction with the closing technique, was the most important factor in obtaining a satisfactory seal. The Laboratory tests, conducted by the Forest Products Laboratory and Wright Air Development Center, concluded that the twist-lock container will meet the performance requirements for Method IId unit protection.

Forest Products Laboratory. WADC TR 56-357. EVALUATION OF AUTO-NAIL FOR FABRICATING THE PANELS OF CLEATED PAPER-OVERLAID-VENEER BOXES. Alvin A. Mohaupt. PO (33-600)-53-4023. April 1957. ASTIA Document No. AD 118234. PB 131 263.

Tests were conducted to determine the suitability of Auto-nails, as compared to regular nails, for fastening cleats to paper-overlaid veneer for use as box

panels. Eight paper-overlaid-veneers were included in lateral-displacement tests from which it was found that approximately three Auto-Nails equaled the lateral resistance afforded by two regular nails. Drop tests of cleated, paper-overlaid-veneer boxes with panels made of Auto-Nails with 2- and 3-inch spacings showed that these boxes performed as well as or better than cleated-plywood boxes fabricated with regular nails spaced 3 inches apart.

Forest Products Laboratory. WADC TR 56-647. EVALUATION OF FIVE TYPES OF CONTAINERS FOR JETTISONABLE FIBERGLASS FUEL TANKS. T. B. Heebink. PO(33-600)-53-4023. May 1957. ASTIA Document No. AD 118345. PB 131 187.

Five types of containers for jettisonable fiberglass fuel tanks were evaluated and compared to determine whether savings could be made in tare weight and cubic displacement over the container now in use for aluminum tanks. Present requirements are for a Specification MIL-C-4349 type of crate. Each of the containers was tested for resistance to rough handling by the edgewise drop, cornerwise drop, and pendulum impact tests. One type of open crate and a cleated plywood box performed best under rough handling. The cleated plywood box had the least tare weight and cube. The open crate would be satisfactory, and could be used if it is found necessary to have unlimited ventilation in storage to minimize corrosion of metal fittings.

Forest Products Laboratory. WADC TR 56-660. AN EVALUATION OF POPCORN AS A CUSHIONING MATERIAL. R. K. Stern. DO 33(600)53-4023. May 1957. ASTIA Document No. 118314. PB 131 162.

This investigation had the purpose of determining if popcorn could be used as a cushioning material. Conventional cushioning materials such as bound hair and cellulosic wadding were used as a basis for comparison. Stress-strain curves were established for popcorn, bound hair, and cellulosic wadding under several conditions of temperature and humidity. Repeated loading tests on these materials were also performed. The mold resistance and the effect of high humidity on treated and untreated popcorn were established. Combustibility of the materials was also investigated. The results showed that popcorn is not a good cushioning material. It is stiff in comparison to other cushioning materials and has very little ability to recover after compression. The extreme hygroscopicity of popcorn is undesirable since it shrinks appreciably in high humidity. Its only conceivable use is indicated to be as a blocking material in moisture-vapor-proof packages.

Forest Products Laboratory. WADC TR 56-539. TESTS TO EVALUATE AND COMPARE VARIOUS OPEN CRATE SPECIFICATIONS FOR AIRCRAFT SURFACES. Thomas B. Heebink. PO 33(600)53-4023. July 1957. ASTIA Document No. AD 130873. PB 131376.

Thirteen open crates for aircraft surfaces were tested to evaluate the construction details of various open crate specifications. The results of these tests suggested various advantageous modifications which were incorporated into a final design and tested. The result was that the three crates built according to the final modified design were superior generally in all respects including performance, tare weight, cubic displacement, and cost.

The final design was incorporated into the proposed specification, being prepared by the Forest Products Laboratory for WADC entitled "Crates, Wood (For Lightweight, Bulky Airframe Items)."

Forest Products Laboratory. WADC TR 56-547. INVESTIGATION OF SHOCK WAVES DEVELOPED DURING DYNAMIC TESTS OF CUSHIONING MATERIALS. Vern N. Smiley. AF 33(600)53-4023. August 1957. ASTIA Document No. AD 131019. PB 131429.

Certain irregularities of acceleration-time pulses were recorded during dynamic compression tests of package cushioning materials for determining their energy absorption characteristics. These irregularities were theorized to have been caused by shock wave oscillations in the material. The validity of this theory was checked by deriving mathematical expressions based upon shock wave theory and then comparing theoretically expected results with actual test results. Sufficiently close agreement was found to prove that the irregularities were caused by shock waves. A discussion of the significance of shock waves in relation to cushioning performance is also made.

Forest Products Laboratory. WADC TN 55-328. EVALUATION OF CONTAINER-GRADE PAPER-OVERLAID VENEER PANEL BOXES FOR OVERSEAS USE. Edward H. Clarke. AF 33(600)53-4023. September 1957. ASTIA Document No. AD 142004. PB 131553.

Laboratory tests were conducted in an effort to investigate the suitability of some commercially available paper-overlaid veneers for use in overseas-type, cleated panel boxes. Rough-handling tests and diagonal-compression tests involving some 220 overseas-type boxes provided information regarding the relative performance of various paper-overlaid veneers and two currently acceptable panel materials, V3s fiberboard and container-grade plywood.

After immersion in water for 24 hours, the cleated plywood boxes were superior to V3s fiberboard and paper-overlaid veneer in both rough handling and resistance to diagonal distortion or twisting. Only a few paper-overlaid veneer materials approached the rough-handling performance of V3s fiberboard panel boxes, except when intermediate cleats were used as required for the fiberboard. Then the paper-overlaid veneer boxes exhibited a rough-handling performance equal to that of the V3s fiberboard boxes. The paper-overlaid veneer boxes resisted compressive forces better than the cleated fiberboard boxes.

WADC TR 57-682. THE DEVELOPMENT OF A NON-ADHERING CHEMICALLY FOAMED-IN-PLACE POLYURETHANE CUSHIONING MATERIAL FOR PACKAGING PURPOSES. Sidney Childers, Sidney Allinikov. January 1958. ASTIA Document No. AD 142282. PB 131 665.

A chemical process for the production of a non-adhering chemically foamed-in-place polyurethane foam is presented. An evaluation of several organic fatty acids as a release agent for chemical foamed polyurethane cushioning is included. Fatty acids such as stearic acid in proper concentration were found to impart excellent release properties to foam after foaming on bare metal and glass surfaces. Further benefits from the release agent are shown to be improvement of the stress-strain properties and the ability to vary density and hence cushioning properties by the use of sufficient amounts of fatty acid. The effect of the release agent on the foaming process and the finished cushion is discussed. Graphs and a comparison analysis of the dynamic and static cushion properties are presented for a polyurethane cushion with and without a release agent.

WADC TR 56-342 Sup 1.
THE THEORY AND OPERATION OF A DYNAMIC TESTER FOR EVALUATING PACKAGE CUSHIONING MATERIAL. Steward M. Krakover, 1/Lt. May 1958. ASTIA Document No. AD 151195. PB 121692s.

The purpose of the ASTM dynamic cushion testing program is described and data from the laboratories which participated in this program are presented. On the analysis of the platform type tester together with a discussion of the significance of the elasticity of impact of containers is covered. Several redesigns of the dropping carriage are shown. The desirability of using the platform tester to gather design data is discussed. A discussion is included of the peak reading voltmeter system and a portable spring calibrator.

Forest Products Laboratory. WADC TR 58-223.
THE CUSHION FACTOR-STRESS CURVE AND ITS VALUE FOR CLASSIFYING AND SELECTING PACKAGE CUSHIONING MATERIALS. R. K. Stern. PO (33-600)-53-4023. November 1958. ASTIA Document No. AD 205071. PB 151547. Order from OTS $1.50.

"Cushion factor-stress" curves have been considered for use in solving package cushioning problems and for classifying package cushioning materials. To be most suitable for these uses, a single essentially constant curve would express the compressive efficiency of any specific material regardless of its dimensions or loading rate. The dynamic compression test data derived from this work showed that the curve for at least one important material varies considerably with thickness, and the use of this type of curve is considered to be impractical. The report contains data for the compressive efficiency and variability of four common kinds of package cushioning materials differing in thickness, density, and manufacturing origin.

United Engineers, Incorporated. WADC TR 57-541.
DEVELOPMENT OF PLASTIC FOAMING EQUIPMENT. George W. Pearce, Sidney Allinikov. AF 33(600)-32235. November 1958. ASTIA Document No. AD 205550.

The design, development, fabrication and evaluation of an operable foaming equipment system for producing mechanically foamed plastic materials is described. Engineering research into the selection of plastic materials suitable for use with the equipment is discussed. Polyethylene was selected as the initial material to be mechanically foamed. A 12 lb. per cu. ft. density foam was achieved with polyethylene. This proved to be the lowest density foam produced with the equipment at present state of development. Polyvinyl chloride, styrene, and nylon were foamed in densities of 18, 15, and 17 lbs. per cu. ft. respectively.

Densities of all the foams produced were higher than the 1.5 to 6 lbs. per cu. ft. target range considered acceptable for cushioning purposes. However, it was successfully demonstrated that plastic materials can be foamed by mechanical means.

Fairchild Guided Missiles Division. WADC TR 58-268.
FIBERGLASS CONTAINERS FOR J-57 ENGINE. Harry E. Keller. AF 01(608)832. February 1959. ASTIA Document No. AD 209911. PB 144 991. Order from LC Mi $4.50, Ph $12.30.

Twenty fiberglass containers were manufactured for the Air Force to serve as shipping containers for the J-57 jet aircraft engine. The application of fiberglass for this purpose has proved successful with the exception of a liner sealing problem. The fiberglass container assembly weighs approximately 50% less than its metal counterpart, and testing has proved it to be rugged enough to withstand the rough handling it may receive in shipment.

The container consists of two shells, upper and lower, with the basic construction utilizing plies of bidirectional roving fabric impregnated with polyester resin. The sealing liner is made of rigid vinyl, thermoplastically drawn under vacuum into the shells. Provisions have been incorporated for fork lift, cable hoisting, rollover and stacking of the containers three high while in storage.

WADC TR 58-639.
INVESTIGATION OF DESIGN CRITERIA FOR CUSHIONING MATERIALS. Stewart M. Krakover, 1/Lt, Albert Olevitch. March 1959. ASTIA Document No. AD 201227. PB 151720. Order from OTS $1.00.

This report discusses the properties of cushioning materials significant for purposes of package cushioning design. Acceleration-static stress curves are the required dynamic property. It is shown that the minimum points of such curves reflect the most efficient operation of the cushioning materials. The other properties of significance are the static stress-strain and creep characteristics.

Examples of the application of the significant properties in designing package cushioning are presented. The economic aspects of utilizing cushioning materials are dependent on knowledge of these properties and a formula relating cost to the significant properties is presented.

The report also presents some design data on several different materials commonly available and the procedures for securing the design data. The report essentially is a text on the properties of cushioning materials and the design of package cushioning.

WADC TR 58-601.
MATERIALS, TECHNIQUES, AND ECONOMICS OF FOAMED-IN-PLACE POLYURETHANE CUSHIONING FOR PACKAGING. Sidney Childers. April 1959. ASTIA Document No. AD 211913. PB 151831. Order from OTS $1.00.

This report describes an investigation into various aspects of foaming-in-place molded shapes of polyurethane cushioning material. The chemistry of the foam reactions and the foam systems are described. The aging characteristics of the materials and the effect on metals and humidity indicators of the vapors given off during foaming are discussed. Various facts about foaming methods and equipment are presented.

Of the foaming techniques investigated, it was found that completely premolding or a combination of premolding and foaming into the container was the best technique. It was also found that one can create molds by utilization of simple methods and low cost, reusable materials, such as hot melt plastics.

The cost of creating the molded shape is shown to be less, in most cases, than the present practice of procuring molded curled hair shapes.

TR 4596.
SPECTROGRAPHIC METHOD FOR THE DETERMINATION OF IRON IN MAGNESIUM ALLOYS. C. B. Pittinger, Jr. December 1940.

TR 4617, Addendum 1.
DEVELOPMENT OF GLOSS SPECIFICATION FOR CAMOUFLAGE FINISHES. ADDENDUM 1 -- MEASUREMENT OF SPECULAR GLOSS. Harry Schecter. January 1943.

TR 5014.
LABORATORY MANUAL FOR ORGANIC SYNTHETIC PLASTICS. E. J. Fischer (1937), Leah K. Hendriksen, Translator. September 1943.

TR 4995.
DEVELOPMENT OF SPECTROPHOTOMETRIC PROCEDURES. E. H. Young. August 1943.

TR 5628.
METHOD OF SPECTROGRAPHIC ANALYSIS FOR BERYLLIUM IN MAGNESIUM ALLOYS. Robert E. Brocklehurst. August 1947.

The Engineering Experimental Station, University of Illinois. TR 6389.
SOME STATIC FATIGUE, AND CREEP TESTS OF A GLASS FABRIC LAMINATED WITH A POLYESTER RESIN. William N. Findley, Will J. Worley. W33-038-ac-21089. April 1951.

Armour Research Foundation, Illinois Institute of Technology. TR 6383.
RESEARCH ON THE ELEMENT SILICON AND SILICON ALLOYS. W. R. Johnson, M. Hansen. AF 33(038)-8517. June 1951.

TR 6520.
AN EVALUATION OF X-RAY METHODS FOR THE QUANTITATIVE DETERMINATION OF TETRAETHYL-LEAD IN AVIATION GASOLINE. Jack T. Humphries. November 1951.

A brief description of the X-ray absorption and fluorescence methods for the determination of tetraethyllead is given. Samples of aviation gasoline, Specification MIL-F-5572, were tested with each method and the results are included in the report. These results indicate that the fluorescence method is applicable to the need of the Air Force for a rapid, accurate analysis. The absorption method does not give the desired accuracy.

The methods of sample preparation and the arrangement of equipment are described.

It is found that an X-ray fluorescence analysis for tetraethyllead content requires approximately eight minutes, a feature which makes the X-ray analysis much more desirable than the usual chemical method.

TR 6610.
COMPOSITIONAL ANALYSIS OF AN-G-25 TYPE GREASES. Nora E. Funkhouser, George Rappaport. E. O. No. R601-299. December 1951.

TR 6387.
STUDIES OF THE EFFECTS OF AVIATION FUEL COMPONENTS ON THE ACCURACY OF THE KARL FISCHER ELECTROMETRIC TITRATION METHOD FOR DETERMINING THE WATER CONTENT OF FUELS. Robert W. Altman, Richard W. Sneed, James C. Mosteller. March 1952.

A study was conducted to determine the effect of various concentrations of fuel components such as tetraethyl lead, aromatics, olefins, mercaptans and oxidation inhibitors on the accuracy of results obtained when determining water content of fuels by the Karl Fischer Electrometric Titration Method. Of the compounds investigated, only mercaptans appear to give any appreciable interference in water determination by this method.

TR 6390.
MODIFIED METHOD FOR DETERMINING OXIDATION STABILITY OF AVIATION FUELS. Robert W. Altman, Richard W. Sneed and James C. Mosteller. August 1952.

A modified test procedure developed in the Materials Laboratory, Directorate of Research, WADC, for the determination of the accelerated aging characteristics of aviation fuels is described. This test method appears particularly applicable to jet propulsion fuels forming large quantities of insoluble gum during oxidation. The major feature of the new procedure is the equipment modification which makes it possible to oxidize a sample and, without subsequent handling, determine the residue formed. This is considered an improvement over the present ASTM and Army-Navy gum methods. The results obtained with a wide variety of aviation fuels indicate the modified method to be generally as suitable as the current methods, but superior for fuels forming insoluble residues during accelerated aging.

University of Texas. TR 52-246.
DEVELOPMENT OF COMPOSITE SPECTROPHOTOMETRIC PROCEDURES FOR THE ANALYSIS OF LOW-ALLOY STEELS AND OF ALUMINUM AND ITS ALLOYS. Stanley H. Simonsen and Gilbert H. Ayres. AF 33(038)-23168. November 1952. PB 111 287. Order from OTS $1.50.

Procedures have been developed for the spectrophotometric analysis of the constituents of low-alloy steels and of aluminum and its alloys, using a single sample to give a composite solution in which most of the elements are determined. In general, the conventional spectrophotometric methods for the various elements have been employed, modified as necessary for the use of the single composite solution technique. The methods have been tested thoroughly, in the hands of several different operators, by applying them to a variety of standard samples; the results of these tests are tabulated in the report.

Detailed procedures, with explanatory notes, to serve as a practical laboratory manual are given for the spectrophotometric analysis of low-alloy steels for chromium, copper, manganese, molybdenum, nickel, phosphorus, silicon, and vanadium, and for the analysis of aluminum and its alloys for chromium, copper, iron, manganese, molybdenum, nickel, titanium, and vanadium. The molybdisilicic acid method for silicon in the aluminum samples did not give satisfactory results; the conventional gravimetric method for this determination is recommended.

TR 6381.
AN EXAMINATION OF SOME AIRCRAFT GREASES WITH

THE ELECTRON MICROSCOPE. Jules I. Wittebort and Bernard Rubin. RDO 601-299. November 1952.

TR 6385.
THE EFFECT OF STORAGE ON THE PHYSICAL CHARACTERISTICS OF GREASE. Robert J. Burger and Bernard Rubin. March 1953.

The effect of prolonged storage under accelerated conditions on three types of specification greases is described. Periodic tests using standard methods including penetration, oxidation stability, and low temperature torque were performed on samples of the greases packed both in cans and in bearings. Data thus obtained over the 24 months storage period at 130°F are tabulated and trends are presented graphically. The Specification AN-G-15 types of greases were found to harden appreciably in storage while two of the Specification AN-G-3 greases studied showed marked increases in consistency after being stored only a few months. Other Specification AN-G-3 greases, as well as one Specification AN-G-25 grease tested, appeared to be affected only slightly by extended storage under the conditions described.

TR 53-44.
ELECTRON MICROSCOPE AND ELECTRON DIFFRACTION STUDY OF OPTICALLY CONDUCTING COATINGS ON GLASS AND ACRYLIC PLASTIC. Stanley A. Szawlewicz. March 1953. PB 118 266. Order from LC Mi $2.70, Ph $4.80.

Electron microscopy and electron diffraction were used to determine the microstructure of several types of commercially produced transparent conducting coatings on glass. This work was conducted as part of a program to evaluate various conducting coatings intended for use on aircraft windshields in preventing fog, frost, or ice and to aid in the development of conducting coatings which could be applied to transparent aircraft canopies for the purpose of dissipating precipitation static charges.

Several experimental transparent conducting coatings produced under Air Force contract were also studied. This included the study of the latest development in the preparation of a conducting coating for acrylic plastic canopies which consists essentially of the mechanical application of fine graphite to the plastic surface.

TR 6386.
A STUDY OF THE RUST PREVENTIVE PROPERTIES OF GREASES. Bernard Rubin and Robert J. Burger. March 1953.

As a result of numerous complaints of corrosion of wheel bearings of aircraft in semi-storage conditions, an investigation into the rust preventive properties of wheel bearing greases was initiated. Concurrent studies were carried out in the laboratory and in service aircraft.

A laboratory procedure using ball bearings as specimens exposed to 95% relative humidity at 100°F was found to grade greases with respect to corrosion preventive properties under high humidity conditions. Furthermore, the test showed correlation with service results.

The laboratory investigation was enlarged to include many different types of greases. These included petroleum, various types of diesters, and silicone greases. Factors influencing the degree of corrosion resistance were investigated, including the effect of rust and oxidation inhibitors.

The service investigation was conducted on a group of operational aircraft over a period of one year. During this time, samples of the wheel bearing greases were removed periodically and records were maintained showing flying time and weather data.

The results of the investigation indicate that certain properties such as high alkalinity and some water-emulsifiability with a degree of water resistance are useful in controlling the degree of corrosion protection which a grease affords, to a coated bearing surface. Some difference in corrosion protective qualities was noted among the different classes of diester greases studied. Fingerprint suppression appears to have a role in protection against corrosion of steel surfaces under service conditions.

Hellige, Inc. TR 53-112.
DEVELOPMENT OF GLASS COLOR STANDARDS. H. C. Hellige. AF 33(600)-6395. April 1953. PB 118 898. Order from LC Mi $1.80, Ph $1.80.

The development of glass color standards to be used as visual comparison standards in adjusting the concentration of four dye additives in aircraft fuels is discussed. A practical and satisfactory color comparison vessel which would permit easy differentiation between maximum and minimum allowable colors of each of four dye colors was selected. A reproducible source of illumination for comparison of the glass standards to the dyed fuels was determined. A master set of glass standards exactly matching fresh fuel samples was made. A few months later a second set of glass standards was completed on the basis of the master set; however, after this time the contractor's set of fuels had changed color slightly, and counter samples retained by the Air Force had changed greatly. No positive mathematical relationship can be applied to comparing the glass color standards to the original spectrophotometric curves of the dyed fuels due to differences in viewing depth, the effect of a discrimination increasing color filter, and differences in illumination. It is concluded, however, that on the basis of visual comparison, the color standards are satisfactory for the intended purpose.

National Research Corporation. TR 53-465.
PREPARATION OF HOMOGENEOUS OXYGEN BEARING IODIDE TITANIUM SPECIMENS. Rodger D. Veneklasen. AF 33(616)-2041. October 1953. ASTIA Document No. AD 62770.

Five 5-pound ingots have been alloyed, melted and fabricated to produce the homogeneous oxygen bearing iodide purity titanium specified in the contract. Chemical analyses for interstitial elements, carbon, nitrogen and tungsten have been made on the ingots. Oxygen and hydrogen analyses have been made by vacuum fusion analytical methods developed in the laboratories of National Research Corporation. Analytical results are close to intended oxygen compositions and show the individual samples to be uniform and homogeneous within the limits of accuracy of the analytical methods. These analyses further show the interstitial elements to be lower than the limits set in the contract. The oxygen alloys were to have nominal compositions of 0.1, 0.2, 0.5, 1.0% with one additional ingot serving as a blank.

It is felt that the procedure used for obtaining uniform oxygen content is reliable and accurate and it will be applied to other research work now in progress and requiring uniform additions of oxygen to Ti and Ti alloys. It is anticipated that further research applications will be forthcoming requiring controlled additions of oxygen, nitrogen or carbon to metals requiring cold mold melting.

University of Cincinnati. TR 53-308, Part 1.
DENSITY AND VISCOSITY OF MOLTEN MATERIALS. PART 1 - DENSITY OF SODIUM AND SODIUM HYDROXIDE. M. Nishibayashi. AF 33(616)-9. November 1953. PB 127 927. Order from LC Mi $3.60, Ph $9.30.

A densitometer to be used for liquids at temperatures up to ca. 1000°C in an inert atmosphere was constructed from grade "A" nickel. In the construction of this densitometer, the hydrostatic weighing method of Kohlrausch was selected as the method most suitable for the determination of density under the conditions imposed. This method utilizes the buoyancy principle of Archimedes. A plummet is suspended from one arm of an analytical balance and is immersed in the sample contained in the densitometer tube. The apparent loss in weight of the plummet upon immersion is measured by a chainomatic balance. The entire apparatus is gas-tight; observations and manipulations of the balance are made through gas-tight seals. With this apparatus the density of a material can be measured from its melting point to ca. 1000°C using a single sample of the material.

The density of sodium and sodium hydroxide was measured from 254° to 860°C and from 447° to 736°C respectively. The following equations relating density to temperature were derived from the experimental data:

$$d_{Na} = (0.9378 \pm 0.0086) - (0.000268 \pm 0.000018)(t - 97.5^{\circ})$$

$$d_{NaOH} = (1.753 \pm 0.002) - (0.000565 \pm 0.000008)(t - 318.4^{\circ})$$

Using these equations, the average deviations between the observed and the calculated values of density were found to be 0.93% for sodium and 0.23% for sodium hydroxide.

WADC TR 54-393.
PRELIMINARY STUDY OF GLASS AND PLASTIC FILMS USING ELECTRON DIFFRACTION AND ELECTRON MICROSCOPY. L. Harold Bullis. December 1954. ASTIA Document No. AD 62120. PB 133 562. Order from LC Mi $2.70, Ph $4.80.

Thin films of acrylic plastic prepared from solutions of plastic in amyl acetate are shown by electron diffraction to be highly crystalline, and the crystallinity is shown to change with the aging of the films. The crystallinity of the films and the observed effects of aging are explained on the basis of the "micelle" theory commonly accepted as the explanation of somewhat similar effects in rubber. Electron micrographs of the films tend to substantiate this view.

Thin films of silica are shown by electron diffraction to be devoid of crystalline structure and to exhibit no observable changes upon aging.

University of Cincinnati. WADC TR 53-308, Pt. 2. DENSITY AND VISCOSITY OF MOLTEN MATERIALS. PART 2 SUMMARY OF VISCOSITY MEASUREMENTS JANUARY 1952 to AUGUST 1953. J. W. Sausville. AF 33 (616)-52-9. February 1955. PB 127 926. Order from LC Mi $3.60, Ph $9.30.

University of Michigan. WADC TR 53-455. APPLICATION OF THE POLAROGRAPH TO ANALYSIS OF TITANIUM-BASE ALLOYS. Philip J. Elving, Charles L. Rulfs, Joseph L. Lagowski, Julian Lakritz, Robert J. Meyer. AF 33(600)-379. March 1955. ASTIA Document No. AD 66445. PB 130 392. Order from LC Mi $3.30, Ph $7.80.

The possible application of the polarographic techniques of direct concentration measurement and amperometric titration to the analysis of titanium-base alloys has been investigated. In view of the limitations of time, budget, and personnel, attention was focused on the development of analytical methods for only three or four elements. A procedure is described for the simultaneous determination of chromium and estimation of iron in titanium-base materials. The procedure involves a general technique for the dissolution of titanium-rich materials in hydrofluoric acid followed by separation of most of the titanium as insoluble potassium hexafluorotitanate. In the specific procedure described, excess fluoride is then largely removed by an evaporation in the presence of sulfuric acid and the chromium is oxidized to chromate by persulfate. Treatment with ammonia and cyanide serves simultaneously to complex the iron as the soluble ferricyanide and precipitate the residual titanium as hydroxide. The solution is then made strongly alkaline and polarographed. Chromium results are excellent, while those for iron are only fair. A rapid method for the polarographic determination of iron in titanium-base alloys has been developed, using a modified citrate procedure. Hydrofluoric acid is used to dissolve the sample. Aluminum ion is then introduced to complex the fluoride ion and potassium citrate is added as a supporting electrolyte. The polarogram is recorded and the current, after a blank correction has been applied, is compared to a previously determined standardization curve. It is possible to obtain accurate results for iron contents ranging between 0.2 and 5.0%. A complete determination can be completed in 15 to 20 minutes. A rapid and precise amperometric titration method for the determination of vanadium in titanium-base alloys has been developed. Sulfuric acid or hydrofluoric acid followed by sulfuric acid evaporation is used to dissolve the sample. Standard ferrous solution is the titrant and a rotating microplatinum electrode is used as the indicator electrode. Specifically, titanium, iron, manganese, chromium, copper, magnesium, aluminum, and moderate amounts of molybdenum and tungsten do not interfere. In addition, a large number of other elements do not interfere.

WADC TR 54-601.
A MODIFIED ADIABATIC CALORIMETER. Myron W. Belaga, David Coddington, Hyman Marcus. March 1955.

A modified adiabatic type calorimeter, capable of measuring the heat capacities of organic liquids (natural and synthetic) in the temperature range of 100-500°F, was designed, built, and calibrated.

In this calorimeter a fixed rate of power is supplied to the sample, and a bath which surrounds the sample container is maintained at the same temperature in order to prevent heat losses from the sample. The heat capacity of an unknown material may be calculated from measurements of the time rate of temperature rise, heat input, and mass of the sample.

The calorimeter has been designed for rapid, simple sample changing.

The calorimeter was calibrated with materials of known heat capacity (water, castor oil, and linseed oil), and the following relation between the calorimeter constant and temperature was obtained:

$$K(t) = 0.256 - 3.706 \times 10^{-5}t + 6.839 \times 10^{-7}t^2$$

University of Cincinnati. WADC TR 53-308, Pt. 3. DENSITY AND VISCOSITY OF MOLTEN MATERIALS. PART 3 SUMMARY OF VISCOSITY MEASUREMENTS SEPTEMBER 1953 TO AUGUST 1954. Curtis C. Beusman. May 1955. PB 111 945. Order from OTS $0.75.

A viscometer was fabricated which uses a torsionally vibrating piezoelectric crystal attached to a metal sensing element. Electronic components for measuring the resonant frequency and the resonant resistance of the damped crystal did not yield adequate sensitivity of reproducibility with the instrument. A servomechanism designed to stabilize the instrument was not successful in remedying the difficulties encountered.

Spectrochemical Laboratories, Inc.
WADC TR 55-108.
THE EMISSION SPECTROGRAPHIC ANALYSES OF TITANIUM METALS AND ALLOYS. . M. DuBois Arno Tuteur and Lt. J. L. Mahan, 1/Lt USAF. AF 33(616)-2315. August 1955. PB 121 016. Order from OTS $2.50.

A Quantometric Point to Plane technique has been developed which is capable of giving accurate results, with reasonable speed, on solid samples of titanium alloys.

A vapor Injection Solution technique has been developed which will give excellent precision, accuracy and speed.

A statistical evaluation has been conducted on the following spectrographic techniques:

1. Fusion Coating
2. Electrode Dried Solution
3. Porous Cup
4. Rotating Disc
5. Lucite Cup
6. Vapor Injection
7. Point to Plane

Duplicate wet-chemical analyses have been performed on all samples submitted, for each of the elements studied, and these results have been used to determine the accuracies of the various quantometric procedures.

WADC TR 55-421.
COMPARATIVE METHODS OF FLUORINE ANALYSIS. Gordon D. Oshesky, 1/Lt. USAF. February 1956. PB 121 190. Order from OTS $1.75.

There are many methods for analyzing the fluorine content of fluoro-organic compounds. These methods involve varying degrees of convenience with regard to simplicity of apparatus, time required, and the skill of the operator and can be applied to a wide range of compounds. An attempt has been made in this project to determine which were best suited for compounds containing 10 to 70% fluorine. The methods discussed in this report include oxidation, reduction, gravimetric, and titrametric procedures. These methods are by no means all of the proposed methods, but a few of the more commonly used ones.

Several of the methods described here are applicable in the determination of fluorine in fluorinated organics. Depending upon the operator's preference, any one of the recommended methods can be used with good accuracy. Three of the most promising methods are: (1) combustion of the sample in moist oxygen, converting the resulting fluorine to silicone tetrafluoride and hydrolyzing the latter to hydrofluoric acid before titrating with sodium hydroxide; (2) fusion of the sample in a platinum crucible with sodium potassium carbonate, dissolving this residue, precipitating as lead chlorofluoride and determining the fluorine gravimetrically or decomposing the lead chlorofluoride precipitate and titrating the librated chlorine; (3) fusion of the sample with metallic potassium and titrating the fluorine content with standard thorium nitrate solution.

Of the three suggested methods the procedure in which fluorine is titrated with standard thorium nitrate appears to be the most convenient, more suitable, and the most accurate.

WADC TR 54-268.
QUANTITATIVE ANALYSIS OF ELASTOMERS THROUGH THE INFRARED SPECTRA OF THEIR PYROLYZATES. Freeman F. Bentley. February 1956. PB 121 115. Order from OTS $0.75.

A semi-quantitative method is described for determining polymer blends and co-polymer ratios from the infrared spectra of their pyrolysates (distillates). The method was established through the analysis of phenolic resin blends in Buna N rubber, and by determining the percentage acrylonitrile in butadine acrylonitrile co-polymers.

The method described here permits the semi-quantitative analysis of Buna N phenolic resin blends, which because of their physical state, cannot be analyzed by conventional methods. It also makes it possible to estimate the amount of non-combustible materials in these polymers. The method is based on the pyrolyzate technique combined with standard infrared quantitative procedures.

Emerson & Ewming, Inc. WADC TR 55-255.
PRODUCTION OF METALLIZED FIBERS FOR DIPOLE CHAFF. C. L. Emerson, Jr. and George E. Niles. AF 33(616)-2604. April 1956.

The development of a metallized nylon fiber suitable for dipole chaff has been completed. The finished fiber is approximately 2.9 mils in diameter. It consists of a nylon monofilament 2.8 mils in diameter coated first with silver and then with nylon deposited from solvent. The electrical resistance is continuously less than 15 ohms/inch. Abrasion resistance and non-clumping is good. The cost of the packaged product is less than 0.01¢/foot of length.

WADC TN 56-196.
GAMMA DOSIMETRY PROPERTIES OF LINEAR POLYETHYLENE BY INFRARED ANALYSIS. James H. Tillotson, 1/Lt and Nora E. Srp. May 1956.

Changes in the molecular structure of long chain polymers by infrared analysis gives an indication of damage to material after its exposure to nuclear radiations. The intensity changes of the 5.8 and 11.0 micron infrared absorption bands in gamma irradiated Marlex 50, a high density linear polyethylene, are analyzed and presented as a measure of relative gamma dosimetry. All radiation exposures were conducted at the National Reactor Test Station, Idaho Falls, Idaho. The infrared absorption spectra were recorded at the Wright Air Development Center using a Baird double beam recording spectrophotometer. The graphs of optical density versus gamma dosage are believed to represent in a realistic manner the gamma dosimetry properties of the polyethylene used in this investigation.

Armour Research Foundation. WADC TR 55-495 Part I.
MEASUREMENTS OF THERMAL PROPERTIES. I. B. Fieldhouse, J. C. Hedge, J. I. Lang, A. N. Takata and T. E. Waterman. AF 33(616)-2903. September 1956. ASTIA Document No. AD 110404.

The objective of this program was the measurement of the high temperature thermal properties of materials. The materials investigated were electrolytic copper, grade A nickel, arc melted unalloyed molybdenum, graphite type 3474D (Speer Carbon Company), graphite type GBE (National Carbon Company), and sintered tantalum. The thermal conductivity, specific heat, and linear coefficient of thermal expansion were measured from 1000°F to 3000° F or the melting point of the material, whichever was lower. All measurements were made in the Heat Transfer laboratory of the Propulsion and Fluid Mechanics Research Department, Armour Research Foundation of Illinois Institute of Technology. Both the experimental measurements and the results of the conversion of these measurements to the desired physical properties are given.

WADC TR 56-263.
POLAROGRAPHIC DETERMINATION OF GALLIUM IN ALUMINUM AND ALUMINUM ALLOYS. George W. Latimer, Jr., 2/Lt and Charles D. Houston. September 1956. ASTIA Document No. AD 97280. PB 121720.

Polarographic determinations of gallium have been limited to concentrations less than 3×10^{-5} molar because of the interference of the hydrogen wave. By adapting a method utilized by Williard and Dean to determine aluminum, the interference caused by the evolution of hydrogen is eliminated and gallium concentrations up to 15×10^{-5} molar can easily be determined. The gallium is separated from aluminum and most interfering ions by ethyl ether extraction from 5M HCl. The ether is evaporated and the gallium is complexed with Eriochrome Violet. This complex has a polarographic wave beginning at -.3v which is directly proportional to the concentration of gallium.

WADC TR 56-299.
SIMULTANEOUS POLAROGRAPHIC DETERMINATION OF CADMIUM AND TIN. George W. Latimer, Jr., 2/Lt C. D. Houston, and Keith E. Eubank. September 1956. ASTIA Document No. AD 97281. PB 121 685.

The development of new and improved platings for copper sheets has led to a need for improved methods of analyzing the plated material. The simultaneous polarographic determination of cadmium and tin provides a rapid method of analyzing these coatings. Analysis of synthetic standards by this method shows that there are very few interfering substances and that this procedure is applicable to a wide range of concentrations with reasonable accuracy.

Armour Research Foundation.
WADC TR 56-222 Pt I.
DETERMINATION OF EMISSIVITY AND REFLECTIVITY DATA ON AIRCRAFT STRUCTURAL MATERIALS. Part I. Techniques for Measurement of Total Normal Emissivity and Reflectivity with Some Data on Copper and Nickel. Howard T. Betz, O. H. Olson, B. D. Schurin and James C. Morris. AF 33(616)-3002. October 1956. ASTIA Document No. AD 110458. PB 121 817.

Equipment has been designed, constructed, and calibrated for the measurement of total normal emissivity in the range -300°F to +3000°F. The procedure consists in comparing the total normal radiance of a sample to that of a comparison blackbody, the ratio of the signals being taken as the emissivity. A thermistor detector is used in conjunction with a folded optical system to record the radiation which is chopped at 16 cps. Measurements of the total normal emissivity for a limited number of samples have been completed. The normal spectral emissivity measurements are obtained by an optical pyrometer method, which relates the true temperature of a sample to its apparent brightness temperature. A Leeds and Northrup disappearing filament pyrometer is used to make these measurements.

An integrating sphere reflectometer is used to measure spectral reflectivity from 0.3 microns to 3.0 microns. This utilizes a Perkin-Elmer monochromator, a comparison type integrating sphere and a PbS cell detector. Spectral reflectivity data can be combined with solar spectral energy data for any location to obtain the solar absorptivity at that point.

WADC TR 56-452.
DESIGN OF A HIGH-INTENSITY GAMMA IRRADIATION FACILITY. Marvin C. Atkins, 1/Lt. December 1956. ASTIA Document No. AD 110542. PB 131 160. Order from OTS $1.50.

Construction plans have been prepared for a gamma irradiation facility capable of handling 100,000 curies of cobalt-60. In a facility built according to these plans, the radioactive source would be stored in a water well located in a 12 x 12 foot irradiation room. The source would rest on a platform in the well and be lifted up into the irradiation room for all experiments. The facility would be built underground with almost all shielding being provided by the natural earth. Normal laboratory utilities are provided in the plans, with valves and switches located on a control panel in an adjacent building. A closed circuit television system would be installed for viewing of experiments with the source in the operating position. Experimental apparatus would be equipped with suitable instruments so that general purpose manipulators would not be required. Six construction drawings are included in this report.

Tracerlab, Incorporated. WADC TR 56-433.
NEUTRON INDUCED RADIOACTIVITIES IN IF-6 IN-FLIGHT FOOD PACKETS. Robert P. Epple. AF 33(616)-2616. Jan. 1957. ASTIA Document No. AD 110733. PB 121994.

A study has been made of the radioactivities induced by neutron irradiation of IF-6 In-Flight food packets. The results show that an integrated exposure of 4×10^{14} nvt was produced.

Na-24 and K-42 were produced in amounts which are nearly 1000 times the accepted tolerances for these isotopes in the human body.

WADC TR 56-434.
INFRARED SPECTRA OF CYCLOPENTAMETHYLENEDIALKYL SILANES IN THE 2-35 MICRON REGION. Gordon D. Oshesky and Freeman F. Bentley. February 1957. ASTIA Document No. AD 118046. PB 131064. Order from OTS $0.75.

The difficulty in establishing the presence of a ring in organic molecules by chemical means has promoted interest in identifying cyclic structures by their infrared spectra. Saturated cyclic systems have characteristic absorption frequencies arising from the bond stretching and valence angle modes which occur in the fundamental region and the out-of-plane and torsional bending modes which occur below 20 microns. In this study the infrared spectra of a series of cyclic silanes has been correlated with their molecular structura and empircal assignments made for the absorption frequencies characteristic of the hetercyclic ring.

The infrared absorption spectra of 21 cyclopentamethylenedialkyl-silanes has been obtained in the liquid state over the range 2-35 microns using NaCl and CsBr prisms. The series begins with cyclopentamethylenedisilane and ends with cyclopentamethylenedioctyldecylsilane. Absorption bands at 10.93 to 11.00 microns and 20.20 to 20.85 microns in the spectra of these compounds are tentatively assigned to vibrations arising from the heterocyclic ring.

Absorption bands attributed to the alkyl-silicon linkage and the spectra of these molecules in the long wavelength region are also discussed. All the observed absorption bands are given in a table showing their position and relative intensities.

WADC TR 55-302.
ACCESSORY EQUIPMENT AND PROCEDURES FOR USE OF A 1500 CURIE COBALT-60 GAMMA-RAY SOURCE. Marvin C. Atkins, 1/Lt. April 1957.

The Materials Laboratory is using a 1500 curie cobalt-60 source to investigate the effects of gamma radiation on materials. The source is a cylinder, 1.78 in I.D. x 2.31 in. O.D. x 13.5 in. long. It is shielded by a lead container which is mounted on a table behind a concrete block wall. A rotating-beam hoist is used to change

samples. Other items of accessory equipment include specialised plugs for the container, additional sample canisters, and a cooling system.

Measurements have been made of scattered radiation from the source. A standard operating procedure for use of the source has been established. This procedure has significantly reduced the probability of personnel exposure to harmful radiation.

WADC TR 57-38.
HEAT CAPACITIES OF SYNTHETIC ENGINE OILS AND LUBRICANTS. Hyman Marcus. April 1957. ASTIA Document No. AD 118225. PB 131 038.

A modified adiabatic type calorimeter, previously designed, built and calibrated (Rev. Sci. Instr 27 p 948, 1956), was used in the measurement of the heat capacities. The variation of the heat capacity as a function of temperature was determined for eleven synthetic fluids conforming to Military Specification MIL-L-7808 and the data are presented graphically.

The expressions for the heat capacity were derived from a least squares statistical analysis of the time-temperature data and an attempt was made to develop a heat capacity envelope of all the data obtained and calculate a median curve. No comparison or analysis was made of each set of individual data nor were any set of data identified with individual products measured.

Technical Operations, Incorporated. WADC TR 56-440.
INVESTIGATION OF ISOTOPES FOR AIRCRAFT RADIOGRAPHY. Eric T. Clarke. AF 33(616)-3042. April 1957. ASTIA Document No. AD 118244.

To determine if isotope radiography will permit inspection of aircraft components, without the need for disassembly, a study was made of the properties of the isotopes Eu-155, Ce-144, Am-241, Ir-192, and Tm-170 in the radiography of light metal structures. On the basis of ease of production, available intensity, and ability to produce sufficient radiographic contrast in typical aircraft materials, the isotopes Ir-192 and Tm-170 were selected and their characteristics in stainless steel, titanium, aluminum alloy, and magnesium were measured. Radiographic exposure charts and sensitivity data were established for these materials in conjunction with suitable photographic materials, including lead and fluorescent screens. Data are given on production of these isotopes in nuclear reactors.

Results of radiography of actual aircraft are shown which indicate that internal structural detail can be readily made visible, and some types of flaws can be detected, without the need for excessive exposure times. Reproductions of actual radiographs permit comparisons with X-rays.

WADC TR 56-548.
MEASUREMENT OF RADON GAS IN CYLINDRICAL IONIZATION CHAMBERS. Edward A. Burke, 1/Lt. May 1957. ASTIA Document No. AD 118062. PB 131 186. Order from OTS $0.50.

Three of the fundamental problems encountered in the measurement of radon gas using cylindrical ionization chambers are discussed and solutions presented. These are: variation of ionization current with short half-life radon daughters, effect of dielectric absorption, and calibration of the ionization chamber.

WADC TR 56-500.
SPECTROGRAPHIC ANALYSES OF SOLID TITANIUM. Jack A. Winstead, 1/Lt. May 1957. ASTIA Document No. AD 118273. PB 131 185.

The purpose of this work was to establish satisfactory excitation conditions for spectrographic analysis of solid titanium and to obtain data from analysis by the established technique.

This work was done in cooperation with the Task Force on Spectrographic Analysis by Direct Metal Excitation. Watertown Arsenal supplied each member of the Task Force seven samples (two specimens each from different locations).

Each specimen was cut in half and then nine analyses were made on each individual piece, totalling thirty-six analyses for each alloy sample. These analyses were made and data included in this report.

WADC TR 56-657.
THEORETICAL ANALYSIS OF THE RESPONSE OF A PROTON RECOIL TYPE NEUTRON DETECTOR. Sven R. Hartmann, 1/Lt. May 1957. ASTIA Document No. AD 118272. PB 131 204.

A calculation is made to determine the number of recoil protons produced per unit area, by a neutron flux of arbitrary energy, in an infinite hydrogenous slab which reaches but does not pass through an infinite detector slab located immediately behind a hydrogenous slab. A general case is considered for an isotropic neutron flux incident upon the above system in which an arbitrary thickness of some proton absorbing material separates the hydrogenous and detector slabs. A discussion of the results is given indicating how one can control the energy and the energy interval over which the system is sensitive to neutrons. A final calculation is made to determine the response of the system to a collimated neutron flux for an arbitrary orientation of the hydrogenous-detector system. This is done so that the effect of a nonisotropic flux can be determined.

Schwarzkopf Microanalytical Laboratory Materials Laboratory. WADC TR 56-19 Pt I.
ANALYSIS OF FLUORINATED ORGANO-METALLICS. Part I - Determination of Fluorine and Silicon in Organic Fluoro-Silicon Compounds. Otto Schwarzkopf, Rosemarie Heinlein and Nora E. Srp. AF 33(616)-3067. June 1957. ASTIA Document No. AD 130855. PB 131 359.

Development of analytical methods to determine fluorine and silicon in organic fluoro-silicon compounds was the object of this project. A method having a precision of $\pm 1\%$ for fluorine and $\pm 0.7\%$ for silicon was developed. The test sample is reacted with potassium metal in a pure nickel bomb heated to 650°C for two hours. The fusion mixture is dissolved in water and after filtering, fluorine is precipitated as PbClF and filtered. The filtrate containing silicon is used for the determination of silicon in the form of SiO_2.

This method needs more testing with a variety of organic fluoro-silicon compounds.

Schwarzkopf Microanalytical Laboratory. WADC TR 56-19 Pt II.
ANALYSIS OF FLUORINATED ORGANO-METALLICS. Part II. Determination of Fluorine, Boron and Silicon in Organic Fluoro-Silicon or Fluoro-Boro-Silicon Compounds. Otto Schwarzkopf and Rosemarie Heinlein. AF 33(616)-3552. June 1957. ASTIA Document No. AD 130781. PB 131 088.

Development of analytical methods, for the determination of the elements silicon, fluorine and boron present in organic compounds containing two or three of the elements mentioned, was the object of this project.

For the determination of fluorine and silicon present in organo-fluoro-silicon compounds a method has been devised wherein fluorine is determined as PbClF and silicon as SiO_2 in the filtrate from PbClF.

A more satisfactory method was the determination of fluorine as PbClF and the determination of silicon as oxine-silicomolybdate. It was possible to determine silicon either in the presence of fluorine after converting fluorine to fluoboric acid, or to precipitate PbClF and determine silicon in the filtrate.

The three methods were tested with a variety of test samples. The preferred method is the determination of fluorine as PbClF and the determination of silicon as oxine salt of silicomolybdic acid after conversion of fluoride to fluoborate. The precision of this method was found to be ± 0.3% (absolute).

The presence of boron did not interfere in the determination of fluorine or of silicon carried out by the above mentioned method.

While silicon was without influence, fluorine interfered in the usual determination of boron by the boric acid mannitol titration method. Fluoride and boric acid react in acid medium forming fluoboric acid. This reaction made it impossible to eliminate carbon-dioxide completely prior to the addition of mannitol. The first neutralization endpoint was uncertain and high results for boron were the consequence.

Satisfactory results were obtained in the following manner. Fluorine is precipitated as PbClF at pH≈r, and filtered. To the filtrate sulfuric acid is added precipitating the excess lead and bringing the pH below pH≈1. The aqueous solution is concentrated in a distilling apparatus. Volatile weak acids - organic and inorganic are distilled and the distillate is discarded. Methanol is added to the residue and by keeping the water content low, boric acid is converted into methylborate which is distilled, hydrolized by NaOH and titrated by the mannitol titration method.

WADC TN 56-191 Pt 1.
CALCULATION OF THE RADIATION DOSE DELIVERED BY BETA EMITTING ISOTOPES. Part I - Point and Thin Plane Sources in Air. Edward A. Burke, 1/Lt. June 1957. ASTIA Document No. AD 130864. PB 131 343.

This report is the third in a series concerned with the development of methods for calculating the radiation dose delivered by beta emitting isotopes. This report extends the basic equations previously developed for all beta emitting isotopes. The publication by R. Loevinger of extensive experimental data on beta isotopes has been a major factor in making the present study possible since the equations developed are empirical and depend solely upon such data for their validity.

An empirical function for the dosage distribution around a point source of phosphorous -32 presented in WADC TN 56-101 has been extended to include all other beta emitting isotopes and found to be in excellent agreement with available experimental data.

Stanford Research Institute. WADC TR 57-141.
A COMPARISON OF HIGH-ENERGY ELECTRON AND GAMMA IRRADIATION EFFECTS ON ORGANIC LIQUIDS. Edwin L. Zebroski and Edwin M. Kinderman. AF 33 (616)-3738. June 1957. ASTIA Document No. AD 130857. PB 131 362.

It has been proposed that high-energy electron irradiation be substituted for gamma irradiation in testing the radiation resistance of aircraft materials, components, and systems. To determine the feasibility of the proposal, the effects of electron and gamma radiation on selected organic liquids were compared. Results of this study and the experimental work reported by others indicate that there is qualitative similarity between electron and gamma radiation effects on simple compounds. Some differences in quantitative yields are observed, but these may be a result of differences in time of irradiation rather than of differences in radiation type.

WADC TR 56-465.
ELECTRICAL PROPERTIES OF IRRADIATED POLYMERS. Ralph E. Woodard. June 1957. ASTIA Document No. AD 130801. PB 131 254.

A brief discussion is given of the electronic nature of solids as it applies to insulators and then polymers. The fundamental theory of nuclear radiation effects is discussed and shown to be associated with the laws of energy dissipation of these radiation particles within a material. A survey is given on the role that nuclear radiation plays in polymer kinetics. Work of various investigators on the electrical properties of irradiated polymers is reported. An analogy is drawn between photoconductivity in semiconductors and nuclear radiation induced conductivity in certain polymers. The instantaneous nature of this induced conductivity is emphasized. The change produced in the dielectric constant of an irradiated polymer is considered in terms of crosslinking and dipole moments. A survey is included of the available engineering data for irradiated polymers. Factors affecting radiation to the electrical properties of two tetraarylmonosilanes is given.

WADC TR 56-455.
STUDIES OF QUANTITATIVE METHODS FOR THE SEPARATION AND DETERMINATION OF ZIRCONIUM AND THORIUM IN MAGNESIUM ALLOYS. B. A. Raby, 1/Lt. June 1957. ASTIA Document No. AD 130797. PB 131 244.

This paper describes the results of a comparative study of analytical methods suitable for the determination of thorium and zirconium in magnesium base alloys.

In addition, this paper describes an attempted amalgamation of selected analytical methods and ion exchange techniques into a scheme for the quantitative analysis of thorium and zirconium in these alloys. Manganese and zinc, common constituents of magnesium base alloys, were carried in the experiments, but only to study their interference in the proposed scheme.

The sample is dissolved in 12N-HCl and the resulting solution is percolated through a column containing Dowex 2-X8 anion exchange resin. Magnesium and thorium pass through the column while zirconium, zinc, and manganese are absorbed. The latter elements are removed by eluting the column with water. The thorium and zirconium in the separated fractions can be determined by means of the complexometric titration methods described by J. S. Fritz.

WADC TN 56-183.
THE CONSOLIDATED ANALYTICAL MASS SPECTROMETER AND ITS APPLICATIONS. George Hovan. July 1957. ASTIA Document No. AD 130926.

The mass spectrometer principle of separating ionized molecules according to their mass forms the basis of one of today's most effective methods of analyzing highly complex gas or liquid mixtures. Routine control analyses, purity determinations, exploratory analyses, and fundamental research investigations are all proven applications of the analytical mass spectrometer.

The scope is extremely broad as low vapor pressure materials can be analyzed by using the heated sample inlet at temperatures up to 150°C. An overall range of 1 to 700 mass units is provided for use by the operator. High sensitivity detects impurities in concentrations as low as 5 ppm. Sample size can be as little as 0.1 ml gas and less than 0.001 cc liquid. Deviation normally is within the range of 0.05 to 1.0 mol percent and usually less than might be expected from most other methods of analysis.

The instrument described in this report is the Consolidated Electrodynamics Corporation (CEC) 180° Analytical Mass Spectrometer. Details regarding the scope, theory of operation, and various applications of the mass spectrometer are included.

United States Radium Corporation.
WADC TR 56-623.
DEVELOPMENT OF A TRITIUM IMPREGNATED METAL FOR USE AS THE ACTIVATOR IN A SELF-LUMINOUS PHOSPHOR AND FOR USE AS AN IONIZATION SOURCE. D. L. Prosser, C. H. Wright, J. E. Atherton, J. G. MacHutchin and H. H. Dooley. AF 33(616)-3036. July 1957. ASTIA Document No. AD 131030.

A description of the methods and equipment used in the preparation of tritiated foils is presented. The results of the experimental studies indicate that maximum beta emission is obtained from extremely thin layers of titanium tritide which have been prepared on the surface of sandblasted stainless steel backing foils.

Phosphor response studies have indicated that the main factors effecting the response of ZnS type phosphors to tritium beta radiation are: (a) phosphor particle size (b) type and concentration of activator and (c) cadmium content.

Six research models of tritium foil-activated self-luminous sources have been prepared for submission to WADC. A description of the research models is provided.

Stanford Research Institute. WADC TR 57-465.
EFFECTS OF HIGH-ENERGY, HIGH-INTENSITY ELECTROMAGNETIC RADIATION ON ORGANIC LIQUIDS. E. M. Kinderman. AF 33(616)-3738. July 1957. ASTIA Document No. AD 157459. PB 131 936. Order from OTS $1.50.

Effective development of practical nuclear power required that materials be developed to withstand its attendant nuclear radiation. For example, among the materials which must withstand radiation in a nuclear-propelled aircraft are hydraulic fluids and lubricants. A knowledge of the effects of structure on the radiation stability of organic materials is of advantage in designing and producing such radiation-resistant hydraulic fluids and lubricants. This investigation attempts to provide such background information.

A series of organic esters, hydrocarbons, and miscellaneous compounds were subjected to radiation. A quantitative measurement of the amounts and kinds of radiation products produced was made. From the observed products and their amounts, some inferences were made as to the stability of these materials and the influence that their structure had on their stability.

Radiolysis of organic liquids is influenced by structure. Molecules containing branch chain and benzyl groupings are more susceptible to radiolytic damage than are molecules containing only straight chain groupings. This greater damage may be explainable in terms of the greater inductive effects possible with the branch chain and benzyl groupings. Greater radiation stability is observed with higher molecular weight compounds. This effect may be explained in terms of the mechanisms of energy absorption and disintegration by the molecule.

Comparison of the radiolytic with mass spectrographic data shows some correlation between the modes of molecular disintegration induced in the two cases. The correlation is best when conditions of radiolysis are similar to those in the mass spectrograph, that is when radiolysis occurs in an essentially low pressure condition through activated species whose half-life is short and whose diffusion constants or reaction times are long.

Both electron and gamma radiation was used. The former received emphasis because of its speed and convenience in the test program. Comparison of the effects of the two types of radiation in six cases shows some quantitative differences in radiolytic yield. The products identified are the same, and the same structural effects are evident. The available data do not permit definite statements of equivalence between electron and gamma effects, although this is indicated.

WADC TR 57-359.
ANALYTICAL APPLICATIONS OF FAR INFRARED SPECTRA I HISTORICAL REVIEW, APPARATUS AND TECHNIQUES. Freeman F. Bentley, Eugene F. Wolfarth, Nora E. Srp and Wilbert R. Powell. September 1957. ASTIA Document No. AD 142010. PB 131 514.

The infrared spectrum of a molecule is one of its most unique physical properties, and perhaps for this reason a large amount of spectral data have been amassed over the last few decades. Most of the work has been limited to the fundamental region of the spectrum since characteristic group frequencies capable of giving structural information of great value and precision occur in this region, and because it has been the easiest region to study accurately and rapidly.

Many characteristic group frequencies are also observed in the long wavelength region of the spectrum which are useful in analytical and structural determinations. Furthermore, spectra obtained in this region are particularly useful in material characterization. Slight changes in structure produce considerable changes in spectra, giving a more specific "fingerprint." Investigations in the far infrared region have been limited due to instrumental difficulties such as high atmospheric absorption, low energy, stray radiation, and the lack of suitable prism materials.

A review of the work accomplished to date in the far infrared region is presented in this paper, and most of the available literature is cited. A double beam double pass spectrophotometer equipped with cesium bromide optics is described. This instrument extends the useful spectral range to 35 microns. The general features of the instrument's construction and its performance capabilities are discussed.

Conventional infrared techniques are used, and in some instances simplified in obtaining infrared spectra in the cesium bromide region. Many common solvents such as carbon disulfide, furan, benzene, and dioxane, have large "open windows" in the long wavelength region and the infrared spectra of these and other solvents are given. The advantages of far infrared spectra, and some of the special techniques of obtaining infrared spectra in the cesium bromide region, are also discussed.

Stanford Research Institute. WADC TR 56-4 Pt I.
THE THERMODYNAMIC PROPERTIES OF MOLTEN SALTS. Part I. Molybdenum Hexafluoride. A. P. Brady, J. K. Clauss, and O. E. Myers. AF 33(616)-2558. September 1957. ASTIA Document No. AD 131096.

Assembling and testing of apparatus for measuring heat capacities from about 50°K to room temperature has been completed, including calibration of thermocouples by comparison with a platinum resistance thermometer and with a copper one. This apparatus has been used to determine the absolute entropy of molybdenum hexafluoride vapor at 298.16°K. The result, 79.7 ± 0.6 cal/mole·deg, agrees well with that calculated from its spectrum, 80.05 cal/mole·deg. A previously unreported solid-solid transition was found to occur at 263.6°K (-9.6°C). A solution calorimeter has also been assembled and tested. By comparison of the heat of solution in sodium hydroxide of molybdenum hexafluoride with that of the oxide, the heat

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of formation of MoF_6 (l) at 298.16°K was found to be -390.9 kcal/mole. From these values the corresponding standard free energy of formation is computed to be -363.1 kcal/mole. These data, together with data in the literature, have allowed calculation of the desired thermodynamic functions over the temperature range of interest.

Stanford Research Institute. WADC TR 56-4 Pt II. THE THERMODYNAMIC PROPERTIES OF MOLTEN SALTS: Part II. Tungsten Hexafluoride, Niobium Pentafluoride, and Vanadium Trifluoride. Orlo E. Myers. AF 33(616)-2558. September 1957. ASTIA Document No. AD 131097.

Assembly and adjustment of apparatus for measuring enthalpies from 50 to 1200°C has been completed. A new solution calorimeter suitable for use with metallic ampoules for measurements with fluorides has been constructed. An improved low temperature calorimeter has been built with the aid of electroforming techniques. This equipment has been used in conjunction with low temperature equipment previously described to obtain thermodynamic data for the following:

Tungsten hexafluoride vapor (from heat of solution and spectra):

$\Delta H_{form,\ 298.16} = -416.3$ kcal/mole
$\Delta F_{form,\ 298.16} = -394.8$ kcal/mole
$S_{298.16} = 81.75$ cal/mole·deg

Niobium pentafluoride (from heat capacity, enthalpy, and heat of solution):

$\Delta H_{form,\ 298.16} = -431$ kcal/mole
$\Delta F_{form,\ 298.16} = -404$ kcal/mole
$S_{298.16} = 38.30$ cal/mole·deg
$\Delta H_{fusion} = 2.92$ kcal/mole

Vanadium trifluoride (from heat capacity and enthalpy):

$S_{298.16} = 23.05$ cal/mole·deg

Excellent agreement was found between the last entropy and the predicted value from the theory of the late Prof. Latimer concerning the summation of ionic entropy contributions. Values of the above properties at temperatures up to 1400°K have been calculated when possible or estimated from experimental data and standard methods.

WADC TR 57-212.
X-RAY EMISSION LINES AND 2θ VALUES FOR LITHIUM FLUORIDE ANALYZING CRYSTAL. W. L. Baun and R. E. Brocklehurst. September 1957. ASTIA Document No. AD 131063. PB 131 440.

A complete table of x-ray emission lines and 2θ values, using a lithium fluoride analyzing crystal, is presented. This table includes all the lines of the K, L, and M series, order 1 to order 9.

Herrick L. Johnston, Inc. WADC TR 57-667. HIGH TEMPERATURE X-RAY DIFFRACTION CAMERA. AF 33(600)-32214. October 1957. ASTIA Document No. AD 150801.

A high temperature furnace was designed for simple attachment to a Norelco diffractometer. The furnace is constructed so as to permit X-ray diffraction studies of solid or powder specimens at temperatures up to 1800°C in vacuum or under pressures of 2 atmospheres or less. Scanning from 2θ of 20° to 122° is possible. Heating elements are made of .005 inch tantalum and temperature is measured by a Pt (87% Pt, 13% Rh) thermocouple or by an optical pyrometer. Temperatures up to to 1600°C are automatically controlled to within ±0.1%. Automatic control is also possible from 1600° to 1800°C. Measurements of the lattice parameters of tantalum at various temperatures, including 1800°C, were in agreement with previously published values.

WADC TR 57-375.
A METHOD FOR DETERMINING NEUTRON FLUX SPECTRA FROM ACTIVATION MEASUREMENTS. Sven R. Hartmann, 1/Lt. October 1957. ASTIA Document No. AD 142029. PB 131 611.

A method for the determination of neutron spectra which approximates the neutron flux by a linear combination of the response functions (cross sections) of the detectors used in the spectral measurement is discussed. This linear combination is shown to approximate the neutron flux in the mean and to provide a calculated activation, for each detector, identical with that found in the experiment. Some practical cases are considered herein. A set of seven cross sections are used to approximate a fission, constant, and monoenergetic spectrum. A discussion of uncertainty propagation is given with illustrative examples for the fission and constant spectras. The integral neutron flux is also calculated and shown to be more adequately representable than the neutron flux.

WADC TR 56-297.
A MODIFIED ICE CALORIMETER. Myron W. Belaga, 1/Lt and Robert F. Trapp, 1/Lt. October 1957. ASTIA Document No. AD 110435.

The design, construction, and calibration of a Bunsen type ice calorimeter and accompanying furnace have been completed.

The calorimeter was electrically calibrated and had a calibration constant of 64.56 calories per gram of mercury change relative to an accepted value of 64.631 calories per gram of mercury as determined by the National Bureau of Standards.

The enthalpy of standard aluminum oxide (supplied by NBS) was determined over a temperature range of 100° - 1650°F and the results were compared with those of the National Bureau of Standards. The enthalpy of aluminum oxide was determined within 6% of the standard values.

WADC TN 57-114.
X-RAY DIFFRACTION STUDY OF CRYSTALLINE SILANES. W. L. Baun, 1/Lt. October 1957. ASTIA Document No. AD 142066. PB 131 551.

X-ray diffraction patterns for nineteen crystalline silanes are presented. These compounds are of interest because of their high thermal stability and low vapor pressure. They are particularly well suited for X-ray diffraction analysis because of their crystalline nature and the individualistic patterns obtained.

WADC TN 57-308.
CRITIQUE ON THE ANALYTICAL REPRESENTATION OF SPECIFIC HEAT DATA. Richard Pawel, 1/Lt. November 1957. ASTIA Document No. AD 142095. PB 131 552.

A considerable lack of agreement exists in the literature for high temperature specific heat values. Since the majority of these data have been obtained indirectly through enthalpy measurements, the problem of data treatment is added to those of the experiment. It is thought that the present uncertainties in the methods of curve fitting the enthalpy data can yield misleading trends to the resulting specific heat curves. For this reason, it appears wise to consider the final result in the light of simple specific heat theory and the accuracy and reproducibility of the original enthalpy data. The complexity of the analysis should be based upon the reliability of the enthalpy data to avoid "over-fitting." It appears that in the absence of exceptionally precise enthalpy data, a quadratic expression will

satisfactorily describe the data, resulting in a linear specific heat representation. This simple representation is sometimes more compatible than those obtained by other means.

WADC TR 57-498.
DESIGN AND USE OF A 23,000 CURIE COBALT-60 FACILITY. Marvin C. Atkins, 1/Lt, Kurt Wolfsberg, 1/Lt, William N. Lorentz, 1/Lt and Donald R. Smith, 1/Lt. November 1957. ASTIA Document No. AD 142157. PB 131 619.

A two-chambered hot cell has been designed, built, and used. The hot cell is suitable for radioactive materials testing, hot chemistry, or handling of radiation sources. A 23,000 curie cobalt-60 source has been assembled and is being used for materials irradiations. The source in its normal form is 11.6 inch I. D. x 28 inches long. The activity of each of the 2450 slugs comprising the source was individually calibrated. Slugs of different activities were arranged to give the desired flux distribution inside the cylindrical source. Dose rates in and around the source have been measured with an ion chamber and glass dosimeters. An engineering approach to design of radiation sources has been formulated and tested with the 23,000 curie source. The report includes a complete description of the hot cell, design and assembly of the source, dosimetry methods, and mathematical design of cylindrical sources.

Armour Research Foundation. WADC TR 57-488.
MEASUREMENT OF THE HEAT CAPACITY AND DENSITY OF LIQUID COPPER. J. I. Lang. AF 33(616)-3795. November 1957. ASTIA Document No. AD 142138. PB 143 647. Order from LC M1 $2.70, Ph $4.80.

The density and specific heat of molten electrolytic tough-pitch copper meeting Federal Specification QQ-C-576 was determined for the temperature range 2000 to 4000°F.

Vitro Laboratories. WADC TR 57-226.
MEASUREMENT OF THERMAL DIFFUSIVITY OF VARIOUS MATERIALS BY MEANS OF THE HIGH INTENSITY ELECTRIC ARC TECHNIQUE. Charles Sheer, Lawrence H. Mead, Donald L. Rothacker and Leonard H. Johnson. AF 33(616)-3669. November 1957. ASTIA Document No. AD 142093. PB 131 601.

Studies have been made of the tail flame of a high-intensity electric arc to determine its suitability as a medium for testing materials under sustained high-temperature gaseous flow. The first year effort reported herein centered on the establishment of an electrode geometry for stable operation, measurements of the temperature and velocity distribution in the tail flame at varying ambient pressures, and qualitative observations of the electrodynamic, magnetic and thermodynamic properties of the tail flame.

The electrode geometry giving optimum flame stability consists of a triple cathode arrangement symmetrical with respect to the anode. A somewhat less favorable configuration utilizes a single cathode forming an acute angle with the anode axis.

The axial temperature distribution in the tail flame was measured by spectral line reversal technique for temperatures below 3200°K, and by spectral band analysis above this temperature. The data obtained by line reversal show that the temperature decreases with decreasing ambient pressure and increasing distance from the crater. The spectrographic data are currently in the process of reduction.

Flame velocity was measured by a modification of Rohloff's method, involving a temporary interruption of the arc and observation of the downstream propagation of the resulting disturbance by high-speed cinematography. The velocities measured thus far were 47, 78 and 150 m/sec at pressures of 1.0, 0.5 and 0.1 atmospheres, respectively. The increase of velocity with decreasing pressure is in general accordance with classical laws although the variation here appears to be less than linear, probably due to an increase in flame diameter with decreasing pressure.

Miscellaneous exploratory observations have shown the tail flame to be markedly diamagnetic and to possess a statistical dipole distribution of positive ions and free electrons. Also observed were strong plasma oscillations in the order of 2 kc and with peak amplitudes of 20 volts; this effect suggests the possibility of measuring charge density of both signs in the flame.

Surface heat flux rates were measured at 1.0 and 0.1 atmospheres on copper bodies in thermal equilibrium. The values obtained were 4.0 and 1.0 kw/cm^2 respectively, but it is emphasized that further measurements are required to corroborate these figures. The appearance of the gaseous flow about blunt and conical wedges in the presence and absence of coulomb potentials showed a distinct separation zone between body and flame, the thickness depending on ambient pressure and electrical polarity. This phenomenon is believed to be associated with a magneto-hydrodynamic effect.

Finally, diffusivity measurements were made in the tail flame on OFHC copper and graphite plates. The data obtained thus far agree with literature values in order of magnitude. Closer refinements in the instrumentation and evaluation techniques appear necessary before the tail flame can be used for measurements of this type.

Armour Research Foundation. WADC TR 57-627.
PHOTO-CONDUCTIVE MATERIALS FOR USE IN HIGH INTENSITY NUCLEAR RADIATION MEASUREMENTS. Robert J. Robinson and Leonid V. Azaroff. AF 33(616)-3762. November 1957. PB 151258. Order from OTS $1.75.

This report describes the results of a one year program for the study of photoconductive materials for use in high intensity nuclear radiation measurements. The major accomplishment of this program is the correlation of gamma ray induced photoconductivity to the well known visible light induced photoconductivity. The phenomenological theory of photoconductivity is shown to apply to the gamma ray region as well as to the visible light region. The energy dependence observed in the response of CdS crystals is shown to be due to the energy dependence of the absorption process. The average ionization energy is shown to be in good agreement with independent measurements employing alpha and beta rays.

Two methods for growing single crystals, a flow system and a static system, were employed in the preparation of single crystals of cadmium sulfide. These can be grown large enough for device purposes, and with the desired electrical properties by control of the impurities added. Polycrystalline detectors can be prepared in several ways; however, their response properties are not as good as those obtained with single crystals.

National Bureau of Standards. WADC TR 57-374 Part I.
PHYSICAL PROPERTIES OF HIGH TEMPERATURE MATERIALS. Part I. New Apparatus for the Precise Measurement of Heat Content and Heat Capacity from 0° to 1500°C. Thomas B. Douglas and William H. Payne.

AF 33(616)-56-21. November 1957. ASTIA Document No. AD 142119.

There is described in detail new apparatus for the accurate measurement of the heat content and heat capacity of solids and liquids over the temperature range 0° to approximately 1500°C (32° to 2750°F). For use of the 'drop' method, a new Bunsen ice calorimeter and resistance-heated furnace were designed, and have been partly constructed. The ice calorimeter is similar to that developed earlier at the National Bureau of Standards, but embodies several minor modifications. To achieve regions of known and uniform temperature, the furnace was designed to have three concentric cores wound with 17 independently controlled wire-heater sections. Temperatures will be measured by several platinum-rhodium thermocouples, which are arranged to permit their periodic recalibration in place. After the performance of the apparatus has been tested, the first measurements undertaken are to be on standard-sample synthetic sapphire (aluminum oxide). These data will augment the Bureau's earlier measurements on this material from 0° to 900°C (32° to 1650°F), and permit its use as a heat-capacity standard up to approximately 1500°C (2750°F).

WADC TN 57-294

DETERMINATION OF HYDROGEN IN ZIRCONIUM HYDRIDE. Wade H. Jones. December 1957. ASTIA Document No. AD 142186. PB 131 972.

It is now generally recognized that control of the hydrogen content in zirconium is important in the manufacutre, melting, fabrication and subsequent use of the metal. It is therefore highly desirable to have a simple, rapid, precise method for determination of hydrogen in zirconium. Such a method is described. It is based on the measurement of the equilibrium pressure of hydrogen over the metal in a closed system under predetermined conditions.

This report covers the analysis of thirty-five samples of zirconium hydride with temperatures ranging from 1900°C to 1000°C and collection times from 10 to 30 minutes.

WADC TN 57-335.

FEASIBILITY OF A GRAPHITE-CARBON DIOXIDE IONIZATION CHAMBER TO MEASURE CARBON DOSE AT HIGH DOSE RATES. Robert L. Hickmott. December 1957. ASTIA Document No. AD 142249. PB 131 671.

This report discusses the feasibility of designing a graphite-carbon dioxide ion chamber for the absolute measurement of the carbon dose rate from a pure gamma field up to 10^{10} erg/g/hr. There is insufficient data on neutron response to judge the feasibility of using the ion chamber in mixed fields with large neutron components.

WADC TN 57-207.

STANDARD INSTRUMENTATION TECHNIQUES FOR NUCLEAR ENVIRONMENTAL TESTING. Walter R. Burrus, 1/Lt. December 1957. ASTIA Document No. AD 142179. PB 131 590.

The ANP Advisory Committee for Nuclear Measurements and Standards was established to standardize nuclear measurements for all Air Force programs supporting the development of nuclear weapon systems. The first phase of this Committee's effort is nearly complete and is concerned with the standardization of nuclear measurements for radiation damage studies. This Technical Note presents and discusses Committee recommendations for standardizing radiation damage measurements. A minimum number of measurements were selected which can provide a consistent basis for comparison of experiments and correlation of data. A measurement of "absorbed dose" in carbon is recommended as the minimum measurement of the gamma radiation field. In addition, determination of the activation of U-238, Pu-239 (boron shielded) and sulphur is recommended as the minimum measurement for neutron radiation. Consistent units and symbols for reporting these measurements are recommended. Additional techniques will be recommended in the future.

Armour Research Foundation. WADC TR 58-42.

PRODUCTION OF HOMOGENEOUS TITANIUM ALLOY STANDARDS. Donald H. Turner. AF 33(616)-3098. January 1958.

Three compositional variations of each of nine commercial titanium alloys were prepared by a triple arc melting procedure for use as standards for spectrographic and X-ray fluorescent analysis of titanium alloys. Analysis of each triple-melted, as-cast ingot has been carried out by three laboratories.

The material was supplied to the National Bureau of Standards as 1 1/4 inch diameter, centerless-ground bar. In general, there is good agreement between analyses and nominal composition. Where there are differences, trends are similar.

WADC TN 57-342.

THE QUANTITATIVE ANALYSIS OF THE FUNGICIDE 3, 3'-DIFLUORO 4, 4'-DIHYDROXYBIPHENYL. Myron L. Dunton. January 1958. ASTIA Document No. AD 142277. PB 131 971.

A quantitative method is described for determining the quantity of the fungicide 3, 3'-difluoro 4, 4'-dihydroxybiphenyl which has been added to white duck cloth. This method involves extraction with methanol and subsequent ultra-violet spectrophotometric measurement. It also is suggested that this method may be applied to the analysis of other related fungicides.

Oklahoma Agricultural and Mechanical College.
WADC TR 57-319.

DETERMINATION OF THE THERMAL CONDUCTIVITY OF MIXED AND SYNTHETIC ENGINE OILS BY THE UNSTEADY-STATE METHOD. E. E. Kohnke, A. R. Schmidt. AF 33(616)-3544. February 1958. ASTIA Document No. AD 150961.

Results are reported for the thermal diffusivities of five mineral and synthetic oils as measured by an unsteady-state method at temperatures between 80°F and 430°F. Maximum measurement temperatures vary depending upon the chemical nature of the various oils.

Experimental apparatus is described and methods of data analysis discussed. A critical evaluation of the potentialities of unsteady-state methods in general and of the particular experimental procedure employed are presented. Suggestions for improvement are offered.

WADC TR 56-515.

EFFECTS OF GAMMA RADIATION ON LINEAR POLYETHYLENE. William B. Terrell, Captain, and Jack T. Humphries, 1/Lt. February 1958. ASTIA Document No. AD 142343. PB 131 700.

Linear polyethylenes have recently been developed which have empirical chemical formulas identical to those of common polyethylenes, but which differ in the molecular arrangement of the polymer chain. The physical properties of two linear polyethylenes were measured before and after gamma irradiation to afford a comparison with conventional polyethylene.

Measurements were made of the following properties as functions of total radiation dosage up to 10^9 roentgens: tensile strength, elongation, hardness, impact strength, specific gravity, water absorption, heat distortion temperature, dielectric constant, arc resistance, color, and infrared absorption. In addition, densities were determined as functions of both radiation dosage and temperature.

WADC TN 57-298 Pt I.
PROCEEDINGS OF THE ANP SPECTROSCOPY INFORMATION MEETING HELD AUGUST 6-7 AT WRIGHT AIR DEVELOPMENT CENTER. Robert E. Brocklehurst. February 1958. ASTIA Document No. AD 142342. PB 131 773.

This report is a compilation of the unclassified papers given at the ANP Spectroscopy Information Meeting held at Wright-Patterson Air Force Base, August 6 and 7, 1957. The subjects covered in these proceedings are instrumentation for gamma and neutron spectral measurements, treatment of experimental data, characteristics of scintillation crystals, and the use of beam collinators.

Trg, Incorporated. WADC TR 57-771.
APPROXIMATION OF ALL ORDERS OF THE EXPONENTIAL INTEGRAL. Francis Clark. AF 33(616)-5187. March 1958. ASTIA Document No. AD 151084.

Approximations are sought for the exponential integral functions, i.e.,

$$E_m(p) = p^{m-1}\int_p^\infty \frac{e^{-x}dx}{x^m} \quad , m>0.$$

The function $E_m(p)$ is set equal to

$$\frac{e^{-p}}{p+m+q_m(p)}$$

and suitable algebraic approximations to the function $q_m(p)$ are then found. Consideration is given to ways of estimating the error of the approximations.

WADC TR 56-436.
THE RETENTION OF FISSION PRODUCTS BY SOILS UNDERLYING THE NUCLEAR ENGINEERING TEST FACILITY AT WADC. Bruce Raby and Dr. George John. March 1958. ASTIA Document No. AD 151081.

This report primarily concerns the retention of fission products by soils underlying the site for the Nuclear Engineering Test Facility. The purpose of the study was to provide some data which would be of value in planning for the contingency of a reactor "run-away" and for possible seepage of contaminated water during normal operation.

Three types of experiments were conducted: (1) total capacity studies of the soils, (2) equilibrium sorption of ions by the soil in a static system, and (3) elution of ions from the soil by water. These experiments were of an exploratory nature, and although not complete, do give an indication of the soil's ability to retain various fission products. However, on the basis of the few experiments performed, it is possible to provide only qualitative information and to indicate the direction and extent of future studies.

Quantum, Incorporated. WADC TR 57-512.
STATE OF DEVELOPMENT OF THERMAL RADIOMETER. Arnold Pfenninger, Harold E. Henry, Maurice Godet and Conrad A. Wogrin. AF 33(616)-3628. March 1958. ASTIA Document No. AD 151060. PB 131 746.

The objective of the work covered by this report was the development and construction of a thermal radiometer.

This report describes the progress made to date on the design and manufacture of the components of the instrument and includes mathematical analyses of both the heat transfer and control system problems where these were necessary and feasible. It also includes a discussion of the specifications as originally outlined and revisions suggested on the basis of analytical and experimental work performed on this contract.

Armour Research Foundation. WADC TR 57-334 Sup. 1.
EVALUATION OF THE MECHANISMS WHICH AFFECT THE PERFORMANCE OF "THERMAL RADIATION RESISTANT COATINGS." C. Roland McCully, Louise E. Moore, F. V. Schossberger and Edwin H. Tompkins. AF 33(616)-3595. April 1958. ASTIA Document No. AD 151164.

The work covered in this report was done for the Wright Air Development Center under Contract No. AF 33 (616)-3595 over the period from May 1, 1956 to May 1957. This report is a supplement to the Final Report: Theory, which gives an analysis of the mechanisms of scattering and absorption in highly-reflective paint coatings. This supplement is intended to serve as a guide in the formulation of reflective coatings.

The thermal sources for the energy to be reflected were specified in the contract to be 3000°K and 6000°K black bodies, which yield peak intensities at 1.0 micron and 0.50 micron, respectively. The modification of the energy distributions from these sources by atmospheric absorption is discussed to show how such absorption might be used to protect an aircraft.

The optical characteristics required in vehicles and pigments suitable for reflective coatings are summarized, and tentative recommendations are made. For vehicles, fluoroethylenes (Kel-F, Teflon), silicones, vinylidene chloride-acrylonitrile copolymers, and epoxy resins are considered suitable; for pigments, rutile and colorless silicon carbide are outstanding, but boron nitride and colorless barium titanate also have possibilities. Since titanium dioxide is readily available, its methods of manufacture are discussed in some detail to show how particle size and impurity content are controlled.

On the basis of several approximations, first-order particle size distributions required in rutile pigments for the reflection of 3000°K and 6000°K radiation are presented. Centrifugal sedimentation and elutriation techniques are described for the classification of pigments and the formulation of specified size distributions. The presence of considerable infrared in the radiations to be reflected requires the presence of larger particle sizes than are normally present in conventional white pigments. This is particularly true for 3000°K radiation, where the median particle size should be about 0.45 micron instead of the 0.25 micron size present in commercial rutile pigments. If a coating having a median size of 0.25 micron were exposed to 3000°K radiation, about half of the radiation would penetrate to the base of the coating. For 6000°K radiation the penetration would be about 20%, which is still seriously large.

Armour Research Foundation. WADC TR 57-334.
EVALUATION OF THE MECHANISMS WHICH AFFECT THE PERFORMANCE OF "THERMAL RADIATION RESISTANT COATINGS." Louise E. Moore, Matthew Prastein, Edwin H. Tompkins and Donald O. Van Ostenburg. AF 33(616)-3595. April 1958. ASTIA Document No. AD 151163.

This is the final report on Contract AF 33(616)-3595, ARF Project C 097, for Wright Air Development Center,

covering the period from May 1, 1956 through April 30, 1957. The theory of absorption, reflection, and scattering of electromagnetic radiation is presented in a classical review and for harmonically bound electrons and spheres. The absorption spectra of possible pigments and vehicles are considered in terms of incident radiation from 3000°K and 6000°K black body radiators, with possible modification by atmospheric absorption. Although refractive indices of vehicles are largely between 1.4 and 1.7, pigments may have high refractive indices, and calculations are made to determine indices of pigments. In systems less than 6% pigment by volume, the statistical distribution of the particles is largely singly or in groups of two. Procedures to determine the size distribution of the particles are illustrated with rutile. A first approximation to an optimum size distribution is a modified black body curve. The wavelength axis, contracted by the factor 0.41, is the diameter axis for rutile in linseed oil.

WADC TR 58-198.
ANALYTICAL APPLICATIONS OF FAR INFRARED SPECTRA II. SPECTRA-STRUCTURE CORRELATIONS FOR ALIPHATIC AND AROMATIC HYDROCARBONS IN THE CESIUM BROMIDE REGION. Freeman F. Bentley and Eugene F. Wolfarth. May 1958. ASTIA Document No. AD 155566. PB 151677. Order from OTS $1.50.

The infrared absorption spectra of some 400 aliphatic and aromatic hydrocarbons have been investigated from 15 to 35 microns and the characteristic absorption frequencies incorporated into spectrastructure correlation charts. The classes of compounds studied were alkanes, alkenes, cyclopropanes, cyclobutanes, cyclopentanes, cyclohexanes, substituted benzenes, naphthalenes and biphenyls. The skeletal bending frequencies of the alkenes and alkanes and the nonplanar bending frequencies of cycloalkanes and aromatics are the most useful. The wavelength and intensity of the out-of-plane ring frequencies of aromatic molecules give some indication of the nature of the substituents. Typical infrared spectra of the hydrocarbons are presented.

WADC TN 58-105.
BIBLIOGRAPHY ON PHOTOGRAPHIC FILM DOSIMETRY. May 1958. Gordon H. Griffith, 2/Lt. USAF. ASTIA Document No. AD 155571. PB 151026.

This report contains a bibliography of several books and articles that have been published through 1957 on photographic film dosimetry. It has been divided into two parts, one for X- and gamma radiation and the other for neutrons. Brief abstract type comments are included with each reference.

WADC TN 57-395.
DENSITY MEASUREMENTS FOR SMALL PARTICLES. Nora E. Srp. May 1958. ASTIA Document No. AD 151188. PB 151003. Order from OTS $0.50.

A method is described for determining the density of small solid objects. A capillary buret is used to draw liquid from a convex meniscus at the top of a small metal cup until the meniscus breaks. The small object is then dropped into the cup and the excess liquid is drawn into the buret until the meniscus breaks again. The difference between the two levels on the buret measures the volume of the material. The weight of the material divided by the volume gives the density of the object in grams per cubic centimeter. These samples were measured at 80° ± 0.5°F. Since standards were not obtained the accuracy was not determined. The expected deviation from the mean for a single determination is ±0.07 g/cc.

WADC TN 58-46.
AN EXTRACTION PROCEDURE FOR THE DETERMINATION OF THE METALLIC CONSTITUENTS IN GREASES. Larry A. Harrah, 1/Lt. May 1958. ASTIA Document No. AD 155522. PB 151027.

A discussion is given of conditions required for the extraction of the metallic ions from an organic liquid soap to an aqueous phase previous to the determination of the metallic ion concentration.

An analytical procedure for these ions, using the flame photometer, is proposed and evaluated.

WADC TN 57-413.
INFRARED ABSORPTION SPECTRA OF BROMOHYDROCARBONS IN THE 15-35 MICRON REGION. Wilbert R. Powell. May 1958. ASTIA Document No. AD 151189. PB 151134. Order from OTS $1.00.

The infrared spectra of a series of aliphatic and aromatic bromides have been studied with the intent of establishing the carbon-bromine stretching vibration absorption band. Infrared spectra of the straight chain aliphatic series produce two strong absorption bands in the 15.5-15.7 micron and the 17.7 to 18.0 micron region due to the trans and gauche vibrations of the RCH_2-Br group. At least one strong absorption band, which appears to be characteristic of a C-Br stretching vibration, is present in the 15-20 micron region in all the aliphatic bromides studied.

The assignment of bands to the carbon-bromine stretching vibration in aromatic bromides is complicated by the presence of bands due to vibrations of the aromatic ring structure.

Mellon Institute. WADC TR 58-59.
QUANTITATIVE DETERMINATION OF FLUORINATED FUNGICIDAL AGENTS IN AIR FORCE MATERIEL. Harry V. Drushel, Doris J. Toft. AF 33(616)-3998. ASTIA Document No. AD 155524. PB 131993. Order from OTS $2.25.

Methods for the extraction of the fungicides 1-fluoro-3-methyl-4,6-dinitrobenzene and 5,5'-difluoro-2,2'-dihydroxydiphenyl sulfide have been developed on the basis of the mechanism of interaction between the fungicide and the treated material. Numerous methods including polarography, spectrophotometry, and colorimetry have been shown to be useful for the determination of these fungicides. Leather, however, offers considerable difficulty during analysis because of serious interference from tannins which are simultaneously extracted. Steam distillation or azeotropic distillation with organic solvents was not successful for separating the fungicide from tannins. Anion exchange chromatography was used to separate the fungicide but this technique suffers from the disadvantage of being time-consuming. Some additional methods of separation and determination which have not, as yet, been examined are discussed. The choice of any one of the analytical methods described will depend upon the nature of the material which has been treated with the fungicide.

WADC TN 58-58.
A SIMPLIFIED AIR SAMPLING METHOD. Robert D. Thomas. May 1958. ASTIA Document No. AD 155523.

This report describes a fast and efficient method of determining the radioactive particulate matter content of air. A comparison is made with the previous time consuming method to show the advantages of the new method. The method is unique for calibration since the counting system can be easily calibrated for any isotope to be used for com-

parison purposes. This method is definitely an advancement in the state-of-the-art for air sampling techniques.

Midwest Research Institute. WADC TR 58-70. HEAT CAPACITY DETERMINATION OF MINERAL AND SYNTHETIC ENGINE OILS, LUBRICANTS, FUELS, AND HYDRAULIC FLUIDS IN THE TEMPERATURE RANGE 70°- 500°F. J. W. Barger, C. C. Bolse, T. M. Medved. AF 33 (616)-5269. June 1958. ASTIA Document No. AD 155630. PB 151210. Order from OTS $1.50.

An adiabatic calorimeter for measuring heat capacities of liquids in the temperature range 70° to 500°F was designed and constructed. Experimental technique and methods of calculation were devised to minimize errors. Using these calorimeters, the heat capacities of 32 mineral and synthetic engine oils, lubricants, fuels and hydraulic fluids were measured. The heat capacities were measured at five temperatures spanning the 70° to 500°F range. The calculated errors in the final results were 2 to 5 percent.

WADC TN 58-142. THE IDENTIFICATION OF CRYSTALLINE FERROCENES BY X-RAY DIFFRACTION. William L. Baun, 1/Lt USAF. July 1958. ASTIA Document No. AD 155761. PB 151310.

X-ray diffraction patterns for twenty-three solid crystalline ferrocenes are presented. These compounds are of interest because of their high thermal stability and wide potential applications. They are particularly well suited for x-ray diffraction analysis because of their crystalline nature and the individualistic patterns obtained from these compounds.

Battelle Memorial Institute. WADC TR 58-158. INVESTIGATION OF EFFECTS OF RADIATION APPLICABLE AS GAMMA RADIATION DOSIMETERS. J. F. Kircher, M. J. Oestmann, J. H. Cahn, P. Schall, B. W. King, W. A. Hedden, J. Moody, G. D. Calkins. AF 33 (616)-3905. July 1958. ASTIA Document No. 155727. PB 151314. Order from OTS $2.50.

This project was initiated to survey the field of radiation effects to determine which effects might be useful for high-level gamma dosimetry. Accordingly, the approach has been to survey the literature in broad areas and then to initiate experimental studies in those cases where additional information was required for evaluation of certain systems. It was not possible, however, to carry out experimental programs with all promising systems.

The areas investigated fall into four general categories: organic systems, inorganic oxidation-reduction reactions, semiconductor materials and devices, and ceramics.

Several promising organic systems are evaluated. Polymer systems and dye solutions will serve as dosimeters, but probably not to the high dosages desired. Reduction of metal oxides by hydrogen evolution from organic material holds promise but needs more study for proper evaluation.

The literature on radiation effects on inorganic reactions was surveyed but no experimental work was undertaken. However, some promising systems are pointed out.

A theoretical study of gamma-ray effects in semiconductor materials points up the energy dependence of this effect. It was demonstrated, however, that simple semiconductor devices can function as very high-intensity dose rate meters.

Several promising glass formulations were uncovered. These are primarily antimonate glasses, with and without added polyvalent metal oxides, and high-lead glasses. These glasses show promise of being useful dosimeters to the highest dosage of interest, 10^{12} ergs/g(C).

The Anderson Physical Laboratory. WADC TN 58-134. THE DETERMINATION OF NEAR INFRARED SPECTRA. Scott Anderson, Raymond Isaac. AF 33(616)-5283. August 1958. ASTIA Document No. AD 155773. PB 151309. Order from OTS $1.00.

Eight hundred and fifty (850) near infrared spectra of various classes of organic compounds and commercial mixtures, of interest to the Air Force Research and Development Program, were determined between the region 0.7μ to 2.8μ with the use of a Beckman DK-2 Ratio Recording Spectrophotometer.

Rapid sorting of spectra was provided with the use of Wyandotte - ASTM structure and name-formula IBM cards. A method for coding near infrared absorption bands on the standard Wyandotte - ASTM card was devised.

Methods of sample preparation and the use of solvents is discussed.

WADC TN 58-56. THE EVALUATION AND ADAPTATION OF THE BROMINATION TECHNIQUE OF DETERMINING OXYGEN IN METALS. Lois A. Keyser, Charles D. Houston. August 1958. ASTIA Document No. AD 155821. PB 151250. Order from OTS $1.00.

This paper contains laboratory results and discussion of the problem of determining oxygen in titanium and other metals using the technique of bromination.

The gravimetric bromination equipment was adapted for use with the conductometric carbon dioxide determinator so that the length of time of bromination could be shortened and a lower range of oxygen could be measured accurately.

An evaluation of the method and the recommendations for its use is given.

TRG, Incorporated. WADC TR 58-112. TRANSFORMATIONS OF SOLUTIONS OF DIFFUSION EQUATIONS IN TWO-MEDIUM GEOMETRIES. Visvaldis Mangulis. AF 33 (616)-5187. August 1958. ASTIA Document No. AD 155771. PB 151313.

The purpose of the work performed was to find transformations of solutions of diffusion equations in certain two-medium situations for isotropic sources having simple geometric shapes. It is expected that the results will be applicable to some shielding problems. The transformations were obtained for the following sources: point, disk, infinite plane, and solid cylinder sources interior to a semi-infinite medium joined at an infinite plane surface to a source free semi-infinite medium of a different material, and spherical shell or solid sphere sources interior to a spherical medium imbedded in an infinite medium of a different material.

WADC TR 58-78. THE EFFECTS OF GAMMA IRRADIATION ON ACRYLONITRILE-BUTADIENE COPOLYMERS. John A. Parker, Freeman F. Bentley, E. A. Peterson, Denver Hale. September 1958. ASTIA Document No. AD 155871. PB 151261. Order from OTS $2.50.

Changes in the chemical and physical properties produced by the irradiation of a series of acrylonitrile-butadiene copolymers varying in composition from 20 to 50% acrylonitrile with gamma photons from the cobalt-60

source at Wright Air Development Center have been measured and correlated with the nature of the copolymer, initial type of cure (sulfur, peroxide, or radiation), and the total integrated dose. This dose has been converted to the energy equivalent in graphite units now being recommended for standardized dosage terminology.

The efficiencies of these cross-linking processes induced by radiation have been estimated from the molecular weights between cross-links, M, determined by swelling volume measurements by a procedure similar to that used for natural rubber by Charlesby. This molecular weight varies with the radiation dose according to the general relationship:

$$M_c = \frac{m}{r^* + r_i}$$

Where r^* is the actual radiation dose incurred by the sample and r_i is an amount of radiation equivalent to the initial number of cross-links in the system. The slope varies with the particular copolymer.

The efficiencies of cross-linking determined in this way are a more realistic measure of the relative stabilities of these copolymers than a comparison of the complex physical properties. Nevertheless, reasonable agreement exists between cross-linking densities determined by swelling volume and those determined by modulus measurements. The very regular behavior of the change in cross linking density as a function of the total amount of energy deposited in terms of equivalent in graphite suggests the use of such polymers in dosimeter applications. These relationships enable one to predict the energy equivalent per gram of graphite for compositions cured by either sulfur or peroxides.

The qualitative changes in the chemical structure associated with the radiation induced cross-linking have been characterized by infrared spectra. By using the incremental change in modulus it is possible to apply the empirical equation proposed by Sisman and Bopp relating the change in Young's modulus to total dose. Specific constants for these equations have been calculated for this series of elastomers by means of which one may predict the useful life of mechanical rubber goods compounded from them in a radiation field of known intensity.

Armour Research Foundation. WADC TR 56-222 Pt II.
DETERMINATION OF EMISSIVITY AND REFLECTIVITY DATA ON AIRCRAFT STRUCTURAL MATERIALS. Part II - Techniques for Measurement of Total Normal Emissivity, Normal Spectral Emissivity, Solar Absorptivity and Presentation of Results. Howard T. Betz, O. Harry Olson, Bert D. Schurin, James C. Morris. AF 33(616)-3002. October 1958. ASTIA Document No. AD 202493. PB 151603. Order from OTS $3.00.

Equipment has been designed, constructed and calibrated for the measurement of total normal emissivity in the range -300°F to +3000°F. The procedure consists in comparing the total normal radiance of a sample to that of a comparison blackbody, the ratio of the signals being taken as the emissivity. A thermistor detector is used in conjunction with a folded optical system to record the radiation which is chopped at 16 cps.

The normal spectral emissivity values are obtained by an optical pyrometer method which makes use of the relationship between the true temperature of a sample to its apparent brightness temperature. A Leeds and Northrup disappearing filament pyrometer is used to make the necessary measurements.

An integrating sphere reflectometer is used to measure spectral reflectivity in the wavelength range, 0.3 to 3.0 microns. A General Electric Recording Spectrophotometer is used for the visible range and an apparatus of our own design for the ultraviolet and infrared. The latter employs a Perkin-Elmer monochromator, a comparison type integrating sphere, and a lead sulfide cell detector. Spectral reflectivity data are combined with solar spectral energy data to determine solar absorptivity at sea level and above the earth's atmosphere.

Armour Research Foundation. WADC TR 56-222 Part II Sup I.
DETERMINATION OF EMISSIVITY AND REFLECTIVITY DATA ON AIRCRAFT STRUCTURAL MATERIALS. O. Harry Olson, James C. Morris. AF 33(616)-3002. October 1958. ASTIA Document No. AD 202494. PB 151604. Order from OTS $1.00.

Total normal emissivity data are presented for a list of materials which could not be measured with the equipment described in the main body of the report. Instead they were measured with equipment developed subsequently to handle refractories and glasses. In the former, samples were heated by conduction heating in vacuum, in the latter, samples were heated in a tubular furnace in air.

Spectral Reflectivity curves from 0.3 to 2.7 microns are presented for a list of materials and their solar absorptivity values have been calculated.

Stanford Research Institute. WADC TR 58-206. EFFECTS OF HIGH ENERGY, HIGH INTENSITY ELECTROMAGNETIC RADIATION ON ORGANIC LIQUIDS. Robert M. Wagner, Leland H. Towle. AF 33(616)-3738. October 1958. ASTIA Document No. AD 202499. PB 151606.

Materials employed for practical nuclear power applications must possess optimum resistance to radiation effects on their molecular structure. Although certain radiation effects are considered beneficial, the majority are degradative to an unacceptable degree at high dose levels. The functions of hydraulic and lubricating fluids in nuclear power auxiliary equipment are cases in point. These fluids must maintain viscosity and flash point at optimum levels. A knowledge of the effects of molecular structural characteristics upon radiolytic stability of organic materials is needed to aid in the efficient design of radiation-resistant equipment. Some materials may of necessity be located in a high flux density radiation field. This study attemps to provide, through the study of several representatives of different types of organic structure, background information which may suggest methods of judicious choice for a given application.

The following compounds were subjected to high energy electron bombardment: alkyl aromatic hydrocarbons, aromatic ethers, nitro aromatics, amino aromatics, and aliphatic mixed ether-alcohol types. Quantitative measurements were made of the amounts and types of radiolytic products formed. Inferences were drawn from such measurements concerning (1) the relative stability of compounds, and (2) the influence of functional groups upon such stability.

Higher molecular weight compounds in general, and condensed aromatic ring systems in particular, are the most resistant to radiation. Aliphatic or heterophatic linkages between aromatic ring systems are sites of primary radiolytic scission.

The extent to which species of higher molecular weight than parent material are formed is directly dependent upon the fate of the initial products of the scission of each radiolytically labile linkage. The primary products of scission may (1) hydrogenate and revert to stable form; (2) if energetic enough, they may attack parent material; or (3) they may condense with each other producing products comparable to the parent in molecular weight.

Compounds possessing the ability to assume certain

spatial configurations, allowing resonance effects to exist, may permit absorbed energy to be innocuously reduced to levels which are below that required to rupture the molecular structure.

The presence of different functional groups on aromatic ring systems markedly influence the mode of radiolytic scission. These differences are reflected in either gas yield, polymer yield, or both.

The data do not permit complete and unequivocal definition of either the mode of radiolytic decomposition or the delineation of the structure of all products detected.

Mechanisms for product formation are suggested in cases where the data are sufficiently coherent and complete to permit rational systemization.

WADC TR 58-198 Sup I.
ANALYTICAL APPLICATIONS OF FAR INFRARED SPECTRA II. SPECTRA-STRUCTURE CORRELATIONS FOR ALIPHATIC AND AROMATIC HYDROCARBONS IN THE CESIUM BROMIDE REGION. Freeman F. Bentley Eugene F. Wolfarth. December 1958. ASTIA Document No. AD 207796. PB 151677S. Order from OTS $1.50.

The infrared absorption spectra of some 400 aliphatic and aromatic hydrocarbons have been investigated from 15 to 35 microns and the characteristic absorption frequencies incorporated into spectra-structure correlation charts. The classes of compounds studied were alkanes, alkenes, cyclopropanes, cyclopentanes, cyclohexanes, substituted benzenes, naphthalenes and biphenyls. The skeletal bending frequencies of the alkanes and alkenes and the non-planar bending frequencies of the aromatic hydrocarbons are the most useful for qualitative analysis in this region. The wavelength and intensity of the out-of-plane ring frequencies of aromatic molecules give some indication of the nature of the substituents. Typical infrared spectra of the hydrocarbons are presented.

WADC TN 58-146.
THE MECHANISM OF SOME SIMPLE CHEMICAL REACTIONS OCCURRING UNDER THE ACTION OF IONIZING RADIATIONS. Lowell A. King, 1/Lt. USAF. February 1959. ASTIA Document No. AD 209909. PB 59-11946.

A comparison of the suspected initial species formed in the radiation interaction primary act with the characteristics of subsequent chemical reactions affords the possibility to follow the roles of ionization, excitation, dissociation, and other initial processes in a radiation chemistry reaction and, in this way, explain its mechanism. Such comparisons are carried out in this report, and a summary of data is given pertaining to simple radiation chemistry reactions of hydrogen, oxygen, carbon, nitrogen, and a few of their most simple compounds.

Stanford Research Institute. WADC TR 58-683.
EFFECTS OF HIGH ENERGY, HIGH INTENSITY ELECTROMAGNETIC RADIATION ON ORGANIC LIQUIDS. Robert M. Wagner, Leland H. Towle. AF 33(616)-3738. March 1959. ASTIA Document No. AD 211915. PB 151836. Order from OTS $1.25.

Practical nuclear power applications require organic materials which offer optimum resistance to the undesirable effects of radiation. Hydraulic and lubricating fluids in nuclear power auxiliary equipment are cases in point. Some of these may of necessity be located in a high flux density radiation field.

This study of several representatives of different types of organic structure was an attempt to provide background information which may suggest methods of judicious choice for the efficient design of radiation-resistant equipment.

The following compounds were subjected to high energy electron or gamma bombardment: aromatic amines, aromatic nitro compounds, alkyl aromatic ethers, alkyl aromatic hydrocarbons, and aliphatic esters. In addition, alkyl aromatic compounds and aliphatic esters were subjected to high energy neutron bombardment.

The data do not permit either complete and unequivocal definition of the mode of radiolytic decomposition, or complete delineation of the structures of all products detected. Conclusions were drawn concerning (1) the relative stability of compounds, and (2) the influence upon stability of the location and nature of functional groups. The conclusions derived from the thirty-month study are listed below.

1. Higher molecular weight compounds in general, and condensed aromatic ring systems in particular, are the most resistant to radiation. Aliphatic or heterocyclic linkages between aromatic ring systems are sites of primary radiolytic scission.
2. The extent of formation of species of higher molecular weight than the parent material is directly dependent upon the fate of the initial products of the scission of each radiolytically labile linkage. The primary products may either (1) hydrogenate and revert to stable form, (2) attack parent material if energetic enough, or (3) condense with each other, producing products comparable to the parent in molecular weight.
3. Compounds not possessing the ability to assume certain spatial configurations, cannot utilize otherwise available resonance energy dissipation processes. Such processes would permit absorbed energy to be reduced to levels below that required to rupture the molecular structure.
4. The presence of different functional groups on aromatic ring systems markedly influences the mode of radiolytic scission. These differences are reflected in either gas yield, polymer yield, or both.
5. There is no linear relationship between the amount of methylene insulation between two aromatic systems and differences in radiolytic stability.
6. Unsymmetrical phenyl loading of a short alkyl chain does not alter the degree of radiolytic stability.
7. Alkyl unsaturation introduced between two aromatic systems facilitates hydrogen transfer to the target but does not reduce the total radiolytic instability of the system.
8. There is no essential difference in the yield or the nature of radiolytic products when alkyl aromatic hydrocarbons and aliphatic esters are irradiated with gamma rays, electrons, or neutrons.
9. An oxygen-rich atmosphere did not effectively scavenge energy, and thus inhibit radiolysis damage during electron bombardment of either alkyl aromatic hydrocarbons or aliphatic esters.
10. Branching of aliphatic chains induces radiolytic instability in either esters or hydrocarbons.

Arthur D. Little, Inc. WADC TR 58-623.
A NEW INSTRUMENT FOR THE DETERMINATION OF MOLECULAR WEIGHT BY DIFFERENTIAL VAPOR PRESSURE. Norman M. Wiederhorn, Jay H. Vreeland, Robert R. Perron. AF 33(616)-5483. March 1959. ASTIA Document No. AD 211914. PB 151832. Order from OTS $1.00.

A differential vapor pressure molecular weight instrument has been developed and constructed. The method developed is suitable for determining molecular weights of sparingly soluble (0.1% by weight) compounds in the 400 to 2000 molecular weight range to an accuracy of ±5%. The measurement involves a determination of the lowering of the vapor pressure by a known weight of material. For this purpose a differential pressure cell possessing the requisite sensitivity of 4 microns of

mercury as well as necessary cells were constructed. This high sensitivity has been attained by using a metal foil as sensing element and determining its displacement by measuring the change in capacitance between the foil and a fixed test electrode. The necessary control of temperature gradients to ± 0.0001°C was accomplished and preliminary calibration and investigation on sucrose solutions.

University of Michigan. WADC TR 58-405.
THERMAL CONDUCTIVITY OF LUBRICATING OILS AND HYDRAULIC FLUIDS. D. W. McCready. AF 33(616)-3543. March 1959. ASTIA Document No. 211693. PB 151780. Order from OTS $1.50.

An all-metal concentric cylinder type of thermal conductivity cell was designed, fabricated, and calibrated to measure the thermal conductivity of forty natural and synthetic base lubricating fluids.

Thermal conductivity values in the temperature range of from 70 to 500°F are reported for fluids considered stable to the higher temperature. The maximum temperatures for other fluids were limited by their instabilities under test conditions. Since each fluid has individual characteristics, no correlation of conductivity values appears possible. Values are considered precise and for possible correlation can be compared to those of a fluid chosen as a "standard reference".

WADC TR 59-119.
REMOVAL DOSE AS AN ENVIRONMENTAL MEASUREMENT OF X-RAY AND GAMMA RAYS. R. L. Hickmott. April 1959. ASTIA Document No. AD 211918. PB 151838. Order from OTS $1.00.

The Removal Dose of any material is the net energy per unit mass that a limitingly small mass of the material transfers from the incident photon flux to the kinetic energy of the associated flux of charged particles. Removal Dose is compared to both Absorbed Dose and Exposure Dose (roentgen) in terms of cross sections and spectra. Its relation to radiation effects is compared to the similar relation of Absorbed Dose.

WADC TN 59-79.
INVESTIGATION OF A NEW DYE-GLASS GAMMA RADIATION DOSIMETER. Denver Hale. May 1959. ASTIA Document No. AD 214763. PB 151964. Order from OTS $0.50.

This report was an investigation of the effects of gamma radiation on a unique organic dye - porous glass system. Effect of gamma ray dosage on the optical absorbancy of Methylene Blue, Fluorescein, Rhodamine B, Fast Reds S, and Brilliant Green dyes absorbed in Corning "thirsty glass" are presented. Data appear to be analytical and to follow Beer's law, thereby allowing one to relate optical absorbency with radiation dosage.

These experiments were of an exploratory nature, and although not complete, do indicate that this absorption of dyes in a porous glass matrix merits additional study for possible use as dosimeters in the range of 10^9 or higher erg gm^{-1} carbon gamma dosage.

WADC TN 58-368.
THE QUANTITATIVE ANALYSIS OF 5,ETHYL-10,10-DIPHENYLPHENAZASILINE IN AROMATIC BASE STOCK FLUID PRIOR TO PERFORMANCE TESTING. Lt James T. Thompson. May 1959.

A quantitative ultra-violet method, based on Beer's Law, has been developed for determining the quantity of an antioxidant additive (5-ethyl-10,10-diphenylphenazasiline) when blended with an aromatic base stock fluid. The method is applicable prior to performance testing for lubricity.

WADC TN 58-361.
RAPID SEPARATION AND GRAVIMETRIC DETERMINATION OF ALUMINUM IN FERROUS METALS. Lois A. Keyser, Charles D. Houston. May 1959.

A rapid method separating iron and aluminum has been developed, with the aluminum being determined gravimetrically. It is applicable to analyzing for aluminum in ferrous metals in the absence of nickel.

The separation of iron and aluminum is accomplished via the sodium hydroxide method and the aluminum is precipitated with 8-hydroxyquinoline.

Consolidated Electrodynamics Corp. WADC TR 59-107.
A MASS SPECTROMETER SYSTEM FOR MATERIALS RESEARCH SUMMARY REPORT, PHASE I. Charles F. Robinson, George D. Perkins, Norton W. Bell. AF 33(616)-5571. July 1959. ASTIA Document No. 216714. PB 161073. Order from OTS $1.00.

There are three areas in which mass spectrometry may be expected to contribute importantly to science and technology within the foreseeable future. They are (1) identification and estimation of minor components and trace impurities in solids; (2) structural studies of high-molecular weight materials; (3) identification and estimation of unknown materials by precise measurement of molecular weight. No mass spectrometer useful in all three of these fields has yet been built; however, the requirements on the instrumentation for these fields have sufficient similarity to encourage the belief that a single high-performance mass resolving system, together with a limited number of ion source and ion detector modules, might be adaptable to any of these fields. This discussion is a report of the progress made during the first phase of a three-phase program of research and development on such a system.

Anderson Physical Laboratory. WADC TR 59-344.
THE DETERMINATION OF NEAR INFRARED SPECTRA. Scott Anderson, Raymond Isaac, Myra Blankenship. AF 33(616)-5283. September 1959. PB 161336. Order from OTS $1.00.

Near infrared spectra of organic compounds of interest to the Air Force Research and Development Program were determined between the region 0.7 to 3.2 μ on a Beckman DK-2 Ratio Recording Spectrophotometer. Modification of the instrument's recording, to accommodate a chart which presents spectra at a single scale expansion of 2x, is discussed.

Rapid sorting of spectra was provided with the use of Wyandotte-ASTM structure and name-formula IBM cards.

Ledoux & Co. WADC TR 59-325.
ION EXCHANGE AND OTHER CHEMICAL METHODS FOR BERYLLIUM BASE ALLOYS. Silve Kallmann, Robert Liu, Hans Oberthin. AF 33(616)-5743. September 1959. PB 161295. Order from OTS $1.25.

While individual elements in association with beryllium can be determined by conventional chemical methods, ion exchange procedures are extremely useful when beryllium is combined with a number of alloying constituents.

Procedures are described in this report involving both cation and anion exchange resins which allow the determination of all elements in successive steps using one sample portion. The elements covered in the range of 0.1 to 10% are copper, aluminum, iron, nickel, cobalt, cerium,

silver, gold and palladium.

WADC TR 59-68.
ELECTRON MICROSCOPY OF ETCH PITS. Richard E. Pawel, 1/Lt, USAF. October 1959. ASTIA Document No. AD 231280. PB 161838. Order from OTS $0.75.

This paper presents some preliminary results on the study of etch pits in copper, alpha brass, and aluminum. Electron micrographs illustrate the crystallography of these pits, and serve as evidence of their potential use in metallographic studies involving orientation, deformation, and dissolution phenomena.

WADC TR 59-431.
SYNTHESIS, INFRARED SPECTRA, AND X-RAY POWDER DIFFRACTION DATA OF SOME 1, 3, 5, TRIAZINE DERIVATIVES. Herbert K. Reimschuessel, Neil T. McDevitt, William L. Baun. December 1959.

The synthetic methods are described for a series of triazine compounds which have not been reported previously. The infrared absorption spectra of this series of triazine derivatives have been obtained from 2-15 microns and assignments made for the specific vibrations. Absorption bands at 6.4, 6.65 and 6.9 microns have been assigned to vibrations arising from the triazine ring system, with the 6.65 micron band being the most prominent. X-ray powder diffraction data are presented for fifteen of the polycrystalline triazine derivatives. Methods of minimizing preferred orientation in these compounds and analysis of mixtures and impure compounds are discussed. The data presented permit rapid identification regardless of the complexity of the substitution.

WADC TR 59-666.
THE QUANTITATIVE ANALYSIS OF N-PHENYL-ALPHA-NAPHTHYLAMINE AND 5-ETHYL-10, 10-DIPHENYL-PHENAZASILINE IN ALIPHATIC TRIESTER BASE STOCK FLUID. Lee D. Smithson, James T. Thompson. January 1960. PB 161502. Order from OTS $0.50.

A quantitative ultra-violet method has been developed for determining the quantity of two antioxidant additives (n-phenyl-alpha-naphthylamine and 5-ethyl-10,10-diphenyl-phenazasiline) when blended with an aliphatic triester base stock fluid. The method has been applied to engine tested samples.

Massachusetts Institute of Technology. WADC TR 59-498.
INVESTIGATION OF FAR INFRARED SPECTRA. Richard C. Lord. AF 33(616)-5578. February 1960. PB 161738. Order from OTS $1.00.

Applications of a small grating spectrometer to problems of molecular structure by investigation of infrared absorption spectra in the region 50 - 300 cm^{-1} are reviewed. All molecules were studied in the vapor phase and both rotational and vibrational spectra were obtained. Pure rotational spectra of nitrogen dioxide, ozone, and sulfur dioxide are mentioned briefly. The value of far infrared spectra for study of low vibrational frequencies is discussed. It is pointed out that molecules with several potential minima usually have low frequencies and that observation of these frequencies can be useful in determination of the heights of barriers separating minima. Three origins of multiple minima are discussed: hindered rotation, inversion and quasi-linearity. Far infrared spectra of molecules illustrating each of these three types are described. Data are presented for methyl amine, hydrazine, trimethylene oxide and disiloxane.

The infrared spectra (50 - 600 cm^{-1}) are reported and discussed briefly for cyclopentane, cyclohexane, cycloheptane, cyclooctane, oxycyclopentane (tetrahydrofuran) and 1, 4-dioxy-cyclohexane (dioxane). Isolated low frequencies are given for thiacyclobutane, disilylacetylene and dimethylacetylene. New values of the thermodynamic functions for hydrazine are listed, and values from the literature are included for diborane and cyclobutane.

Arthur D. Little, Inc. WADC TR 59-724.
A NEW INSTRUMENT FOR THE DETERMINATION OF MOLECULAR WEIGHT BY DIFFERENTIAL VAPOR PRESSURE. N. M. Wiederhorn, J. H. Vreeland, R. H. Thompson. AF 33(616)-5483. February 1960. PB 161711. Order from OTS $0.50.

The differential vapor pressure molecular weight instrument developed previously was subjected to use and further evaluated with regard to sensitivity, reproducibility, and accuracy. In addition to this, a number of materials having molecular weight in the range 300 to 20,000 were used. The device is capable of measuring number average molecular weights up to 2000 at solution concentrations of 0.1% (gms/100cc). In order to extend the range of the instrument to molecular weights of 20,000, it is necessary to work with at least 1% solutions. At these concentrations, the instrument is accurate to approximately ±5%. A glass valve assembly and metal-coated Mylar sensing foils have been developed for use with the instrument. These facilitate the manipulations when making measurements and minimize maintenance problems.

General Electric Company. WADC TR 59-346.
PLASMA JET TEMPERATURE STUDY. Dr. Willard J. Pearce. AF 33(616)-5848. February 1960. ASTIA Document No. AD 236669. PB 161735. Order from OTS $2.50.

Spectral methods for measuring plasma temperatures in the range 5,000 to 15,000°K have been compared on theoretical and experimental grounds. For plasma jets, the best method is to determine the temperature from the intensities of atomic lines or of ionic lines, from the relation $I = KgA\nu \exp(-E/kT)$, in which

- I = intensity of emitted line in arbitrary units
- K = instrumental constant which can be assigned a value of unity
- g = statistical weight of upper energy level
- A = transition probability for the line and may be only a relative value
- ν = photon frequency of the wavelength being studied.
- E = energy of excitation of the atom before emission.
- k = Boltzmann molecular gas constant
- T = the absolute temperature which characterizes the relative population of excited states.

Tables of values of the required constants have been compiled and examples of various modifications of the method have been given.

Technique and constants have been given which permit determination of radial distributions of temperature with the least difficulty.

WADC TR 59-273.
A RELAXATION TIME TECHNIQUE FOR MEASUREMENT OF THERMAL DIFFUSIVITY. George Sonnenschein, Robert A. Winn. February 1960. PB 161752. Order from OTS $0.75.

A method has been developed for measuring the thermal diffusivity of solids under conditions of one-dimensional transient heat flow in a semi-infinite plate of finite thickness, adiabatically insulated at one face and subject to a constant thermal flux at the other. The method involves measurement of the time elapsed from start of exposure and of the temperature rise at a point along the direction of

heat flow. It is unique in that it confines thermometric sensing to a single point which, when located at one face of the test specimen, renders the method "non-destructive". With appropriate recording apparatus, measurements can be made on thin samples and on materials that preclude internal instrumentation.

The feasibility of the technique has been demonstrated on aluminum, Armco iron, copper, yttrium and on a plastic laminate, covering a diffusivity range from 7×10^{-4} to 1.0 cm^2/sec. and temperatures between 25 and 950°C.

University of Dayton. WADD TR 60-55.
ON THE FEASIBILITY OF USING X-RAY FLASH TECHNIQUES FOR DIFFRACTION STUDIES. Werner R. Rambaske. AF 33(616)-6607. March 1960. PB 161797. Order from OTS $1.75.

X-ray diffraction and related methods which are concerned with the analysis of the microstructure of matter are restricted in their possible variety of application by the relatively small radiation density emitted from ordinary X-ray tubes. Intensities about a million times higher than used now would be necessary to conduct high speed diffraction analyses for dynamic problems, and to produce in static problems, and to produce in static problems diffraction and related pictures from very small areas or volumes of matter. The highly intense X-ray flash probably can be developed into such a source. By successive studies, the radiation and energy properties of X-ray flashes are investigated, other literature about this field is collected, and the feasibility of the project, to produce diffractomgrams with X-ray flashes, is demonstrated. The first X-ray Laue diffraction picture from a crystal (NaCl) obtained in an irradiation time or less than one microsecond and another one exposed within about 4 microseconds are shown at the end of this report; they demonstrate the feasibility of the methods as proposed by the author in March 1959. Many new ideas and information were gathered for still necessary wide improvements in the future.

National Bureau of Standards. WADC TR 59-510.
STANDARDIZATION OF THERMAL EMITTANCE MEASUREMENTS. W. N. Harrison, J. C. Richmond, E. K. Plyler, R. Stair, H. K. Skramstad. AF 33(616)-58-20. March 1960. PB 161879. Order from OTS $1.00.

The principles governing emission, transmission, reflection and absorption of radiant energy are briefly reviewed; definitions of pertinent terms as used in the text are given; and mathematical relationships between properties and quantities that are involved in the project are presented.

A double-beam, ratio-recording spectrometer was modified so that it would compare radiant flux in beams emitted by a blackbody furnace and by a specimen at the same temperature, and record automatically a curve of normal spectral emittance of the specimen versus wavelength. Also, equipment for evaluating spectral reflectance over the wavelength range 1 to 15 microns, under conditions approximating normal illumination and hemispherical viewing, was designed and the necessary components procured. Preliminary designs of equipment were formulated for the automatic recording of spectral emittance data in a form suitable for direct entry into a computer, and for the online, automatic processing of the data. Either device would expedite the compilation from spectral data of total emittance, solar absorptance, or absorptance from any other source for which the spectral distribution of flux is known. Materials were selected by elimination tests for use in preparing working standards of spectral emittance, and such standards were prepared from polished platinum and oxidized Inconel and given the first two of triplicate calibrations for normal spectral emittance that are planned for the first of the working standards.

WADD TN 60-32.
AN APPARATUS FOR THERMAL ANALYSIS OF REACTIVE ALLOYS. Lt Donald J. Evans, Dr. Karl Strnat. April 1960. PB 171083. Order from OTS $2.75.

An apparatus for thermal analysis of small alloy samples has been developed and tested. It is to be used in an experimental study of binary alloys containing a rare metal as one constituent. The alloys to be investigated are highly reactive. The apparatus is suitable for phase diagram work at temperatures up to 1500°C. It is almost fully automatic and yields a continuous record of the sample temperature vs. time.

In this report, some general remarks on the method of thermal analysis are presented first, the apparatus is described, a few examples of heating and cooling curves obtained with the apparatus are shown, and the accuracy of the measurements is discussed on the basis of calibration runs.

Armour Research Foundation. WADC TR 56-222, Part III.
DETERMINATION OF EMISSIVITY AND REFLECTIVITY DATA ON AIRCRAFT STRUCTURAL MATERIALS - PART III. Techniques for Measurement of Total Normal Emissivity, Normal Spectral Emissivity at 0.665 Microns, Solar Absorptivity and Presentation of Results. O. Harry Olson, James C. Morris. AF 33(616)-3002. April 1960. PB 161839. Order from OTS $2.50.

Total normal emissivity is the ratio of total normal radiance emitted by a material to that emitted by an ideal blackbody at the same temperature. Parts I and II of this report described equipment and techniques employed for obtaining total normal emissivities of conducting materials, such as metals, at elevated temperatures. Part II describes equipment and techniques developed to make similar measurements on non-conducting materials such as refractories and glasses. Several samples are heated simultaneously in a tubular, wire wound furnace and presented successively in the field of view of a thermistor detector through a port in the furnace wall. The equipment and methods are described and data are presented for a list of materials. Low temperature measurements were made with equipment described in Parts I and II.

Normal spectral emissivity is the ratio of normal radiance emitted by a material to that emitted by an ideal blackbody at the same temperature at a given wave length. Most commonly, the wave length used is a narrow band at 0.665 microns and this is the sense in which the term is used in this report. Methods are described and results included.

An integrating sphere reflectometer was used to measure spectral reflectivity and transmission in the spectral range, 0.3 to 3.0 microns. This employs a Perkin-Elmer monochromator, a comparison integrating sphere, and a lead sulfide detector. Spectral reflectivity and transmission data are combined with solar spectral energy data to determine solar absorptivity at sea level and above the earth's atmosphere. Results are presented for a list of materials.

Perkin-Elmer Corporation. WADC TR 59-763.
DEVELOPMENT OF AN INFRARED, PRISM-GRATING, DOUBLE BEAM RECORDING SPECTROPHOTOMETER. Hamilton W. Marshall. AF 33(616)-5190. April 1960.

The design and construction of a Double Beam

Recording Wide Range Infrared Spectophotometer to automatically cover the range from 2.5 approximately 50 microns through four ranges is discussed. Experiments with gratings showed that to cover this range, gratings blazed at 6 microns through 30 microns, both used in the first and second orders were required. By selecting the beginning and end points of the regions as all exact integral multiples of one another, one wave number cam is used to drive the gratings thus assuring simple and accurate abscissa presentation. In order to separate the overlapping orders of the gratings, three prisms are used, NaCl, KBr, and CsI, with the prism monochromator double passed, since the dispersion from the prism must be large when wide slits are used to gain high signal-to-noise ratios. Because slit widths can become as great as 20 mm, an attenuator using two blades which may be tilted, similar to the action of a venetian blind, is used. A Golay cell was chosen as the detector since a large target was necessary to receive the energy from the wide slits.

Polytechnic Institute of Brooklyn. WADC TN 59-210.

EXPERIMENTAL INVESTIGATION OF THE DOWNSTREAM INFLUENCE OF STAGNATION POINT MASS TRANSFER. Paul A. Libby, Robert J. Cresci. AF 33(616)-5944. April 1960. ASTIA Document No. AD 216630. PB 161041. Order from OTS $2.25.

This report presents the results of an experimental investigation of the downstream influence of localized mass transfer in the stagnation region of a blunt body under hypersonic flow conditions. The coolant is injected through a porous plug coaxial with the center line of symmetry of the model. The tests were carried out in a Mach 6 wind tunnel with stagnation temperatures of approximately 1600°R and with a stagnation pressure of approximately 600 psia. Four different gases were injected over a range of mass flows. The heat transfer on the impermeable section was measured under isothermal wall conditions; for the higher rates of mass flow, adiabatic surface temperatures were also determined. The theoretical analysis of the boundary layer flow is investigated in order to establish the similarity parameters for the flow system. These parameters permit the extrapolation of the test results to other flow conditions provided laminar flow prevails. Helium is found to be the most efficacious coolant.

*Photochemistry Lab., GRD, AFCRL, Bedford, Mass.

AEROSPACE COMPOSITION MEASUREMENTS, C. Stergis. Project 6687(770), Internal.

The objective of this task is the determination of the composition of the upper atmosphere as a function of position and time. This includes the quantitative analysis of the molecular, atomic, and ionic constituents of the upper atmosphere, their relative concentrations as well as absolute densities. Direct composition measurements are made by suitable instrumentation in balloons, sounding rockets, and orbiting vehicles. The principal instruments to be used are: (1) Time-of-flight mass spectrometer. This instrument, with suitable modifications, will be used on all three types of vehicles: balloons, sounding rockets and orbiting vehicles; (2) The Paul-type mass spectrometer. This instrument is in the research and development stage but promises to be much superior to the time-of-flight instrument; and (3) Desorption spectrometer. This instrument, which is now being developed, has promise of operating even in interplanetary space.

*Cornell University, Ithaca, N. Y.

ANALYTICAL APPLICATIONS OF FLAME SPECTROSCOPY, W. D. Cooke. Project 9763 (802), Contract AF 49(638)-484; AFOSR, DCS.

This research is concerned with the development of new methods of excitation of atomic and molecular spectra and the application of such procedures to analytical problems. The procedures proposed will avoid some of the problems in flame chemistry, such as flame fluorescence spectroscopy, impact excitation, and others.

*Chemistry Research Lab., ARL, Dayton, Ohio.

ANALYTICAL CHEMISTRY OF SPECIAL ALLOYING METALS, R. W. Moshier. Project 7364(802), Internal.

This work emphasizes theoretical and experimental investigations of the properties of new chelated compounds of the metals: titanium, zirconium, hafnium, vanadium, niobium, tantalum, molybdenum and tungsten. It further determines the applicability of the properties of these new compounds for use in the development of a superior procedure for the analytical determination of each of these metals in the presence of eight of them.

*Owens-Illinois Glass Co., Toledo, Ohio.

ANALYTICAL METHODS FOR DETERMINATION OF VARIOUS CHEMICAL COMPONENTS IN REFRACTORY SYSTEMS, P. Close. Project 7022(802), Contract AF 33(616)-7431; ARL, MCB.

Various chemical analytical methods shall be investigated and/or developed for the following: (a) the amount of free magnesium oxide (MgO) in a heterogeneous mixture of ZrO_2 - MgO and the amount of MgO in solid solution in the ZrO_2 phase. The oxides, calcium oxide (CaO) and cerium oxide (CeO_2), will also be considered; (b) the amount of free metal (Ti, Zr, or Cr) in a heterogeneous mixture of ZrO_2 - (Ti, Zr, or Cr) and the amount of Ti, Zr, or Cr in solid solution in the ZrO_2 phase are to be determined. This phase of work will not be started until the ZrO_2 - MgO analyses have progressed satisfactorily; (c) chemical analysis of silicon-boron and boron-oxygen compounds; and (d) determination of the silicon and boron content of borosilicate phases in the presence of silicon and silicon-borides. Accuracy to three significant figures is desired for the various analyses.

*Huffman Microanalytical Labs., Wheatridge, Colo.

ANALYTICAL RESEARCH DATA FOR DETERMINING EVALUATION AND INTERPRETATION OF MOLECULAR STRUCTURES, E. W. D. Huffman. Project 7023(802), Contract AF 33(616)-6742; ARL, CRB.

This contract calls for the performance of quantitative chemical microanalyses and physical measurements on chemical compounds to be supplied by the Chemistry Research Branch, Aeronautical Research Laboratory, Wright Air Development Division. When standard analytical methods do not provide results which are within the limits of accuracy and precision of standard chemical practices, research shall be done to devise new methods to bring the results within such limits.

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* Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

*Dayton Univ., Ohio.
APPLICATION OF ION-EXCHANGE METHOD FOR ACCURACY OF ANALYSIS OF ALLOYS, S. L. Everslage. Project 7364 (802), Contract AF 33(616)-5612; ARL, CRB.

Research is being performed on the application of the ion-exchange method for the separation of the metals occurring in special-property alloys. The contractor will present a complete procedure for the quantitative separation of the metals found in special property alloys. To date the following mixtures of metals have been quantitatively resolved: (1) iron, manganese; (2) iron, chromium; (3) iron, titanium; (4) iron, cobalt, nickel; (5) iron, cobalt, aluminum; (6) cobalt, nickel, aluminum; (7) iron, cobalt, nickel, aluminum.

*Washington Univ., St. Louis Mo.
APPLICATION OF MAGNETIC RESONANCE TO SOLID STATE PHYSICS, R. E. Norberg. Project 9760(802), Contract AF 49(638)-808; AFOSR, DSS.

This research will make use of nuclear magnetic resonance and electron spin resonance measurements to learn more about the properties of solids. Four experimental programs are planned: (1) Premelting phenomena will be studied in sodium and if possible in other metals by observation of resonance line width behavior. (2) Spin resonance of conduction electrons in metals will be studied in such metals as the alkalis and beryllium which have been highly purified and then in some of their alloys. The data in this area are important in determining the extent and nature of electron interactions in metals. (3) The coupling between electron and nuclear spins will be studied in such "dilute" metals as sodium dissolved in liquid ammonia. (4) The electron-nuclear spin coupling and nuclear-nuclear coupling through electrons will be studied by measurement of pulsed nuclear magnetic resonance in spinning solids.

*Colorado Univ., Boulder.
ATMOSPHERIC COMPOSITION, W. A. Rense. Project 6687(770), Contract AF 19(604)-5533; GRD, PL.

The objective is to continue studies and experimental investigation of the fundamental properties of reflectivity and transmission of radiation by thin films in the vacuum ultra-violet region of the spectrum, lying between 200 and 2000 Angstroms.

*Yale Univ., New Haven, Conn.
ATOMIC BEAM MAGNETIC RESONANCE AND QUANTUM ELECTRODYNAMICS, V. W. Hughes. Project 9767(803), Contract AF 49(638)-545; AFOSR, DPS.

The purpose of this project is to study the electronic and nuclear properties of metastable and excited states of simple atoms and meson complexes by atomic beam resonance methods. Also studied are the formation and decay of short-lived excited complexes such as positronium and muonium. This work will give cross-section variations between ground and excited states of light- and medium-weight elements and molecules important to fuel energetics, particle decay processes and abnormal cross-sections of excited complexes.

*Cornell Univ., Ithaca, N. Y.
ATOMIC PHENOMENA OCCURRING ON AND NEAR THE SURFACE OF SOLIDS, B. M. Siegel. Project 9761(802), Contract AF 18(600)-674; AFOSR, DSS.

Our present knowledge of the structure of surfaces of solids, and of the initial reactions of a surface with corroding elements, is very fragmentary and, in many instances, contradictory. This work is undertaken in the belief that basic progress awaits studies with "clean" surfaces under conditions of ultra-high vacuum (less than 10^{-9} mm. Hg) and using high-resolution electron diffraction. Thin films of Cu, Ni, Fe, W and alloys will be studied and compared to single crystal of the same materials. The information obtained from the high surface to volume ratio thin films will finally be correlated to the bulk surface properties of the single crystals by electron microscopy using pre-shadowed carbon replicas.

*Arkansas Univ., Fayetteville.
ATOMIC SPECTROSCOPIC INVESTIGATION OF NUCLEAR PROPERTIES, R. H. Hughes. Project 9767(803), Contract AF 49(638)-547; AFOSR, DPS.

High-stability spectrographic equipment will be constructed for the measurement of weak spectral lines of stable isotopes of light- and medium-weight atoms. Isotopic shifts in even-even and even-odd neutron-proton isotopic neighbors will be analysed for particle effects, spectral shifts, and binding energy shifts. This will permit formulation of stability criteria and excitation levels of elements important as primary radiation and as high temperature alloy materials.

*Wisconsin Univ., Madison.
CHARGE TRANSFER COMPLEXES, M. Kosower. Project 9760(802), Contract AF 49(638)-282; AFOSR, DCS.

The research involves a study of the charge-transfer spectra of complexes of simple halide donors with transition metal ions. The findings should provide accurate information on the exchange contribution to molecular binding energy.

*Stanford Univ., Calif.
CHEMICAL APPLICATIONS OF NUCLEAR MAGNETIC RESONANCE, R. A. Ogg. Project 9760(802), Contract AF 49(638)-286; AFOSR, DCS.

The research will apply techniques of high resolution nuclear magnetic resonance to obtain information about molecular structure of chemical substances and of the changes these undergo during reaction. This approach should permit detection and study of short-lived reaction intermediates. Prime emphasis will be given to inorganic compounds of nitrogen and boron.

*Arizona Univ., Tucson.
COMPLEXING AGENTS (CHELATING AGENTS) FOR THE METALS NIOBIUM AND TANTALUM, H. Freiser. Project 7364(802), Contract AF 33(616)-6415; ARL, CRB.

Studies will be performed on the preparation and properties, especially the stability, of complexes of niobium III, with the aim of finding one or more such complexes having stability under laboratory working conditions. The study will extend to the application of separation techniques with the ultimate goal of separating niobium and tantalum one from another in a quantitative degree. Very little is known about the properties of niobium in the valence state of three except in high-temperature metal oxide studies. Niobium (III) exists in aqueous solution and is very reactive, being readily oxidized even by air to the pentavalent state. Meager knowledge of niobium (III) shows it

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

to have much different properties in aqueous solution than niobium (V) the latter being very similar to tantalum (V). Metallic complexes with organic and inorganic agents have great advantage in analytical chemistry because the resultant complex reacts differently with other reagents. A systematic study of the reaction of the better known complexing agents with niobium (III) will be made, prior consideration being given to those agents which have been found to stabilize the lower valence states of one or two other metals.

*Purdue Univ., Lafayette, Ind.
COORDINATION KINETIC CHROMATOGRAPHY, D. W. Margerum. Project 9763(802), Contract AF 49(638)-60; AFOSR, DCS.

The feasibility of using coordination kinetics as a general tool for separation and analysis of metal ions will be determined, along with a study of the coordination reaction rates and the variables affecting them. This research might lead to a method for analysis, separation and purification of metals.

*Sylvania Electric Products, Inc., Bayside, N. Y.
DEVELOPING A METHOD OF ANALYZING CONCENTRATIONS OF IMPURITIES IN SILICON CARBIDE, G. Morrison. Project 5620(802), Contract AF 19(604)-4944; ERD, EML.

The object of this project is the investigation of pre-concentrating techniques in order to lower the limits of detectability of trace impurities in a silicon carbide matrix. The method makes use of electro-kinetic phenomena in order to separate and concentrate an impurity ion in solution and subsequent emission spectrography for qualitative and quantitative determination. This project will add to the knowledge of silicon carbide chemistry, and throw light on the nature of the chemical imperfections in currently available silicon carbide.

*Columbia Univ., New York.
ELECTRON SPIN RESONANCE IN GASES, G. K. Fraenkel. Project 9763(802), Contract AF 49(638)-520; AFOSR, DCS.

This research is concerned with four applications of the technique of electron spin resonance: (1) relaxation phenomena of free radicals; (2) paramagnetic resonance of gases; (3) double resonance experiments; and (4) studies of complex ions. The spin resonance technique is extremely sensitive and is useful in detecting, identifying and studying free radicals.

*Texas A. and M. Research Foundation, College Station.
ELECTRONIC SPECTRA OF SIMPLE MOLECULES IN VACUUM ULTRAVIOLET, J. B. Coon. Project 9751 (801), Contract AF 49(638)-816; AFOSR, DPS.

This work will consist of the study of the absorption spectra of selected polyatomic molecules such H_2O, D_2O, HH_3, ND_3, H_2CO_2 and C_2H_4 in the region from 1000 to 2000 Angstroms. These data will be correlated with current theories of molecular structure and calculations concerning the anharmonicities and shapes of the potential functions will be carried out.

*Arkansas Univ., Fayetteville.
EXPERIMENTAL DEVELOPMENT OF A NEW METHOD FOR MEASURING SURFACE ROUGHNESS ON A MICROSCOPIC SCALE, M. K. Testerman. Project 7022 (802), Contract AF 33(616)-5541; ARL, CRL.

Methods for the determination of molecular scale roughness are under investigation. Two experimental approaches are being used. The first is the adsorption of radioactive isotopes on surfaces with varying degrees of roughness. The surfaces are subsequently analyzed with a scintillation counter. The second method involves measurement of surface roughness by polarization capacity. Capacities of metal surfaces are determined in aqueous and nonaqueous electrolytes. Surface roughness determinations on film-free metal surfaces will be attempted by exclusion of both oxygen and water from the measuring system. Capacity measurements in potassium iodide-glycerol solutions indicate that the system can be used for surface roughness determinations.

*Thermal Radiation Lab., GRD, AFCRL, Bedford, Mass.
FAR INFRARED INTERFEROMETRIC SPECTROSCOPIC TECHNIQUES, G. A. Vanasse. Project 8693(804), Internal.

A large lamellar-grating type two-beam interferometer has been assembled for studies in improving the resolution and signal-to-noise of far infrared spectral information, without increasing recording time. The data is obtained as the auto-correlation function of the spectrum, and the various techniques for transforming these to spectra are being studied.

*Johns Hopkins, Baltimore, Md.
FAR INFRARED RESEARCH USING INTERFEROMETRIC MODULATOR, J. Strong. Project 9768(803), Contract AF 18(600)-1307; AFOSR, DPS.

A special interferometer for the far infrared spectrum has been built by the principal investigator and is being used to study widths and strengths of ammonia lines at about 500 microns and will be used to study submillimeter harmonics of Klystrons and other microwave generators, as well as cyclotron resonance. A spectroscopic study has been made of the pressure broadening of the pure rotation line of ammonia at 20 cm^{-1} and that of hydrogen chloride at 40 cm^{-1}. In addition, the transmission spectra of several discs of sapphire have been measured. The channel spectrum arising with these sapphire plates has been analyzed for index of refraction and for extinction coefficient. This channel spectrum shows clearly that sapphire is birefringent. It is planned to use a form of this interferometric modulator for possible determinations of the water vapor content of planetary atmospheres (Venus, Mars) from a balloon in the upper reaches of the earth's atmosphere.

*Ohio State Univ., Columbus.
FAR INFRARED SPECTROSCOPY, E. E. Bell. Project 8603(804), Contract AF 19(604)-4119; GRD, TRL.

The goal of this research is the study of the far-infrared spectral region, that is, the region extending beyond 50 microns. This involves the design and construction of spectroscopic apparatus and accessories for this region, but is primarily aimed at studies of the molecular absorption in this region by molecules of the atmosphere.

*Texas Univ., Austin.
HIGH PRESSURE EFFECTS ON ELECTRONIC SPECTRA, W. W. Robertson. Project 9763(802), Contract AF 49(638)-35; AFOSR, DCS.

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

The following studies will be made to ascertain the effect of high pressure upon electronic absorption spectra: (1) determination of U.V. absorption frequencies of large molecules in vapor state, from experimental determinations in liquid state, at high pressure; (2) characterization of electronic transitions, from pressure dependent frequency shifts; (3) determination of oscillator strength; (4) investigation of the nature of intermolecular forces; and (5) examination of the connection between statistical and collision theories.

*National Bureau of Standards, Washington, D. C.
HIGH RESOLUTION FLAME STUDIES, E. K. Plyler. Project 8603(804), Contract CSO&A 60-530; GRD, TRL.

The goal of this research is high resolution precision spectroscopy of combustion flames in emission and absorption. The near infrared (1-5 microns) is the region of interest in the electromagnetic spectrum. Studies of the effects of pressure-broadening of absorption lines of H_2O at 3.15 microns continue to be made. A precision wavelength calibration standard system is being established for use in the infrared region from 2-38 microns with high resolution spectra.

*Mellon Inst. of Industrial Research, Pittsburgh, Pa.
HIGH RESOLUTION INFRARED SPECTROSCOPY OF SOLIDS, R. M. Hexter. Project 9760(802), Contract AF 49(638)-542; AFOSR, DCS.

The infrared spectra of pure and mixed crystals of simple molecules such as methane, silane, germane, water, ammonia and the methyl halides are being investigated for the purpose of exploring motions of these molecules where situated in the close proximity dictated by the crystal arrangement characteristic of the solid state. Rotation of water and ammonia molecules dissolved in solid inert gases has been demonstrated conclusively. The infrared spectra of these solids yield important information of the forces acting between molecules and on the effects of these forces on molecule oscillations.

*Liège Univ. (Belgium).
HIGH RESOLUTION INFRARED STUDIES, M. V. Migeotte. Project 8603(804), Contract AF 61(514)-962; GRD, TRL.

This contract supports three separate subgroups of the University of Liège. One group is working at Arosa, Switzerland, on infrared problems associated with atmospheric ozone; another small group is working at the University of Liège on high resolution infrared problems; and the main group works at the Jungfraujoch Scientific Station in Switzerland. At the Joch, studies are underway on the IR spectra of sunspots, on interferometry in the near and far infrared, and on high resolution solar spectrum studies.

*Thermal Radiation Lab., GRD, AFCRL, Bedford, Mass.
HIGH RESOLUTION MOLECULAR STUDIES, J. S. Garing. Project 8603(804), Internal.

Many improvements have been incorporated into the high resolution double-beam grating spectrophotometer of the Geophysics Research Directorate, including the addition of a wavelength calibration system. The instrument is used to study molecular absorption bands in the 3 to 20 micron spectral region. Molecular spectra of the oxides of nitrogen, N_2O and NO, of CS_2, which is very similar to CO_2, and the 9 micron bands of ozone have been obtained. Future studies will include other molecules of atmospheric interest.

*Battelle Memorial Inst., Columbus, Ohio.
INFRARED ABSORPTION SPECTRA OF SELECTED AROMATIC COMPOUNDS, R. Jacobson. Project 7364(802), Contract AF 33(616)-7162; WADD, MC.

This research program is designed to gain the maximum information about the sensitivity of long wavelength infrared absorption for characterization of substituents on aromatic rings, and for elucidation of structural differences existing between the solid and liquid states of aromatic compounds. Complete vibrational assignments have been made so far for phenol, p-xylene, p-cresol and hydroquinone.

*Weizmann Inst. of Science. (Israel).
INFRARED DISPERSION OF GASES, J. H. Jaffe. Project 8603(804), Contract AF 61(052)-57; GRD, TRL.

This project is concerned with the infrared dispersion of gases in the neighborhood of infrared absorption bands. Apparatus has been designed and constructed which records dispersion curves automatically, instead of on a point-to-point basis. The data is being interpreted to obtain values for the intensity and half widths of spectral lines. Primary interest is focused in the lithium fluoride region of approximately 1 to 7 microns. Studies have been made on gases with widely spaced lines such as HCl. As suitable techniques are developed the emphasis will shift to the atmospheric gases such as CO_2, H_2O, N_2O, and CH_4.

*Pittsburgh Univ., Pa.
INFRARED LINE WIDTHS, W. M. Benesch. Project 8603 (804), Contract AF 19(604)-6138; GRD, TRL.

This effort is directed toward a better understanding of the line width of individual rotational absorption lines as a function of the vibration-rotational state of the molecule. Toward this end, a vacuum spectrograph is being finished which has an unusual hydraulically operated grating wavelength drive system and also makes use of a Michelson-type interferometer to measure the angle of rotation of the grating. The study of line widths includes the use of very broadening gases. Also program of observation line widths of molecules in electrodeless discharges has been started.

*Akron Univ., Ohio.
INFRARED SPECTRAL LINE INTENSITIES, H. M. Hanson. Project 8603(804), Contract AF 19(604)-6126; GRD, TRL.

The goal of this research is a theoretical and experimental study of the intensities of infrared spectral lines in vibration-rotation bands of atmospheric molecules.

*Polytechnic Inst. of Brooklyn, N. Y.
INFRARED SPECTRAL STUDY OF INTRAMOLECULAR AND INTERMOLECULAR FORCES, R. P. Bruman. Project 7364(802), Contract AF 33(616)-6778; WADD, MC.

Intermolecular interactions and the results of these on infrared vibrational spectra are being investigated. The work is being conducted along two lines; weak interactions, such as those between tetrahedral molecules in the liquid phase, are being examined and strong interactions, such as hydrogen bonding are receiving attention. In the spectrum of CF_4 in C Cl_4 solution there appears to be some splitting of the normally degenerate vibrations of this tetrahedral molecule. This may be due to the appearance of hot

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research Fiscal Year 1960.

bands and a further investigation of the vapor spectrum at low temperatures is being carried out. Hydrogen bonding is being studied through the use of dihydroxyanthraquinones. Since the bonding groups are fixed in space at positions which can be determined from and knowledge of bond angles and lengths, these molecules are well suited to a study of this type. Attempts are being made to purify 2,6 dihydroxyanthraquinone (anthraflavic acid). It is expected that this work will not only aid in the interpretation of infrared spectra but will help explain certain physical properties (viscosity and vapor pressure) of materials.

*Thermal Radiation Lab., GRD, AFCRL, Bedford, Mass.
INFRARED VIBRATION-ROTATION BAND MEASUREMENTS, B. Schurin. Project 8603(804), Internal.

The high resolution double-beam double-pass grating spectrophotometer of the Geophysics Research Directorate will be used as the basic instrument for measurements of atmospheric molecular band intensities. Auxiliary apparatus is under design and construction to facilitate the use of the spectrophotometer for such measurements. These data are used to obtain radiation transfer properties of atmospheric molecules, such as the oxides of nitrogen.

*Electronic Tech. Lab., WADD, Dayton, Ohio.
INTERACTION OF ENERGY LEVELS IN LIQUIDS, L. Politzer. Project 4150(803), Internal.

A microwave spectrometer for investigation of the dielectric behavior of liquids is being constructed. It is intended to be a broadband unit. When completed it will be used to investigate absorption and transmission of microwave energy in non-polar solvents with various solutes.

*Texas Univ., Austin.
INSTRUMENTATION FOR PLANETOLOGY, W. H. Hartwig. Project 7698(770), Contract AF 19(604)-5717; GRD, RIL.

Theoretical and experimental studies will be directed toward determining the potential of the photodielectric and photomagneto-electric effects for infrared sensors.

*New Mexico Coll. of A. and M.A., State College.
INSTRUMENTATION FOR PLANETOLOGY, B. A. Smith. Project 7698(770), Contract AF 19(604)-5535; GRD, RIL.

The objective is to determine possible methods of improving resolution of astronomical photographs, to construct a prototype in image aberration compensation device and to test these methods.

*Paris Univ. (France).
IONIC THINNING OF SPECIMENS FOR ELECTRON MICROSCOPY, R. Castaing. Project 7022(802), Contract AF 61(052)-338; WADD, MC.

This work will consist of an extensive study of the "ionic thinning" of metallic or non-metallic specimens. The main purpose of the work shall be to understand the true process involved in "ionic thinning" so that an optimum surface condition can be obtained for use with transmission electron microscopy.

*Picker X-Ray Corp., Cleveland, Ohio.
LOW ENERGY X-RAYS, E. White. Project 7364(802), Contract AF 33(616)-7177; WADD, MC.

Basic research on methods of studying X-ray spectra in the 15 to 50 Angstroms region is sought. Techniques and equipment developed will be applied to the study of L spectra shifts and fine structure for such metals as iron, manganese, and titanium, and K spectra of such elements as nitrogen, oxygen and carbon.

*New York Univ., N. Y.
LOW-FREQUENCY ABSORPTION SPECTRA, Y. Beers. Project 9768(803), Contract AF 49(638)-259; AFOSR, DPS.

The purpose of this investigation is to extend the studies of absorption of vapors and gases to the low microwave and ultra-high frequency regions and to develop instruments and techniques for using this radiation as a tool in studying the properties of gases, liquids and vapors. The rotational transition, the doubling transitions, the pure quadrupole transitions and the inversion spectra of heavy ammonia will be studied. The transitions will be checked as to their possibilities as frequency standards. The principal object of this research is the study of molecular spectra at frequencies below 6,000 megacycles, actually down to about 300 mc. This involves techniques of high resolution spectroscopy. One improvement introduced by Professor Beers is the use of a parametric amplifier which is believed to be the first such use in spectroscopy. In addition to the work on gases, research on nuclear resonance in solids has been initiated.

*Sheffield Univ. (Gt. Brit.).
LOW LEVEL EXCITATION AND ENERGY TRANSFER, G. Porter. Project 6694(750), Contract AF 61(052)-32; GRD, PL.

Studies are being performed on the radiationless transitions in molecules where the triplet state is known to occur. The effect of additions such as paramagnetic ions is being investigated. The kinetics of triplet conversion is being investigated by flash photolysis and photoelectric spectrometry.

*Georgia Inst. of Tech., Atlanta.
MASS SPECTROGRAPHIC STUDY OF ION-MOLECULE REACTIONS, E. McDaniel. Project 9767(803), Contract AF 18(600)-1524; AFOSR, DPS.

This research deals with ion identification in gaseous electronics experiments involving complex initial ionization processes which are not well understood. Following ionization, the drifting ion may change its identity in a number of ways such as charge transfer, chemical reaction, clustering through attachment of molecules to the ions, dissociation, and interaction with metallic surfaces. The problem of identification following such changes requires investigation. Present work covers precise mass spectrographic studies of ion-molecule reactions under thermal equilibrium conditions. Research of this nature had not been performed previously as far as is known. Theoretical interpretation of the experimental results is being undertaken. In addition, studies are being initiated on the transport properties of a fully ionized gas.

*Pennsylvania State Univ., University Park.
MEASUREMENT OF BORON AND OTHER IMPURITIES IN SEMICONDUCTOR MATERIALS BY MASS SPECTROMETRY, L. Herzog. Project 5620(802), Contract AF 19(604)-5538;

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research Fiscal Year 1960.

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ERD, EML.

The object of this research is the investigation of mass spectrometry as an analytical tool with a sensitivity in the part per billion range and better. This sensitivity is required for the determination of the level of purity of solid state semiconductor matrices. Since other analytical techniques do not have this required sensitivity for the determination of boron in silicon, this system has been chosen for study. Specific items to be investigated include the use of the most efficient detector, the reduction of background in the detector due to gas scattering, the use of the most efficient technique for ionising the source material, and the use of the best collector for intensity and ratio determination.

*North Carolina Univ., Chapel Hill.
METAL ION INTERACTION WITH COMPLEXING AGENTS, C. N. Reilley. Project 9763(802), Contract AF 49(638)-333; AFOSR, DCS.

This is a study of the correlation between structures of new and of well-established analytically important chelonates and their reactivity with various metal ions from both a thermodynamic and kinetic viewpoint. The resulting analytical applications and techniques will have value where rapid and effective methods for analysis of macro and trace materials are needed.

*Max-Planck-Institut für Chemie, Mains (Germany).
METEOR PHYSICS, H. Wanke. Project 7667(770), Contract AF 61(052)-334; GRD, PL.

Investigations will be made of radioactive spallation products of cosmic rays in meteorites. The geological age of meteorites will also be determined.

*Johns Hopkins Univ., Baltimore, Md.
MICRO-CATALYTIC CHROMATOGRAPHY, P. H. Emmett. Project 9761(802), Contract AF 18(603)-129; AFOSR, DCS.

A study of catalytic reactions by an entirely new technique and procedure. Micro quantities of reactants placed in a stream of carrying-gas flowing over a catalyst and through a chromatographic column will enable one to determine the exact identity and amounts of all of the reaction products in a very short time. The method will be extended to the study of high pressure reactions.

*Central State College, Wilberforce, Ohio.
MICRO-DETERMINATION OF ORGANIC FLUORINE, H. Johnson. Project 9763(802), Contract AF 18(600)-1573; AFOSR, DCS.

The objective of this research is the establishment of a simpler, more rapid, and more generally applicable analytical determination for fluorine in organic compounds.

*Duke Univ., Durham, N. C.
MICROWAVE SPECTRA OF ATMOSPHERIC AND OTHER GASES, W. W. Gordy. Project 9768(803), Contract AF 49(638)-765; AFOSR, DPS.

The purpose of this research is to develop instrumentation and experimental techniques for studying the effects of extreme temperatures and damaging radiation on the microwave spectra and magnetic resonance of matter. Some of the materials to be considered are hormones, vitamins, proteins and other biological substances; flames, crystals, liquids (possible fuels), gases and free radicals. The production and measurement of sub-millimeter waves will also be studied and the superconductivity and dielectric dispersion of substances at these wave-lengths will be measured.

*Washington Univ., St. Louis, Mo.
MOLECULAR ELECTRONIC STATES, W. T. Simpson. Project 9760(802), Contract AF 49(638)-677; AFOSR, DCS.

By means of absorption spectrography, polarized emission, absolute intensity measurements, and reflection measurements, the experimental work on electronic spectra of molecules and molecular crystals is being performed. Correlative quantum mechanical calculations will be carried out to enhance the theoretical understanding of the electronic processes through the comparison between theory and experiment. This research will test the validity of quantum mechanical predictions of the behavior of molecules containing light atoms, such as beryllium.

*Michigan Univ., Ann Arbor.
MULTIPLE-BEAM INFRARED INTERFEROMETRY, C. W. Peters. Project 8603(804), Contract AF 19(604)-2071; GRD, TRI.

A Fabry-Perot (multiple-beam) type interferometer has been built for the near infrared. The ultimate goal is to obtain sensitivity at very low levels of luminosity in the 3.5 micron region. Studies have also been continued on the production of electric field-induced rotation-vibration absorption in homonuclear diatomic molecules, such as H_2 and D_2, and in the direct observation of the derivative of infrared spectra.

*Illinois Univ., Urbana.
NEW SPECTROCHEMICAL METHODS, H. V. Malmstadt. Project 9763(802), Contract AF 18(603)-137; AFOSR, DCS.

This research concerns the development of new emission and absorption spectrochemical methods, techniques and applications, such as molten state excitation technique of emission spectrochemical analysis, and absorption and phosphorescent spectra under high pressure. Some of these analytical techniques have already been applied to the determination of trace metals in oils and minor constituents in titanium-sponge and alloys.

*Florida State Univ., Tallahassee.
NMR STUDY OF THE STRUCTURE AND REACTIVITY OF MOLECULES, R. A. Kromhout, E. Grunwald. Project 9760(802), Contract AF 49(638)-278; AFOSR, DCS.

The program involves a comprehensive study of the NMR spectra of amines and acid salts. Specifically, rates and mechanisms of protolysis (i.e., hydrogen transfer) involving N-H bonds, rates of complex formation of amines with metal ions and with electron acceptor acids and the complexing of amines or ammonium ions with other molecules will be studied.

*Photochemistry Lab., GRD, AFCRL, Bedford, Mass.
NON-OPTICAL SPECTROMETRY, H. E. Hinteregger. Project 6688(770), Internal.

The object of this work is the detection and analysis of extreme ultraviolet and x-ray fluxes for cases that are

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research Fiscal Year 1960.

not conducive to measurements with monochromators (i.e., optical dispersion). Monochromators, at least those for the wavelength range under consideration here, are probably useful only for the measurement of radiations from a point-like source at which the instrument must be kept aimed with a suitable "pointing control." Partially or fully non-optical techniques of extreme ultraviolet and x-ray measurements do not require a pointing control and they can be applied to radiation detection with large aperture and over a wide range of angles of incidence. Therefore, the use of existing techniques of this type and the development of improved or new ones fill an important experimental gap in exploring and surveying the aerospace or radiation environment.

*Rutgers Univ., New Brunswick, N. J.
NUCLEAR MAGNETIC RESONANCE INVESTIGATION OF THE PROPERTIES OF MATTER, H. C. Torrey, H. Y. Carr. Project P 9760(802), Contract AF 49(638)-755; AFOSR, DSS.

The investigation will be concerned with such problems as: (a) dynamic polarization of nuclear spins at low temperatures and the attainment of extremely low temperatures; (b) a study of the Overhauser effect in liquids; (c) nuclear magnetic resonance studies of the solid, liquid, and gaseous states of monatomic systems; (d) use of nuclear magnetic resonance techniques in fundamental studies of self diffusion and liquid structure of water; (e) the application of steady-state free precession techniques to the measurement of nuclear spin relaxation times and self diffusion coefficients; (f) the use of intense rf pulses to eliminate the effect of the inhomogeneity of magnetic fields on response width in nuclear magnetic resonance; development of a high Q filter.

*Texas A and M Research Foundation, College Station.
NUCLEAR MAGNETIC RESONANCE STUDIES OF BINARY SOLUTIONS, M. Eisner. Project 9751(801), Contract AF 18(600)-1300; AFOSR, DPS.

This project concerns the use of nuclear magnetic resonance to study the interactions of two liquids on the microscopic level. This is aimed at a better understanding of the liquid state. The main point is that one is observing the reaction of a microscopic magnet to an applied magnetic field. The microscopic magnet, however, is also immersed in the magnetic field of the surrounding nearest neighbor magnetic dipoles and the rotations or diffuse collisions are affected by this field.

*Liège Univ. (Belgium).
NUCLEAR QUADRUPOLE RESONANCE IN SOLIDS, J. Duchesne. Project 9760(802), Contract AF 61(052)-167; AFOSR, DCS.

This is a study of the nature of chemical bonds and of the interaction of high energy radiation with matter by using nuclear quadrupole resonance techniques on selected organic and inorganic halogen compounds.

*Stanford Univ., Calif.
PARAMAGNETIC RESONANCE, G. Pake. Project 9751 (801), Contract AF 18(603)-131; AFOSR, DPS.

The objective of this research is to develop methods of measuring electronic and nuclear spin lattice relaxation times at extremely low temperature and to analyze these data in terms of the exchange interactions, nuclei electron interaction and dipolar interactions between pairs of electrons and pairs of nuclei. These methods will be tested by applying them to studies of free radicals. More careful studies of the Overhauser effect as a possible means of achieving nuclear alignment at low temperature will be carried out.

*Kansas Univ., Lawrence.
PHOTOMETRIC TITRATION OF ORGANIC GROUPS, C. A. Reynolds. Project 9763(802), Contract AF 49(638)-472; AFOSR, DCS.

This project deals with the adaptation of the photometric titration technique to the analysis of several organic functional groups, thereby improving the speed and accuracy of this type of analysis. The general method of attack will be the same regardless of the specific titration reaction employed.

*Columbia Univ., New York.
PHYSICS AND CHEMISTRY OF GASES AT HIGH TEMPERATURES, P. Kusch. Project 9767(803), Contract AF 49 (638)-557; AFOSR, DPS.

High-temperature, high-resolution atomic and molecular beam sources and spectrometers have been constructed for the investigation of the physics and chemistry of gases and of simple molecules having appreciable vapor pressures only at elevated temperatures. Studies will also be made of single crystal evaporation processes and the formation of metastable states from an initial $1_S O$ state. This work is expected to provide new dissociation data and improved theoretical models of phase equilibria, internal interactions and population balances in metastable states.

*Minnesota Univ., Minneapolis.
POLAROGRAPHY AND AMPEROMETRY IN NON-AQUEOUS SOLVENTS, I. M. Kolthoff. Project 9763(802), Contract AF 49(638)-519; AFOSR, DCS.

This is a systematic study of the polarography and amperometric behavior of inorganic constituents in non-aqueous solvents with emphasis on analytical applications. Procedures may be developed for the polarographic and amperometric determination of those ions, like magnesium and the rare earths, which cannot be determined in aqueous media.

*Johns Hopkins Univ., Baltimore, Md.
POST-DISCHARGE AFTER-GLOW SPECTROSCOPY, D. E. Kerr. Project 9767(803), Contract AF 18(600)-363; AFOSR, DPS.

Studies are being carried out on the ionization and related mechanisms leading to the formation of the stationary state in gaseous discharges, and of the deionization processes in the post-discharge after-glow. New equipment for improved time-resolved spectra of after-glow phenomena have been constructed. Time-resolved atomic line spectra and molecular band structure of light gases are investigated to relate electron density, ambipolar diffusion, and pressure effects to deenergization or relaxation processes. Improved theories of relaxation from excited states with radiative transfer are expected from this research.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research Fiscal Year 1960.

*Syracuse Univ., N. Y.
PRESSURE-BROADENING IN THE FAR INFRARED, N. Ginsburg. Project 8603(804), Contract AF 19(604)-2443; GRD, TRL.

This program involves research in the far infrared (initially 40μ to 100μ) on the structure and spectral line shape of molecules of atmospheric interest. Particular attention is being placed on the measurements of line widths in the pure rotational spectrum of water vapor as a function of the water vapor pressure with and without pressure broadening by foreign gases.

*Kent State Univ., Ohio.
PROTON-PROTON SEPARATIONS IN HYDRATES, J. McGrath. Project 9768(803), Contract AF 49(638)-168; AFOSR, DPS.

This research will try to determine the effects of neighboring atoms and molecules on the resonance characteristics of a paramagnetic molecule. These data will be collected from similar crystals by studying the proton-proton separation. The crystalline hydrates will be studied first. The various possible origins for shifts will be separately analyzed.

*Huffman Microanalytical Lab., Wheatridge, Colo.
PYRIDYLAZONAPHTHOL COMPOUNDS AS SPECTROPHOTOMETRIC REAGENTS, E. W. Huffman. Project 7364(802), Contract AF 33(616)-3964; ARL, CRB.

Research is being performed in applying the reagent, pyridylazonaphthol, to the spectrophotometric determination of the metals niobium and tantalum and those metals with which they are always associated. The results to date are: (1) conditions have been determined whereby the pyridylazonaphthol reacts with each of the eight metals: titanium, zirconium, hafnium, vanadium, niobium, tantalum, molybdenum and tungsten; (2) a colorimetric method has been developed for the determination of vanadium in the presence of iron; and (3) usable methods, for the determination of each of the metals zirconium and hafnium, and for the determination in their mixtures, have been developed.

*Massachusetts Inst. of Tech., Cambridge.
RAPID MEASUREMENT OF GAS TEMPERATURES IN THE RANGE OF 4000°K TO 8000°K, H. T. Hottel. Project 7021(802), Contract AF 33(616)-6570; WADD, ML.

This investigation is directed toward research into methods for the measurement of the gas temperatures of air in the range 4000°K to 8000°K at pressures of 0.01 to 10 atmospheres as might be produced in hypersonic flight induced boundary layers and in the tail flames of air stabilized plasma jets. Several methods including microwave radiometer noise measurements as applied to stellar temperatures, microwave absorption measurements and plasma probe measurements suggest themselves as possible methods for quickly measuring the gas temperature of high temperature air. It is therefore the objective of this program to carefully review the literature and determine the feasibility of various possible methods and to develop at least one of the selected methods into a laboratory tool suitable for measurement of the gas temperatures and apply it to the measurement of gas temperatures in an electrically driven plasma jet or a shock tube.

*Hebrew Univ. (Israel).
SOFT X-RAY AND FAR ULTRA-VIOLET SPECTROSCOPY, E. Alexander, B. S. Fraenkel. Project 9763(802), Contract AF 61(052)-347; AFOSR, DSS.

An apparatus for generating radiant energy in the soft x-ray and far ultra-violet region (10 to 1000A) has been developed. It is planned to exploit this wave-length region by performing experiments in vacuo in five areas, namely: (1) fluorescent soft x-ray solid state spectroscopy. A study of emission and absorption spectra at very low temperatures using fluorescent radiation techniques may help to check the theory of superconductivity in the solid state; (2) soft x-ray spectroscopy will be extended to elements not yet investigated, e.g., gallium. This may add to the knowledge of the electron band structure of the elements; (3) absorption curves of the optimum thickness can be made with different orientations. This would offer valuable information about the crystalline zone structure and its geometrical dependence; (4) soft x-ray spectroscopy of organic crystals by the fluorescent radiation technique will avoid destruction of the crystal by direct electron bombardment and may contribute to knowledge of problems of energy exchange; (5) the spectroscopy of multiple-ionized gases with the sliding spark method may contribute to the solution of problems concerned with thermonuclear energy.

*Cornell Univ., Ithaca, N. Y.
SOLUTE SPECIES IN CONCENTRATED AQUEOUS SOLUTIONS, R. A. Plane. Project 9760(802), Contract AF 49(638)-279; AFOSR, DCS.

Experiments are being conducted to improve understanding of concentrated aqueous solutions. The hydration of ions will be studied by employing O^{18}, an oxygen isotope, as a tracer. The study of complex ions in solution by Raman spectra is being carried forward. Oxygen isotopes will be used to characterize solute species, such as chromic polymers and thorium compounds. Further studies of polymers in solution are being made.

*Johns Hopkins Univ., Baltimore, Md.
SPECTRA OF TRIPLY CHARGED RARE EARTH IONS, G. H. Dieke. Project 9768(803), Contract AF 49(638)-535; AFOSR, DPS.

The purpose of this research is to study the spectra of crystals in such substances where sharp spectrum lines occur. These data will be used to try to determine the nature of the forces responsible for the properties of matter. The spectra of rare earths will be recorded for various states of ionization. The wavelengths and intensities will be measured. The results will be analyzed and attempts made to correlate these spectra with the spectra of these atoms in a crystal lattice.

*Indiana Univ., Bloomington.
SPECTROMETRIC STUDIES OF FAST REACTIONS, E. J. Bair. Project 9760(802), Contract AF 18(603)-93; AFOSR, DCS.

A spectroscopic study of the transient processes involving free radical molecules. These studies will require collateral investigations such as characterizing the methods by which free radicals may be formed and observing processes by which energy is redistributed after the formation of radicals. This investigation will ultimately lead to direct quantitative studies of the intermediate processes characteristic of most high temperature gas reactions.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research Fiscal Year 1960.

*Chicago University, Ill.
SPECTROSCOPIC STUDIES. R. S. Mulliken. Project 6688 (770), Contract AF 19(604)-6662; GRD, PL.

This research relates to molecular structure and high resolution spectroscopy of atmospheric gases.

*Pennsylvania State University, University Park.
SPECTROSCOPIC INSTRUMENTATION, T. K. McCubbin. Project 860?(804), Contract AF 19(604)-5458; GRD, TRL.

This research covers techniques of achieving high resolution (0.1 cm^{-1} or less) beyond 4 microns in the infrared by means of multiple-beam interferometric methods. A high resolution prism grating instrument has been assembled and will be used to investigate interferometric and multichannel methods of improving the luminosity of IR spectrometric instruments.

*Colorado University, Boulder.
SPECTROSCOPIC STUDIES, W. A. Rense. Project 6688(770), Contract AF 19(604)-7215; GRD, PL.

This research is directed toward the study of various features of the ultra-violet portion of the solar spectrum such as emission line shapes and intensities and the extent and nature of any existing continua.

*Johns Hopkins University, Baltimore, Md.
SPECTROSCOPIC STUDIES, J. D. Strong. Project 6688 (770), Contract AF 19(604)-6139; GRD, PL.

The objective is to perform research concerning developing the art of making extreme ultraviolet gratings.

*Thermal Radiation Lab., GRD, AFCRL, Bedford, Mass.
SPECTROSCOPY OF HIGH TEMPERATURE GASES, G. W. Wares. Project 8647(806), Internal.

Gases at high temperatures, simulating astrophysical sources and the luminous shock waves of hypersonic missiles and vehicles, will be studied in the laboratory to measure their fundamental optical properties, particularly their absolute f-values. The latter are essential to the interpretation of astrophysical and shock wave spectra to determine the physical conditions of these high temperature sources from optical observations at a distance. The shifts and profiles of spectral lines will also be studied for the same purpose since they are directly controlled by the temperature, pressure, density, turbulent motion, and magnetic fields prevailing in these sources. The high temperatures required will be obtained in the laboratory principally by the shock tube technique which provides known and controlled high temperatures without insulation and with a minimum of contamination, at the expense of short time duration. This last requires high time resolution spectroscopy and associated electronics and wide-aperture optics. These short time durations in the shock tube have the advantage of providing time resolution of high temperature chemical kinetics of ionization, dissociation and recombination and of the relaxation times for various types of equilibrium.

*Advanced Studies Office, RADC, Rome, N. Y.
SPIN COUPLING INVESTIGATION, C. D. Mollenhauer. Project 8503(803), Internal.

Three permanent magnets have been modified to provide homogeneous fields. The 5-inch magnet has a gap of 1-1/2 inches and a field strength of 39000 oersteds with a homogeneity of .05 oe. over a $1cm^3$ volume. The two smaller magnets have adjustable gaps and three sets of interchangeable pole caps providing a range of fields from 1400 to 3500 oe. with a maximum homogeneity of .02 oe. with a 1/2 inch gap. An improved Pound-Watkins NMR spectrometer was used to measure relaxation times of proton spins in a water solution of ferric ion. A theoretical analysis of the transient wiggles was completed and the theoretical results were related to the experimental results in order to obtain the above estimates of field homogeneity. Future efforts will involve studies of enhanced nuclear polarization in liquids obtained through the inverse Overhauser Effect.

*Stanford University, California.
STRUCTURE OF LIQUIDS. C. Pings. Project 9760(802), Contract AF 49(638)-469; AFOSR, DCS.

This is an investigation by x-ray and neutron diffraction of the structure of liquids. It is planned to determine the radial distribution function as a function of thermodynamic state, giving special emphasis to detailed studies near the regions of the critical state and phase boundaries. Combination of the x-ray and neutron diffraction data is to be used to determine the interaction between unlike molecules in condensed mixtures. This is part of a program aimed at achieving a generally applicable theory of the liquid state, such as those now in use in the solid and gaseous states.

*California Inst. of Tech., Pasadena.
STRUCTURE OF LIQUIDS, C. J. Pings. Project 9760(802), Contract AF 49(638)-800; AFOSR, DCS.

This is an investigation of the structure of liquids by means of x-ray and neutron diffraction. New apparatus advances enable these techniques to be used to determine the radial distribution function as a function of pressure and temperature, near the critical state and at phase boundaries. First liquid metals, then other substances, will be studied. Interactions between unlike molecules in liquids will be determined.

*Miami University, Oxford, Ohio.
STRUCTURE OF LIQUIDS BY X-RAY DIFFRACTION, H. Ritter. Project 9760(802), Contract AF 18(600)-485; AFOSR, DCS.

The objective of this research is to investigate structures of selected liquids by means of their x-ray diffraction patterns. The proposed liquids include: fused salts, iodine solutions in donor and non-donor solvents, mixtures of cis and trans isomers and amalgams. This research represents an area of fundamental importance in the development of the theory of fluids.

*Birmingham University (Gt. Brit).
STRUCTURE OF MOLECULES BY MICROWAVE SPECTROSCOPY, J. Sheridan. Project 9767(803) Contract AF 61 (052)-241; AFOSR, DPS.

The study of microwave spectroscopy has helped in determining the configuration of many molecules. The present work will be concerned with very short wavelengths in the millimeter range and the results used to analyse molecules as to their internuclear distance, nuclear quadrupole and molecular dipole moments, and

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research Fiscal Year 1960.

thermodynamic properties. Some of the secondary benefits from this work will be the possible establishment of frequency standards and the development of new methods of generating and detecting millimeter wavelengths which may be useful in the field of radar.

*Cornell University, Ithaca, N. Y.
STUDIES OF THE SURFACE OF SOLIDS WITH THE MASS SPECTROMETER AND FIELD EMISSION MICROSCOPE, R. C. Bradley. Project 9761(802), Contract AF 49(638)-748; AFOSR, DSS.

This research includes (1) an experimental study of the secondary ion emission from single crystal copper and molybdenum surfaces; (2) a theoretical study of the whole problem of secondary ion emission in an effort to arrive at a better understanding of what is involved in the process; (3) a study of sputtering and ion reflection; (4) A study of the kinetics of the formation and removal of the oxides and carbides; and (5) an investigation of the surfaces of some binary alloys to determine how the composition is modified by temperature, ion bombardment, and environment.

*Johns Hopkins University, Baltimore, Md.
THEORETICAL INTERPRETATION OF INFRARED SPECTRA, W. S. Benedict. Project 8603(804), Contract AF 19(604)-6130; GRD, TRL.

The goal of this research is to interpret theoretically various aspects of the infrared spectra of simple molecules, particularly those of importance in the atmospheres of the earth and planets. The studies will include analysis of high resolution spectra in terms of energy levels and of intensities of lines and bands. The analysis and interpretation of the spectra of NH_3 and its isotopic modifications has been carried out in great detail. A tabulation of all the water vapor lines in the pure rotation band has been prepared, giving the position, intensity, and width of each observable line as a function of temperature in the range of atmospheric interest 200-320°K. The effects on the intensities caused by mutual interactions of the vibrations are being investigated. Attention has also been directed to various problems involved in relating the intensities and widths of the individual lines to the low-resolution transmission of water vapor.

*Mechanics Research Branch, ARL, Dayton, Ohio.
TUNED TORSIONAL VISCOSIMETER, K. A. Erfurth. Project 7063(806), Internal.

A review of the literature on viscosimetry disclosed that there are four general types of viscosimeters, namely: (1) Capillary Tube; (2) Stokes Law Equilibrium Type; (3) Rotating Cylinder; (4) Oscillating Disc. None of these instruments is capable of measuring the viscosity of a fluid under conditions of steady state oscillation. The proposed Tuned Torsional Viscosimeter is a null-reading instrument that should be able to measure the effect of environmental disturbances, such as acoustical, thermal, and radiational inputs. It should also be capable of disclosing the presence of nonlinear viscous forces, thereby adding to the basic knowledge of behavior of damping fluids.

*Baird-Atomic, Inc., Cambridge, Mass.
TWO-BEAM IR INTERFEROMETRY, J. G. N. Braithwaite. Project 8603(804), Contract AF 19(604)-2264; GRD, TRL.

A two-beam Michelson type interferometer has been used for spectral investigations in the PbS region (1 to 2.7 microns), and the design of a new longer wavelength interferometer has been completed and its fabrication started. The data is obtained in the form of a two-beam interferogram function whose cosine Fourier transform yields the desired spectral information. The major effort has been concentrated on developing proper numerical procedures for converting interferograms to spectra.

*Photochemistry Lab., GRD, AFCRL, Bedford, Mass.
UPPER ATMOSPHERE RESEARCH, H. Pond. Project 6690(770), Internal.

The measurement of density, pressure, and temperature of the upper atmosphere involves the aerodynamics of high speed vehicles travelling through a rarefield atmosphere. A study and critical evaluation is to be made of such parameters as the drag coefficient in the transition region between hydrodynamic and free molecular flow. In addition, the relations between the ambient conditions and those measured with gauges and other instruments in rockets and satellites will be critically examined.

*Centre National de la Recherche Scientifique. (France).
VACUUM ULTRA-VIOLET SPECTROGRAPHIC INVESTIGATION OF GASEOUS IMPURITIES IN METALS, B. Vodar. Project 7364(802), Contract AF 61(052)-77; ARL, CRB.

Research is being performed on applying the new technique of vacuum ultraviolet spectrocopy to the determination of the gaseous impurities, oxygen, nitrogen, and hydrogen, which occur in metals to the detriment of their usefulness as structural material. In the spectrographic analysis of materials, the arc or spark volatilisation of the sample excites the electrons of the atoms which emit radiations of definite frequencies characteristic of the atomic number of the atom. The non-metallic elements emit radiations of higher energy which are found in the far ultraviolet. Radiation of this high energy, (less than 1800 Å wave length) is absorbed by the air in the spectrograph before it reaches the recorder. The vacuum spectrograph alleviates this absorption, permitting recording qualitative and quantitative data. This research aims to catalog the characteristic U-V frequencies of oxygen, nitrogen, and hydrogen and to establish relations between concentration of the gas and intensity of radiation. The work on oxygen has been completed, and is underway on the other two gases.

*Columbia University, New York.
VALENCE THEORY AND NUCLEAR RESONANCE, J. Verdieck, R. Bersohn. Project 9751(801), Contract AFOSR Grant 60-26; AFOSR, DPS.

An interpretation of the quadrupole resonance effects and their perturbations in single crystals of polyhalide salts such as $KICl_4^{35}$ will be studied. Specific investigations will be conducted on steric hindrance effects on quadrupole coupling constants and the orientation of the nuclei of gas

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

molecules through optical pumping techniques. A general theoretical study of the molecular orbitals of polyhalide ions will also be attempted.

*Cornell University, Ithaca, N. Y.
VAPORIZATION PROCESSES AT HIGH TEMPERATURE, R. F. Porter. Project 9763(802), Contract AF 18(603)-1; AFOSR, DCS.

A time-of-flight mass spectrometer will be used as a tool for studying high temperature vaporization processes of several oxides, nitrides, and carbides. From the data gathered, determination is to be made of the thermodynamic quantities for individual molecular species in vapors.

*Texas A and M Research Foundation, College Station.
VIBRATIONAL STRUCTURE OF THE ELECTRONIC SPECTRA OF MOLECULES, J. B. Coon. Project 9751 (801), Contract AF 49(638)-593; AFOSR, DPS.

This project deals with experimental and theoretical studies of the excited electronic states and electronic transitions of simple polyatomic molecules. The characteristics of interest are the oscillator strengths, polarizability and symmetry. The experimental work will be done by means of ultraviolet, and visible spectroscopy. The results of the analysis of these spectra will be used to verify the theoretical work. Of special interest will be the cases of double minimum potentials and the improvement of application of the Franck-Condon principle. The results of this type of work are useful in the field of photochemistry, reaction mechanisms, collision cross-section and high temperature thermodynamics.

*Washington University, St. Louis, Mo.
VIBRATIONAL AND VIBRATION-ROTATIONAL SPECTROSCOPY, D. F. Eggers. Project 9760(802), Contract AF 49(638)-797; AFOSR, DCS.

This is a study of vibrations and rotations of small molecules and calculation of atomic motions, forces and energies, by means of infrared absorption spectra, using new measurements made possible by high resolution techniques, partial isotopic substitution and very low temperatures.

*Electronic Material Sciences Lab., Bedford, Mass.
X-RAY MEASUREMENTS, E. R. Czerlinsky. Project 5621(802), Internal.

X-ray work utilizing a Weissenberg Geiger Counter Diffractometer was directed towards refinements to obtain improved accuracy of line intensities. A computer program was developed for determination of oxygen parameters in garnets from the measured intensities. The x-ray facilities were expanded to include the x-ray fluorescent analysis method. The signal to noise level was appreciably improved in the existing apparatus.

Bell & Howell Research Center. WADD TR 60-415.
ELECTROANALYTICAL METHOD FOR THE DETERMINATION OF CARBON AND HYDROGEN IN ORGANIC COMPOUNDS. Herbert S. Haber, Kenneth W. Gardiner. AF 33(616)-6676. September 1960. PB 171409. Order from OTS $0.75.

This report contains the results of research investigations completed for Contract AF 33(616)-6676 during the period from June 15, 1959 to May 14, 1960. Research efforts were directed toward the development of a rapid electroanalytical method for the determination of carbon and hydrogen in non-metallic organic compounds containing carbon, hydrogen and oxygen. The method utilizes a novel combustion technique and is applicable to the solid, liquid or gaseous state of aggregation. The method at the present time is specific for carbon and hydrogen only in the presence of oxygen, but future work is planned to extend the applicability to compounds containing halides, sulfur, nitrogen, etc. The time required for analysis of a sample is approximately 10 minutes including the weighing of the sample. A suitable-packaged prototype instrument designed to accomplish these ends will be available for delivery to WADD at the termination of this contract.

Southern Research Institute. WADD TR 60-333.
FAR INFRARED SPECTRA OF SUBSTITUTED AROMATIC HYDROCARBONS. William S. Wilcox, C. V. Stephenson, W. C. Coburn, Jr. AF 33(616)-5855. September 1960. PB 171300. Order from OTS $3.00.

The results of a fundamental study of the infrared spectra of substituted aromatic hydrocarbons from 2 to 35 microns are discussed. The purpose of this work has been to explore the possibility of correlating the absorption frequencies of substituted bensenes with physical properties of the molecules or of their substituents. Since the far infrared spectra (15 to 35 microns) exhibits a greater sensitivity to changes in the substituents bonded to the aromatic ring than the rocksalt spectra (2 to 15 microns), the correlation studies have been limited to the long wavelength region. The materials apparatus and techniques used in this study are discussed in Appendix I. The far infrared spectra of 94 substituted bensenes are given.

Battelle Memorial Institute. WADD TR 60-204.
INVESTIGATION OF THE INFRARED ABSORPTION OF SELECTED AROMATIC COMPOUNDS IN THE REGION OF 2 TO 35 MICRONS. R. J. Jakobsen. AF 33(616)-6140. September 1960. PB 171342. Order from OTS $1.25.

Complete vibrational assignments have been made for phenol, p-xylene, p-cresol, and hydroquinone. All assignments of ring vibrations have been checked by use of the Pitzer-Scott modification of the Teller-Redlich product rule. These complete vibrational assignments have been made in order to provide a foundation for the assignment of the low-frequency vibrations of a series of para-substituted phenols.

Infrared spectra of the selected para-substituted phenols have been recorded over the 2- to 35-micron range. With the aid of these infrared data and of Raman spectra, most of the low-frequency vibrations have been assigned. The reliability of the assignments and the significance of the assignments are discussed, as well as the determination of the modes of vibration.

Correlation studies of frequency with physical properties have been made for the assigned frequencies that are sensitive to the nature of the substituent. Vibration 20 still shows (although not as good as shown in Progress Report No. 1) a correlation with the substituent constants, $_p$ and $_r$, and possibly shows a correlation with Platt's spectroscopic moments.

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*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

TR 5544
REVIEW OF THE PROBLEMS OF SPECIFICATION, INSPECTION AND PROCUREMENT. Don Brouse, Lt. Colonel. January 1947.

Bjorksten Research Laboratories, Inc. TR 52-51
FOAMED METAL LOW DENSITY CORE MATERIAL FOR SANDWICH CONSTRUCTION. J. Bjorksten, J.C. Elliott, and R. J. Roth. AF 33(-38)-21838. June 1952 PB 131419 Order from OTS $0.75.

Metallic low density foams were produced from magnesium-aluminum alloys using either titanium or zirconium hydride as the foaming agent.

The most effective procedure consisted of the prior preparation of a mixture of the hydride and the magnesium-aluminum eutectic alloy as the foaming agent. Incorporation of the melted foaming agent into the molten alloy to be foamed at a temperature slightly above its melting point produces a mixture, which on solidification is a satisfactory metallic foam.

By this procedure, a foam with a density of 15 pounds per cubic foot was obtained from the 95% Al-5% Mg alloy. This foam had a nominal cell size of about 1/16 inch. The cell size was quite uniform. More than 80% of the cells were 1/16" or less, 2% zirconium hydride was used to produce this foam.

Forest Products Laboratory. TR 52-184.
SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTION FOR AIRCRAFT. Donald G. Coleman. AF 18(600)-70. December 1952.

Developments in the program of research in sandwich aircraft construction conducted by the U.S. Forest Products Laboratory are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Sixty-six technical reports issued during the several years this project has been under way are abstracted.

Goodyear Aircraft Corporation. TR 53-72.
FOAMED-IN-PLACE PLASTIC SANDWICH CONSTRUCTION, METAL FACED. E. Duplaga and B.D. Raffel. AF 33(038)-22258. March 1953. PB 111417. Order from OTS, $1.00.

Control surfaces, such as tabs and ailerons, and helicopter rotor blades and hollow propellors are most efficient as light weight structures made with relatively thin skin construction. However, this procedure results in design difficulties in order to avoid oil canning and fatigue and vibration failures, which cannot always be eliminated. These problems, it was thought, could be solved by filling the cavities of these parts and structures with a low density core material bonded to the faces or skins to produce a light weight, strong, rigid surface as in sandwich construction. An investigation of a foamed-in-place alkyd-isocyanate resin system as a possible means to accomplish this task is described. Significant features discussed are the treatment of the aluminum skins along with the application of various coatings to achieve improved metal to foam bond. It is concluded that the sulfuric acid-sodium dichromate treatment of the metal produces the most effective cleaning of aluminum for coating preparation. The application of Epon VI as a coating for the aluminum improves the physical strength of the sandwich and minimises the "cleavage plane" effect in the foam adjacent to the skin-core bond.

Bjorksten Research Laboratories, Inc. TR 52-51, Part 2.
FOAMED METAL LOW DENSITY CORE MATERIAL FOR SANDWICH CONSTRUCTION. Johan Bjorksten, John C. Elliott and Robert J. Roth. AF 33(038)-21838 S1 (52-126) September 1953. PB 131468 Order from OTS $0.75.

Progress on the development of an extrusion process for the preparation of metallic low density foams from magnesium-aluminum alloys is described. Difficulties were encountered in a number of process variables and further work is required to develop a successful process.

Inasmuch as this work is being continued, any conclusions must be regarded as tentative and subject to modification based on additional investigation.

Forest Products Laboratory. TR 52-184, Supp. 1.
SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTION FOR AIRCRAFT. Donald G. Coleman. AF 33(038)-51-4326E. October 1953.

Developments in the program of research in sandwich aircraft construction conducted by the U.S. Forest Products Laboratory during fiscal year 1953 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Five technical reports issued during the fiscal year are abstracted.

Dow Corning Corporation. TR 53-146.
DEVELOPMENT OF A HEAT-RESISTANT FOAMED-IN-PLACE LOW-DENSITY SILICONE RESIN CORE MATERIAL Kenneth R. Hoffman and Donald E. Weyer. AF 33(600)-6320. December 1953. PB 111555. Order from OTS. $3.25.

Methods for producing low density silicone resin core material have been developed. The foams are unicellular and have excellent heat stability along with low moisture absorption and good electrical properties.

The resins, at 100% solids, were expanded by the thermal dissociation of a blowing agent blended with the resin. None of the materials used in foaming was toxic.

Numerous silicone resins were evaluated and the revised specifications of the contract were met by using Resin XR-544. The expanded resin gave a large variety of physical properties. Controlled density ranged from 8 to 24 pounds per cubic foot and compressive strength varied from 90 to 850 psi at room temperature. Compressive strength at 500 F after 1/2 hour exposure, was from 20 to 240 psi. Thermal life of the foams was over 300 hours at 500 and 600 F with no appreciable weight loss or change of appearance. Specifically, a foam with a 17 lb./cu.ft. density had a room temperature compressive strength of 425 psi. After 1/2 and 200 hours at 500 F its compressive strength was 125 psi and 115 psi respectively. Several of the foams were exposed to 700 F for 10 hours without qualitative change. They were non-flammable and very resistant to a direct flame.

The resins were also foamed-in-place between glass fabric base silicone resin laminated skins. Although comparatively little work has been completed on this phase of foaming, it appeared that the core was fairly uniform and bonded to the faces.

Golladay Aeronautical Laboratory. TR 53-327.
DEVELOPMENT OF PLASTIC EXPANDED PELLET-TYPE CORE MATERIAL FOR SANDWICH CONSTRUCTION.
Arthur Derr Golladay. AF 33(600)-19090. December 1953. ASTIA Document No. AD 24746. PB 125144. Order from L.C. Mi. $3.00 Ph. $6.30.

Preliminary work has been conducted on expanded plastic pellet-type, low density core materials for fabricating sandwich construction. Expanded plastic pellets of a wide range of sizes have been developed and produced on a laboratory basis by methods closely identical with mass production techniques.

This program has been concerned with the development of core materials making use of commercially available resins, not with the development of basic materials. However, some resin investigation has been necessary, but only enough necessary to produce pellets and core compositions suitable for demonstration of the pellet type core material.

Pellet-type core material weighing 10 pounds per cubic foot has been produced which resulted in a maximum compressive strength of 166 psi. at 2% strain. Other formulations investigated varied from 18 to 96 psi. In comparison to other presently available plastic core materials the present pellet type has low compressive strength. Based on 10 pounds per cubic foot density, the pellet core has less than 46 and 10 percent of the ultimate compressive strength of commercially available foamed-in-place and honeycomb cores, respectively.

Bjorksten Research Laboratories, Inc. TR 52-51, Part 3.
FOAMED METAL LOW DENSITY CORE MATERIAL FOR SANDWICH CONSTRUCTION. Johan Bjorksten, John C. Elliott and Robert J. Roth. AF 33(038)-21838 S3 (53-591) May 1954. PB 131467. Order from OTS. $0.75.

Research and development work proving the feasibility of foaming metals by means of a miscible gas former and progress in the design and construction of prototype equipment for the continuous production of low density foams from aluminum-magnesium alloys are described. The most important innovations in the above phases of work performed subsequent to the work described in WADC Technical Report 52-51 Part 2 are improved quality of foam through the introduction of air or oxygen into the foamed mixture prior to its solidification and the introduction of aluminum oxide-coated steel, a material with high corrosion resistance towards molten aluminum, magnesium, and their alloys, as a new structural material for certain components of the foaming equipment. Further improvement in quality and uniformity of product is seen to be largely contingent upon the construction of pilot plant equipment capable of providing a more truly continuous flow of foam in larger quantities.

Forest Products Laboratory. WADC TR 54-138.
COMPARISONS OF TEST METHODS FOR EVALUATING ADHESIVES FOR BONDING METAL FACINGS TO METAL HONEYCOMB CORES. H. W. Eickner, Fred Werren. AF 33(038)51-4326E. July 1954. PB 135746. Order from LC, Mi $3.60. Ph. $9.30.

Various shear, tension, and peel tests for determining the quality of adhesive bonds in sandwich panels were compared on matched specimens for sandwich panels of aluminum facings bonded to aluminum honeycomb core with four adhesive processes.

Flatwise shear and flexural tests gave similar shear values, with failures generally occurring in the core. The tension tests gave good indication of relative strengths of the bonds when stresses were normal to the glue line.

The peel tests included direct peel and drum peel, under both static and impact loading. These tests ranked the peel resistance of bonds in approximately the same order. It appears that there can be standardization on a simple method to rank peel resistance in the same order as do the complicated and wide variety of peel-test methods in use.

Since each type of test, shear, tension, and peel, seemed to evaluate different properties, all should be used for a complete evaluation of adhesives.

Dow Corning Corporation. WADC TR 53-146 (Suppl. 1)
DEVELOPMENT OF A HEAT-RESISTANT FOAMED-IN-PLACE LOW-DENSITY SILICONE RESIN CORE MATERIAL.
Donald E. Weyer, James R. Russell, Kenneth R. Hoffman. AF 33(600)6320. January 1955. PB 111555s. Order from OTS. $3.00.

Methods for producing foamed-in-place sandwich structures from silicone resins have been developed. The most promising method consists of expanding a dry powdered resin containing blowing agent, catalyst and inert filler. The powder melts and expands readily on heating. Core density can be conveniently controlled by adjustments in the expansion temperature. Further density control is possible with small modifications in formulating the powder.

The expansible powder produces a stable, uniform multipore foam with a pore structure predominately spherical and unicellular. None of the materials or by-products are toxic. The foams have low moisture absorption along with excellent electrical properties. They are nonflammable and very resistant to an open flame.

Foamed-in-place sandwich panels have been consistently reproduced. Density variation among the panels was 1 lb./cu.ft. Sandwich panels were made with core density ranging from 11 to 24 lbs./ cu.ft. At room temperature the compressive strength of these panels varied from 80 to 400 psi. Compressive strength after 1/2 hour exposure to 500 F was from 20 to 100 psi . Specifically, a panel having a density of 18 lbs./cu.ft. had a room temperature compressive strength of 200 psi. After 1/2 hour and 200 hours at 500 F its compressive strength was 50 and 70 psi respectively. Room temperature tensile strength of this panel averaged 60 psi.

Thermal life of the core was over 1000 hours at 600° with no appreciable weight loss or dimensional change. Weight loss of the core after 72 hours at 700 F was less than 6 percent.

Forest Products Laboratory. WADC TR 52-184 (Suppl. 2).
SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTION FOR AIRCRAFT
Donald C. Coleman. D.O. 33(616) 53-20. February 1955.

Developments in the program of research in sandwich aircraft construction conducted by the U.S. Forest Products Laboratory during fiscal year 1954 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Three technical reports issued during the fiscal year are abstracted.

Forest Products Laboratory. WADC TR 52-184 Suppl. 3.
SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTION FOR AIRCRAFT
Donald G. Coleman DO 33(616)53-20. November 1955. PB 121152 Order from OTS. $0.50.

Developments in the program of research in sandwich construction for aircraft conducted by the U.S. Forest

Products Laboratory during fiscal year 1955 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Eight technical reports issued during the fiscal year are abstracted.

Glenn L. Martin Company. WADC TR 55-463. DEVELOPMENT OF BRAZED SANDWICH CONSTRUCTION MATERIAL FOR HIGH-TEMPERATURE APPLICATION. W. Maxwell, J. Mueller, J. Siltanen. AF 33(616)-2602. March 1956. ASTIA Document No. AD 94176. PB 130401. Order from L.C. Mi $4.50, Ph. $12.30.

Procedures were developed for brazing honeycomb cores to stainless steel skins to form sandwich material suitable for elevated temperature applications. After a preliminary investigation, 17-7PH stainless steel skin and core material and Coast Metals No. 53 brazing alloy were selected for the process development work. Several brazing methods were studied and muffle brazing in an inert atmosphere was selected for the production of two-by-two foot panels. The protected-core-container methods was also developed and its feasibility demonstrated on a two-by-two foot panel. The large panels produced will be supplied to the Materials Laboratory, Wright Air Development Center, for testing and evaluation. During the course of the investigation, flexure testing and metallographic examinations were used for general screening and evaluation of the methods, heat treatments, and brazing variables.

Forest Products Laboratory. WADC TR 56-86. EFFECT OF MOISTURE SORPTION ON WEIGHT AND DIMENSIONAL STABILITY OF ALKYD-ISOCYANATE FOAM CORE. V.C. Setterholm, E.W. Kuenzi. AF 33 (616)-53-20. September 1956. ASTIA Document No. AD 97289. PB 121 800.

Tests were made to determine changes in weight and dimensions of alkyd-isocyanate foam cores subjected to immersion in water and exposure to high humidity for 60 days. Cores of varying densities were investigated. The results indicated that there are differences between alkyd-isocyanate foams that have been foamed in blocks without facings and those that are foamed between two facings to make sandwich constructions. Exposure to high humidity caused increases in weight as high as 4.0 percent and dimensional increases of 0.7 percent.

Forest Products Laboratory. WADC TR 56-119. PERFORMANCE OF GLASS-FABRIC SANDWICH AND HONEYCOMB CORES AT ELEVATED TEMPERATURES. Vance C. Setterholm. Edward W. Kuenzi. DO No. 33 (616)-56-9. September 1956. ASTIA Document No. AD 97290 PB 121 697.

This report presents the results of flatwise compression and flexure tests of heat-resistant glass-fabric honeycomb cores and sandwich after short and long periods of exposure to temperatures ranging from 75 to 700 F. Increases in test temperature produced reductions in shear strength and compressive strength of cores treated with phenolic, silicone, or heat-resistant polyester resin. Cores treated with phenolic resin were strongest at all temperatures. Cores treated with silicone resin suffered the least reduction in strength due to increases in test temperature, but these cores were much weaker at all temperatures than cores treated with the other resins.

Forest Products Laboratory. WADC TR 55-417. PERFORMANCE OF STAINLESS STEEL SANDWICH CONSTRUCTION AT HIGH TEMPERATURES. V.C. Setterholm, E. W. Kuenzi. DO 33(616)-53-10. September 1956. ASTIA Document No. AD 97288 PB 121 681.

This report presents the results of edgewise-compression and flexture tests which were conducted on stainless steel sandwich constructions at temperatures ranging from 75 to 1,200 F. Although many of the panels were corroded and poorly bonded, results of edgewise-compression tests showed that specimens from panels weighing only 2.2 pounds per square foot can sustain facing stresses as high as 230,000 pounds per square inch at 75 F. and 57,000 pounds per square inch at 1,200 F.

Forest Products Laboratory. WADC TR 52-184, Suppl. 4. SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTION FOR AIRCRAFT. Donald G. Coleman AF 33(616)-56-9. September 1956. ASTIA Document No. AD 97328. PB 121152s.

Developments in the program of research in sandwich construction for aircraft conducted by the U.S. Forest Products Laboratory during fiscal year 1956 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Nine technical reports issued during the fiscal year are abstracted.

Forest Products Laboratory. WADC TR 56-386. DEVELOPMENT AND EVALUATION OF THE CLIMBING PEEL METHOD FOR TESTING ADHESIVE BONDS IN SANDWICH AND METAL-TO-METAL CONSTRUCTIONS. H.W. Eickner, Fred Werren. AF 33(616)-56-9. October 1956. ASTIA Document No. AD 110491.

A method of test was developed for determining the peel strength of adhesive bonds in sandwich and metal-to-metal constructions. This method of test, called the FPL climbing peel test, was compared with several other peel test methods. Test data were obtained on matched specimens from sandwich panels of aluminum faces bonded to aluminum honeycomb cores and from bonded aluminum-to-aluminum panels. Eleven adhesive processes were used in preparing the test panels.

All the peel tests investigated appeared to rank the peel resistance of the different adhesive bonds in approximately the same order. The climbing peel test is recommended as a standard test method, however, because of its simplicity, uniformity of peeling, and its adaptability to tests at other than room temperature and to tests of both sandwich and metal-to-metal constructions.

Forest Products Laboratory. WADC TR 56-230. PERFORMANCE OF SANDWICH WITH CORES OF FOAMED SILICONE AND MODIFIED POLYESTER RESINS AT ELEVATED TEMPERATURES AND AT HIGH HUMIDITY. V.C. Setterholm, E. W. Kuenzi. AF 33(616)-56-9. October 1956. ASTIA Document No. AD 110421. PB 121 707.

Compression, tension, and shear properties of plastic sandwich with foamed-in-place cores were determined at temperatures from 75 to 700 F. Cores were of Dow Corning silicone foam R-7002, with a density range of 12.6 to 17.5 pounds per cubic foot, and triallylcyanurate modified polyester resin (Laminac 4231) reacted with 2,4-toluene diisocyanate, with a density range of 11.9 to 12.2 pounds per cubic foot. Also presented are data on the weight and dimensional stability

of silicone and Laminac 4231 cores exposed to 100 percent relative humidity at 100 F. for 21 days. The strength tests show that Laminac 4231 cores are much stronger than silicone cores of the same density at temperatures below 300 F. Above 300 F., the strength of Laminac 4231 cores approaches the strength of silicone cores. The weight and dimensional ability of silicone cores were not affected by exposure to high humidity. Laminac 4231 cores showed increases in both weight and dimension after the high-humidity exposure.

Forest Products Laboratory. WADC TR 57-132. MECHANICAL PROPERTIES OF 422-J BACFOAM CORE FOR SANDWICH CONSTRUCTION. E.M. Jenkinson, E.W. Kuensi. DO 33(616)-56-9. April 1957. ASTIA Document No. AD 118249 . PB 131077.

This report presents the results of a few edgewise compression and flexure tests conducted at the U.S. Forest Products Laboratory on sandwich constructions of Boeing 422 J Bacfoam core with a density of 17 to 20 pounds per cubic foot and 2024-T3 clad aluminum facings 0.040 inch thick, and the results of flatwise compression tests on the core. Tests were conducted at room temperature, 200°, 300°, 400°, and 500° F. The test specimens and methods of testing are described. Test data are presented in tabular form and as curves illustrating the variation of mechanical properties with temperature. Typical stress-strain curves are included for flatwise compression of core at room temperature.

Strength properties of the core depended on the size of the core air voids. Coarse-textured core was about 60 percent as strong as fine-textured core in flatwise compression, but course-textured core was slightly stronger in shear. Flatwise compression and shear strength values of the core at 500°F. were 40 to 50 percent of the values at room temperature. The shear modulus at 500°F. was about 25 percent of that at 75° F. The edgewise compressive strength of sandwich at 500°F. was about 60 percent of the strength at room temperature.

Forest Products Laboratory. WADC TR 55-417, Pt. II
PERFORMANCE OF STAINLESS STEEL SANDWICH CONSTRUCTION AT HIGH TEMPERATURES. E.W. Kuensi, W.E. Jahnke. DO 33(616)-56-9. October 1957. ASTIA Document No. AD 142052.

This report presents the results of test to determine structural performance of sandwich comprised of facings of stainless steel or titanium bonded to stainless steel honeycomb cores with heat-resistant, epoxy-phenol adhesive. Performance was determined at 75°, 300°, and 600°F. and after exposure at 300°F. for 100 and 500 hours. Included are results of a small-scale investigation of the effect of two methods of preparing facings for bonding and the effect of bonding with two adhesive layers instead of the usual single layer.

Forest Products Laboratory. WADC TR 52-184, Suppl. 5.
SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTIONS FOR AIRCRAFT. Donald G. Coleman. AF 33(616)-56-9. October 1957. ASTIA Document No. AD 142102. PB 154 955. Order from LC Mi $2.40, Ph $3.30.

Developments in the program of research in sandwich construction for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1957 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Seven technical reports issued during the fiscal year are abstracted.

Air Force Institute of Technology. WADC TR 58-76. FLEXURE AND TORSION OF COMPOSITE CYLINDERS. W. S. Ericksen. March 1958. ASTIA Document No. AD 151085.

A solution to the problem of determining the components of stress and displacement in composite cylinders supported as cantilever beams and subjected to flexural and torsional loads is given in this report. The type of cylinder considered is that composed of three circular, coaxial, layers of different materials that are bonded at their junctures. Two main cases are considered; one in which the material in the center layer is cylindrically aeolotropic, the other in which it is isotropic. The material in the inner and outer layers is assumed to be isotropic. By taking the thickness of one of the layers equal to zero, the results are applicable to a two-layer cylinder and, by making the inner radius zero, they are applicable to a solid cylinder.

Forest Products Laboratory. WADC TR 57-609. STRENGTH AND ELECTRICAL TRANSMISSION OF THREE TYPES OF REPAIRS TO SANDWICH CONSTRUCTION FOR RADOMES. Bruce G. Heebink, V. P. Miniutti. DO 33(616)56-9. May 1958. ASTIA Document No. AD 155579. PB 151022. Order from OTS $0.75.

The purpose of this technical report was to evaluate three methods for repairing damage to a typical radome sandwich construction. Six similarly damaged panels were chosen, and WADC sent three to Tinker Air Force Base and three to the Forest Products Laboratory for repair. The repair procedures at Tinker Air Force Base were similar to those used at the Laboratory.

The repaired panels were tested for electrical transmission by WADC and for strength at the Laboratory.

Tests were made to determine edgewise tensile, edgewise compressive, and flatwise tensile strength of the repaired and undamaged sections of the panels. One panel in each set could not be tested because it blistered while the resin was being oven cured.

Results showed, except for edgewise compressive strength of scarf-jointed repairs, that repair efficiency was highest when only one facing was repaired, and that no one method was superior for both one- and two-facing repairs. However, because (a) results from the limited number of strength tests varied considerably, (b) the two repairing facilities used different repair resins, and (c) only one of the three repair methods was duplicated, valid conclusions regarding the repair efficiency of the three methods and a comparison of the repair efficiencies obtained by the two facilities cannot be made. The results of electrical transmission tests on two of the repair methods show that the methods are electrically suitable for general radome repair.

Martin Company. WADC TR 55-463 Part II
DEVELOPMENT OF BRAZED SANDWICH CONSTRUCTION MATERIALS FOR HIGH TEMPERATURE APPLICATIONS. Charles F. Burrows, F.J. Ragland, Jr. AF 33(616)-2602. July 1958. ASTIA Document No. AD 155725 PB 151 272 Order from OTS $2.50.

An investigation was undertaken to study various base metal - braze alloy combinations for honeycomb sandwich structures that would have utility up to a maximum temperature of 1200 F. The base alloys, 17-7PH, 422, A 286, 19-9 DL and 17-14 $_{Cu}$ Mo were evaluated with silver and nickel base brazing alloys to determine their compatability and strength characteristics to develop an optimum sandwich combination.

On the basis of brazing studies and structural tests, it was found that A 286 corrosion resistant steel brazed with a nickel-chromium-silicon-iron-boron alloy offered the optimum sandwich construction for operating temperatures of 1000° to 1200°F.

Static flexure and edgewise compression data are given for all optimum base metal-braze alloy combinations. S/N curves for flexural fatigue studies made at temperatures of 75°, 500°, and 1000°F are presented for the A 286 with Ni-Cr-Si-Fe-B optimum sandwich combination.

Forest Products Laboratory. WADC TR 58-172. EFFECT OF VOIDS IN GLASS-FABRIC-REINFORCED SANDWICH FACING. B.G. Heebink, K.E. Kimball, R.A. Hann. DO 33(616)-56-9. August 1958. ASTIA Document No. AD 155805.

Two sets of composite plastic sandwich panels were fabricated. One set was constructed with premolded, relatively void-free faces and the other was wet laminated, which caused numerous small voids.

Compression tests showed that the panels with void-free, premolded faces were substantially stronger than the wet-laminated panels. Electrical tests showed that although both types passed the minimum electrical transmission specification requirements, exposure to temperature and humidity cycling resulted in practically no change from 100 percent transmission for the premolded panel, whereas the wet lay-up construction was borderline in passing the requirement under the same exposure conditions.

Forest Products Laboratory. WADC TR 55-417 Part III
PERFORMANCE OF BRAZED STAINLESS STEEL SANDWICH AT HIGH TEMPERATURES. W. E. Jahnke, E.W. Kuenzi. DO 33-(616)56-9. October 1958. ASTIA Document No. AD 202492.

This report presents results of tests to investigate structural performance of stainless steel sandwich comprised of facings brazed to honeycomb cores. Performance was determined at 75°, 800°, 1000°, and 1,200°F. after short-time exposure to test temperatures. Included in this report are comparisons between facing stresses actually developed in test specimens and stresses computed theoretically for sandwich that fails either by dimpling of facings into the core cells or by buckling when subjected to edgewise compression.

Columbia University. WADD TR 60-307. FORCEDVIBRATIONS OF SANDWICH STRUCTURES. Alfred M. Freudenthal, Maciej P. Bieniek. AF 33 (616)-7042. January 1960. ASTIA Document No. AD 258536. Order from OTS $1.00.

In Part I of this report, a method is presented for the determination of the frequency response functions of the components of deformation and of stress in orthotropic sandwich plates. It applies to the case of simply supported rectangular plates loaded by dynamic pressure normal to their planes.

In Part II, a similar method is presented for orthotropic sandwich cylindrical shells. The boundaries of the shell are assumed as simply supported, and the dynamic pressure is normal to the middle surface. In both problems, the analysis takes into account the transverse shear deformation of the core and the material damping of core and facings. The results are presented in the form of expressions suitable for numerical evaluations.

WADD TR 60-133.
CYLINDRICAL SANDWICH CONSTRUCTION DESIGN. Sidney Allinkov. February 1960. ASTIA Document No. AD 238176. PB 161793. Order from OTS, $4.00.

This report is a compilation of papers which present a comprehensive treatment of the theories and parameters associated with the design of cylindrical sandwich constructions. Many of the formulas developed are applicable to a wide variety of core and facing combinations. Experimental data on flat and curved sandwich sections are furnished to support the theoretical solutions related to the design of these structures.

*Mechanics Research Branch, ARL, Dayton, Ohio. LAMINATED SHEET STRUCTURES, R.B. Baird, Project 7063(806), Internal.

Various types of laminated sheet structures are being investigated in an attempt to determine the mechanism by which stresses and strains are transmitted in multi-layered materials. Particular attention is given to the grain orientation in adjoining layers. The influence of bond thickness is being studied, since preliminary tests indicate a marked increase in strength results from decreasing thickness of adhesive. The effect of laminations on the damping properties of flat panels will also be studied

*Metallurgy and Ceramics Research Branch, ARL, Dayton, Ohio.
LAMINATE SHEET STRUCTURES, H.A. Lipsitt. Project 7024(802), Internal.

This is a joint Research effort with the Mechanics Research Branch of our Laboratory. Once a crack forms in a structure subjected to repeated loads there is very little that can be done to prevent eventual failure. It is the express purpose of this research to study the possible methods of retarding crack growth under fatigue conditions so as to increase the safe useful lifetime of a structure. This has been partially achieved by laminating foils of metal together (as in plywood) to produce a sheet. It has also been found that these structures are less dense than the parent metal and have an inherent "fail-safe" ability to completely arrest the propagation of a normal tensile failure.

*Massachusetts Inst. of Tech., Cambridge. MECHANICAL BEHAVIOR OF METAL COMPOSITES, R.L. Bisplinghoff, E. Orowan, J. Wulff. Project 9782 (806), Contract AF 49(638)-775; AFOSR, DAS.

The principal objective of this research is to theoretically investigate the mechanical behavior of metal composites. A complementary experimental phase of the research is directed toward investigating the mechanical behavior of such materials subjected to a variety of loading conditions. The loading conditions will include pure shear, tension, compression bending and transverse shear.

*Massachusetts Inst. of Tech., Cambridge. MECHANICS OF REINFORCEMENT IN POLYMER-REINFORCEMENT COMPOSITES, F.J. McGarry. Project 7021(802), Contract AF 33(616)-6280; WADD, MC.

Research to establish the general linear theory for multilayer orthotropic plates has yielded mathematical equations for calculating stress and strain components in various directions within the plate and under various modes of loading. Studies on interfacial pressure after curing, between resin and glass have revealed that

*Abstract of unclassified basic research project supported by U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

differential thermal contraction factors are especially important in promoting strong resin-to-glass bonds. Investigations of interactions between resin ductility, strength and size of material have indicated classical brittle behavior for structural resins now used. Additional research will be conducted to establish analytical solutions for specific laminate constructions and experimentally check these solutions; to study the effects of differential thermal contraction on strength as functions of cure parameters and polymer thermochemistry; and to study the effects of resin modulus and ductility on strength as a function of material size.

*Armour Research Foundation, Chicago, Ill.
MECHANISMS OF REINFORCEMENT OF FIBER REINFORCED STRUCTURAL COMPOSITE MATERIALS, H. R. Nelson. Project 7021(802), Contract AF 33(616)-5983; WADD, MC.

The contractor shall conduct chemical, optical and mechanical investigations of interfacial conditions and other reinforced fiber phenomena which may contribute to the structural capabilities of fiber reinforced materials.

*Polytechnic Inst. of Brooklyn, N. Y.
VIBRATIONS OF CYLINDRICAL SHELLS, Y. Y. Yu. Project 9782(806), Contract AF 49(638)-453; AFOSR, DAS.

The primary objective of this research is to theoretically investigate the vibrational characteristics of finite shells with various degrees of edge restraint. The theoretical developments are to account for the effects of transverse shear deformations and rotational inertia which have not been extensively studied to date. Moreover, consideration will be given to incorporating these effects into the vibrational analysis of both homogeneous and sandwich shells.

Forest Products Laboratory. WADC TR 52-184, Supp. 8.
SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON COMPOSITE CONSTRUCTION FOR FLIGHT VEHICLES. Donald G. Coleman. AF 33(616)-58-1. November 1960. ASTIA Document No. 252620.

Developments in the program of research in composite construction for flight vehicles conducted by the U. S. Forest Products Laboratory during fiscal year 1960 are summarized. In general, the approach has been to derive design criteria mathematically, and then to check by test. Three technical reports issued during the fiscal year are abstracted.

Thermo and Materials Physics

TR 6189.
THERMAL CONDUCTANCES OF JET ENGINE BLANKETS. METHOD FOR DETERMINATION ABOVE 1200°F. L. F. Salzberg. July 1950.

Douglas Aircraft Company, Inc. TR 52-168.
THE INFLUENCE OF EXTRANEOUS ELEMENTS IN D. C. ARC SPECTRA OF FERROUS ALLOYS. Darwin P. Jensen and J. F. Young. AF 33(038)-23304. July 1952. PB 139068. Order from LC, Mi $3.30, Ph $7.80.

The influence of one alloying element upon the spectral line intensity of any other alloying element was subjected to investigation in the D. C. Arc. The study was carried out by making homogeneous metallic alloys in an induction furnace. Manganese, silicon, nickel, chromium, molybdenum, vanadium, aluminum and copper were used as the alloying elements in an iron matrix, with percentage magnitudes commonly found in low alloy steels. It is concluded that:

1. The only influence which could be attributed to any of these elements was that of copper upon the line intensity of manganese.
2. Oxygen contained within the metallic sample has a marked influence on the spectral line intensity of chromium.
3. Some other element or elements not included in this investigation such as carbon, sulfur and/or phosphorus affects the line intensities of nickel, silicon and manganese.
4. Alloys can be melted in an induction furnace without change in chemistry by employing the proper techniques.

TR 52-282.
THE PURKINJE EFFECT IN LUMINANCE MEASUREMENTS OF AIR FORCE PHOSPHORUS. Daniel I. Pomerantz and John R. Cannon. October 1952. PB 111282. Order from OTS $1.00.

A formula is derived for relating effective and photopic units of luminance, making use of Weaver's interpolated data for the mesopic luminosity functions. This formula is applied to the spectra of four typical Air Force luminescent phosphors and the calculated results are compared with experimental data obtained at Wright Air Development Center. It is concluded that Weaver's data may tentatively be accepted for conversion between effective and photopic units in luminance measurements of luminescent materials.

TR 53-32.
THE CATALYTIC OXIDATION OF AMMONIA, AN EXPERIMENTAL STUDY. Hyman Marcus. February 1953. PB 118228. Order from LC Mi $2.70, Ph $4.80.

An investigation was conducted to determine the feasibility of removing ammonia gas (injected by necessity) from an air conditioning system by the catalytic action of promoted and unpromoted cupric oxide and a cobalt-bismuth oxide mixture.

A system was designed to deliver 0.378 pounds per minute of a 5% ammonia-enriched air mixture to the catalyst for conversion. This laboratory model was scaled down by a factor of one hundred so that the facilities of the laboratory could best be utilized. The materials used to promote the action of the cupric oxide included red iron oxide, green nickel oxide, and a mixture of the two. The cobalt-bismuth mixture was prepared from the respective nitrates and was analyzed to contain 3% bismuth oxide and 97% cobaltous oxide. Analyses of the inlet and exit gases were accomplished to maintain the former at not less than 5% enrichment and to determine the nature of the reaction products at the exit.

The conversion reaction proceeded when the temperature of the preheated gas mixture passing over the catalyst bed had reached approximately 800°F. The temperature of the catalyst bed was raised to about 1500°F and

*Abstract of unclassified basic research project supported by the U. S. Air Force Office of Aerospace Research, Fiscal Year 1960.

and higher due to the exothermic nature of the reaction. The energy released in the oxidation of ammonia is of the order of 215 kilocalories per mole. The reaction products consisted of a mixture of nitric oxide, nitrogen dioxide, nitrogen, water vapor, and the ammonium salts of nitrous and nitric acids.

National Bureau of Standards. TR 53-201, Part 2.
THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLTEN MATERIALS. PART 2 - THE HEAT CAPACITY OF ANHYDROUS SODIUM HYDROXIDE FROM 0° to 700°C. Thomas B. Douglas and James L. Dever. AF 33(616)-52-10. August 1953. PB 138466. Order from LC Mi $3.30, Ph $7.80.

Samples of sodium hydroxide whose purity analyses indicated to be approximately 99.3% were sealed in pure silver. Cryoscopic measurements indicated a freezing point of 317° and a transition temperature of 293°C. Using a "drop" method and a Bunsen ice calorimeter, the enthalpy change between 0° and each of eleven temperatures up to 700°C was measured. With reasonable assumptions, the thermal measurements near the freezing point were used to correct for the impurity, giving for pure sodium hydroxide a melting point of 319°C and heats of fusion and transition of 159 and 158 absolute joules per gram, respectively. The heat capacity derived for the liquid is estimated to be correct within two percent. Values of heat capacity, entropy, relative enthalpy, and relative free energy are tabulated between 298° and 1000°K.

National Bureau of Standards. TR 53-201.
THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLTEN MATERIALS. PART 1 - THE HEAT CAPACITY OF LITHIUM FLUORIDE FROM 0°C TO 900°C. Thomas B. Douglas and James L. Dever. AF (33-616) 52-10. October 1953. PB 136845. Order from LC Mi $3.00, Ph $6.30.

By use of an ice calorimeter and a furnace containing a silver core, the relative enthalpy of lithium fluoride was measured from 0° to 900°C. These measurements, at nine temperatures for the solid and three for the liquid, possessed a probably error of the mean of 0.03% on the average, and led to a heat of fusion of 1043.6(±5) absolute joules per gram. The melting point was measured and found to be 848°C(±1°). After the sample had been frozen rapidly, the mean heat capacity between 0° and 550° was found to be lower by 0.2%. The derived heat capacity values of the solid are believed to be accurate in general to within ± 0.75%, and the mean heat capacity of the liquid between 848° and 900°C, to ± 1.5%. The heat capacity - temperature curve of the solid is in good agreement with values recently reported in the literature for the range 19° to 272°K (-254° to -1°C).

National Bureau of Standards. TR 53-201, Part 3.
THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLTEN MATERIALS. PART 3 - THE HEAT CAPACITY OF MOLYBDENUM DISILICIDE FROM 0° TO 900°C. Thomas B. Douglas and William M. Logan. AF 33(616)-52-10. October 1953. PB 137099. Order from LC Mi $2.40, Ph $3.30.

The enthalpy relative to 0°C of a sample of molybdenum disilicide ($MoSi_2$) was measured at 100-degree intervals up to 900°C, using a nichrome-V container for the sample, a silver-core furnace, and a precision ice calorimeter. Corrections were applied for the impurities (approximately 2%) reported to be in the sample. The equation

$$Cp = 0.1019 + 4.32(10^{-5})t - 1.70(10^{-8})t^2,$$

giving the heat capacity in calories per gram - deg C at t°C between 0° and 900°C, represents the unsmoothed corrected values derived from the data with an average deviation of 0.8%. This equation is believed to have an absolute accuracy within ± 3% between 100° and 800°. Values of the enthalpy, heat capacity, and relative entropy derived from this equation are formulated and tabulated at round temperatures.

Balco Research Laboratories. TR 53-378.
THE DEVELOPMENT OF ELECTRICAL CONDUCTING TRANSPARENT COATINGS FOR ACRYLIC PLASTIC SHEET. George A. Dalin, Ph.D. and Robert W. Freedman, Ph.D. AF 33(616)-111. January 1954. PB 121104. Order from OTS $1.25.

Methods are discussed for the preparation of transparent, conductive films on both acrylic and glass surfaces. The theory of the structure of such films is presented as well as a description of techniques involved in the preparation. Techniques described are sputtering of metallic cadmium under conditions such as to form the oxide, and the preparation of thin continuous films of silica and titania.

Naval Research Lab. WADC TR 54-185. Part 1.
THERMAL AND RELATED PHYSICAL PROPERTIES OF MOLTEN MATERIALS. C. T. Ewing, B. E. Boker, C. S. O. & A (33-616)52-829. C. S. O. & A (33-616) 53-178. July 1954. PB 119498. Order from LC Mi $3.00, Ph $6.30.

Thermal conductivity and heat capacity figures for hot-pressed molybdenum disilicide are reported to 840°C. The property measurements were made at the Naval Research Laboratory where work was initiated in July, 1953. The apparatus and method employed for each property study are described in detail. The conductivity coefficients were measured in a longitudinal type system with guard-ring compensation; the heat capacity results were derived from enthalpy measurements made by a drop-method with a copper block calorimeter. A detailed analytical description for each molybdenum disilicide test sample is included, and the change in property value from ideal due to the impurity content or physical form of the test sample is predicted, where possible.

National Bureau of Standards. WADC TR 53-201, Part 5.
THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLTEN MATERIALS (PART 5 THE THERMAL CONDUCTIVITY OF MOLYBDENUM DISILICIDE FROM 300° to 800°C) E. D. West, D. A. Ditmars, D. C. Ginnings. AF 33(616)-52-10. August 1954. PB 138009. Order from LC Mi $2.70, Ph $4.80.

A new apparatus is described for determining at high temperatures the thermal conductivities of liquids and solids having relatively high conductivities. The apparatus employs steady state longitudinal heat flow along a rod surrounded by a matched guard tube. Results on molybdenum disilicide are described.

Balco Research Laboratories. WADC TR 53-388, Part 2.
THE DEVELOPMENT OF ELECTRICAL CONDUCTING TRANSPARENT COATINGS FOR ACRYLIC PLASTIC SHEET. George A. Dalin, Ivan Flores. AF 33(161)-111. December 1954. PB 121262. Order from OTS $1.75.

Methods are discussed for the preparation of transparent, conductive films on both acrylic and glass surfaces. A detailed description of the preparation and application of silica undercoatings is presented in addition to sputtering of metallic cadmium under conditions such as to form the oxide. Although the described sputtering techniques apply principally to flat surfaces, the extension of these methods to curved surfaces is discussed. A description of special equipment needed in the sputtering of curved surfaces is also included.

Naval Research Laboratory. WADC TR 54-185. Part 2.
THERMAL AND RELATED PHYSICAL PROPERTIES OF MOLTEN MATERIALS, PART 2, HIGH TEMPERATURE REACTIONS OF SODIUM HYDROXIDE. D. D. Williams, R. R. Miller. AF 33(616)-54-102. January 1955. PB 111883. Order from OTS $1.75.

WADC TR 54-45.
COMPOSITE SPECTROPHOTOMETRIC PROCEDURES FOR THE ANALYSIS OF LOW-ALLOY STEELS AND OF ALUMINUM ALLOYS. S. B. Summons. February 1955. PB 121216. Order from OTS $1.75.

Southern Research Institute. WADC TR 54-306. Part 1.
INVESTIGATION OF THERMAL PROPERTIES OF PLASTIC LAMINATES. F. R. O'Brien, S. Oglesby Jr. AF 33(616)-2045. February 1955.

National Bureau of Standards. WADC TR 53-201. Part 6.
THERMAL CONDUCTIVITY AND HEAT CAPACITY OF MOLTEN MATERIALS. D. A. Ditmars, D. C. Ginnings, AF 33(616) 52-10. October 1955. PB 121[illegible]4. Order from OTS $0.75.

The thermal conductivity of beryllium oxide has been measured from 40° to 750°C with an apparatus employing steady-state longitudinal heat flow along a rod of high-fired beryllium oxide surrounded by a "guard" tube with matching temperature gradient to minimize radial heat loss. The estimated accuracy of the measurements is about 2%. However, the values of thermal conductivity of the ideal BeO crystal are probably somewhat higher than the values given because of the low density (2.62 g/cm^3) of the sample used.

Southern Research Institute. WADC TR 54-306. Part II.
INVESTIGATION OF THERMAL PROPERTIES OF PLASTIC LAMINATES, CORES, AND SANDWICH PANELS. F. R. O'Brien, S. Oglesby, Jr. AF 33(616)-2045 S2 - (54-1233). November 1955. PB 121191. Order from OTS $2.00.

This report describes the measurement of the thermal properties of a selected group of plastic laminates, cores, and sandwich panels. Thermal conductivity, specific heat, and thermal expansion was determined for four glass-fabric laminates with the same reinforcing fabric but with different resins, four foam-plastic cores, one foam sandwich panel, and one honeycomb panel.

The equipment and procedures used in the thermal property measurements are described in WADC TR 54-306,Part I. The only change in procedure was the use of the method of least mean squares to define the enthalpy-temperature curves of the foam cores and the sandwich panels.

The thermal properties of the plastic laminates were in general agreement with those of the laminates tested in the first portion of this program. The foam cores, except the heat resistant core, decomposed at relatively low temperatures. Also, the low densities of these core materials gave less precision in the specific heat determinations.

Battelle Memorial Institute. WADC TR 55-496.
THERMAL CONDUCTIVITIES, HEAT CAPACITIES, AND LINEAR THERMAL EXPANSION OF FIVE MATERIALS. C. F. Lucks, H. W. Deem. AF 33(616)-2902. August 1956. ASTIA Document No. AD 97185.

Thermal-conductivity measurements on copper, chromium, molybdenum, Grade GBH graphite, and Grade 7087 graphite are reported for the temperature range of about 200 to 1700°F. Calibration of equipment to extend the measurements to 3000°F. is presented.

Heat-capacity measurements have been made on chromium, molybdenum, Grade GBH graphite, and Grade 7087 graphite from 32 to 3000°F., and on copper from 32 to 1950°F.

Measurements on the linear thermal expansions of chromium, molybdenum, Grade GBH graphite, and Grade 7087 graphite from 68 to 3000°F., and of copper from 68 to 1800°F. are reported.

Armour Research Foundation. WADC TR 55-495. Part II.
MEASUREMENTS OF THERMAL PROPERTIES. I. B. Fieldhouse, J. I. Lang, J. C. Hedge, T. E. Waterman. AF 33(616)-2903. November 1956. ASTIA Document No. AD 110510.

The objective of Part II of this program was the measurement of the thermal conductivity and specific heat of liquid electrolytic copper up to 3000°F. All measurements were made in the Heat Transfer laboratory of the Propulsion and Fluid Mechanics Research Department, Armour Research Foundation of Illinois Institute of Technology.

Armour Research Foundation. WADC TR 55-495. Part III.
MEASUREMENTS OF THERMAL PROPERTIES. I. B. Fieldhouse, J. C. Hedge, T. E. Waterman. AF 33(616)-2903. November 1956. ASTIA Document No. AD 110526.

The objective of Part III of this program was the measurement of the thermal conductivity of sintered tantalum (Fansteel Metallurgical Corporation), graphite type 7087 (Speer Carbon Company) and graphite type GBH (National Carbon Company) from 1000°F to 3000°F. All measurements were made in the Heat Transfer Laboratory of the Propulsion and Fluid Mechanics Research Department, Armour Research Foundation of Illinois Institute of Technology.

WADC TR 56-543.
ANALYSIS OF THE THERMAL PROPERTIES OF PLASTIC LAMINATES, CORES, AND SANDWICH PANELS. Robert F. Trapp, 1/Lt. January 1957. ASTIA Document No. AD 110731. PB 121882.

The thermal properties of plastic laminates, cores, and sandwich panels are presented by grouping data from WADC Technical Reports 54-306 Parts I and II into sets, arranged according to the types of resin used in their construction. Curves are presented which are representative of the resin types for each thermal property. The scatter of data about these mean curves varies from ±10% to ±40%.

Battelle Memorial Institute. WADC TR 55-496 Pt II.

THERMAL CONDUCTIVITIES, HEAT CAPACITIES, AND LINEAR THERMAL EXPANSION OF FIVE MATERIALS. Part II. Density and Thermal Conductivity of Molten Copper. C. F. Lucks, H. W. Deem. AF 33(616)-2902. March 1957. ASTIA Document No. AD 118168.

The density of molten electrolytic tough-pitch copper was determined for the temperature range 2000 to 3000°F. The thermal conductivity of molten electrolytic tough-pitch copper was measured at 2161 and 2329°F.

North American Aviation, Inc. WADC TR 56-400 Pt I.

THERMAL PROPERTIES OF MATERIALS. Part I. Properties of Graphite, Molybdenum and Tantalum to Their Destruction Temperatures. N. S Rasor, J. D. McClelland. AF 33(616)-2909. March 1957. ASTIA Document No. AD 118144.

Thermal expansion, specific heat, and thermal conductivity have been determined from 2000°F to near the destruction temperature of molybdenum (M. P. 4700°F), tantalum (M. P. 5400°F), and four types of graphite (sublimes at 6600°F). To obtain and measure the temperatures required, a carbon tube furnace, a carbon helix furnace, and a photoelectric pyrometer were developed. The details of their construction and use are described. The thermal expansion data for the materials investigated show no grossly unusual features. The specific heat of the metals, particularly molybdenum, appreciably exceeded the Dulong-Petit value. However, their thermal conductivities closely followed the Lorenz (Wiedemann-Franz) relation for free electrons. At intermediate temperatures (2000 to 5000°F) the thermal conductivity and specific heat of graphite show no unusual features. However, at higher temperatures large and abrupt increases in specific heat and thermal resistivity occur which are consistent with the occurrence of thermally produced vacant lattice sites. A vacancy concentration of about 0.4 atomic per cent at the sublimation temperature, and a formation energy in the vicinity of 7 ev are indicated.

North American Aviation, Inc. WADC TR 56-400 Pt II.

THERMAL PROPERTIES OF MATERIALS. Part II. Thermal Conductivity of Liquid Copper. J. D. McClelland, R. C. Dahleen, N. S. Rasor, E. H. Zehms. AF 33(616)-2909. April 1957. ASTIA Document No. AD 118243.

Thermal conductivity of liquid copper from approximately 2550°F to 4050°F is found to be nearly constant at about 68 Btu/ft-hr-°F in a study making use of apparatus and techniques developed for and reported in WADC TR 56-400, Part I.

WADC TR 57-92.

EXPANSION CHARACTERISTICS OF MARLEX 20 and MARLEX 50. Hyman Marcus, Frank V. Zaleski, Capt. July 1957. PB 131402. ASTIA Document No. AD 130920.

A suitable technique was developed for volume dilatometry of plastic materials and the thermal behavior of two commercial linear polyethylene plastics, Marlex 20 and Marlex 50, was observed.

The technique consisted of sealing a test sample in a glass tube with mercury as the confining fluid. The change in volume of mercury as evidenced by its rise in a capillary tube was noted at different temperatures. The volume expansion of the test sample was calculated from the known expansivities of glass and mercury.

The dilatometers used were immersed in an electrically heated oil bath equipped with a motor driven stirrer to insure a uniform temperature distribution.

Test data were obtained for a series of six samples of each material, each series containing one non-irradiated sample and five Materials Test Reactor Facility. The doses were of the values 10^6r, 10^7r, 10^8r, 5×10^8r, and 10^9r for the irradiated samples.

Southern Research Institute. WADC TR 57-10.

MEASUREMENTS OF THE THERMAL PROPERTIES OF VARIOUS AIRCRAFT STRUCTURAL MATERIALS. Perry C. Covington, Sabert Oglesby, Jr. AF 33(616)-3328. August 1957. PB 131432. ASTIA Document No. AD 131032.

This report describes the measurements of the thermal properties of a group of structural panels and cores. Thermal expansion, thermal conductivity, and specific heat were determined for three honeycomb cores, one foamed-core sandwich panel, one laminated panel, and five sandwich panels with various honeycomb cores and facing materials. In addition, thermal conductivity determinations were made for two silicone-resin laminates to establish a check for data previously reported on these panels.

The equipment and procedures used in these thermal property determinations are essentially the same as described in WADC TR 54-306 Parts I and II with the exception of additional components necessary to obtain temperatures below ambient temperature.

National Bureau of Standards. WADC TR 57-374 Pt II.

PHYSICAL PROPERTIES OF HIGH TEMPERATURE MATERIALS. Part II. The Heat Capacity of Zirconium, Several Zirconium Hydrides, and Certain Cladding Materials from 0° to 900°C; Relation to Other Thermodynamic Properties of the Zr-H System. Thomas B. Douglas, Andrew C. Victor. AF 33(616)56-21. August 1957. ASTIA Document No. AD 150128.

Using accurate apparatus and a "drop" method, the heat content (enthalpy) of zirconium, five zirconium hydrides (N_H from 1.34 to 4.14,) and Stainless Steel Type 316 were measured over the range from 0° to 900°C. Using the values for the stainless steel and those from the literature for molybdenum and niobium, the heat capacities of typical clad samples of the hydrides may be computed additionally. Thermal hysteresis of the hydrides was investigated in several cases. Corrections were applied for the impurities in the samples measured, but the two sets of hydrides gave heat values believed to be somewhat inconsistent in the range 550° to 800°C through systematic differences in phase compositions. The heat content data were extensively correlated with certain published equilibrium data for the zirconium-hydrogen system in order to extend knowledge of the heats of hydriding, equilibrium hydrogen pressures, and limits of solid solubility to wider ranges of temperature and composition than those covered by direct measurements. The results are discussed critically, and several structural implications are pointed out.

National Bureau of Standards. WADC TR 57-374 Pt IV.

PHYSICAL PROPERTIES OF HIGH TEMPERATURE MATERIALS. PART IV - Thermal Diffusivity Apparatus for 100° to 1,500°C. Howard W. Flieger, Jr., Defoe C. Ginnings. DO 33(616)56-21. October 1957. PB 140817. Order from LC Mi $2.40, Ph $3.30.

An apparatus has been designed and constructed for measuring diffusivities up to about 1500°C. Values of

thermal conductivity may be derived from thermal diffusivity values using density and heat capacity values. The apparatus was designed for measurements on materials having thermal diffusivity values less than 0.1 $cm^2/sec.$, such as most refractory oxides at moderate to high temperatures. A radial heat flow method is used, heating the outside of a cylindrical sample at a constant rate and measuring the resulting radial temperature difference. Preliminary tests at moderate temperatures have indicated satisfactory performance in both precision and in ease of operation. The apparatus will be used to measure thermal diffusivities of certain ceramic materials which seem suitable as standards.

WADC TN 57-362
RESISTANCE OF AIR CREW CLOTHING MATERIAL TO THERMAL RADIATION. Ben J. Lisle, Richard L. Peckham, 2/Lt. November 1957. ASTIA Document No. AD 142168.

Twelve assemblies of flight clothing materials presently in use were tested to determine their resistance to thermal radiation. The assemblies were irradiated at intensities of 10 cal/cm^2 sec and 30 cal/cm^2 sec. The results were evaluated with respect to the total amount of energy necessary to produce the visible effects of scorch, char, and burn. These evaluations were aimed at determining the relative damage caused by a specific quantity of thermal energy incident upon the assembly.

WADC TR 57-381.
TABLES OF INTERPLANAR SPACINGS COMPUTED FOR THE CHARACTERISTIC RADIATIONS OF COPPER, MOLYBDENUM, IRON, CHROMIUM AND COBALT. H. J. Garrett, R. E. Brocklehurst. February 1958. ASTIA Document No. AD 142344. PB 131 764. Order from OTS $8.00.

Computations of interplanar spacings, "d" values, obtained from solutions of the Bragg equation $n\lambda = 2d \sin\theta$, are listed for the radiations normally used in X-ray diffraction analysis. All angular measurements are expressed as (2θ) values. Interplanar spacings are calculated for the K_a, K_{a_1}, K_{a_2}, and K_β radiations of the elements copper, molybdenum, iron, chromium, and cobalt.

Armour Research Foundation. WADC TR 57-487. THERMAL PROPERTIES OF HIGH TEMPERATURE MATERIALS. I. B. Fieldhouse, J. C. Hedge, J. I. Lang, T. E. Waterman. AF 33(616)-3701. February 1958. ASTIA Document No. AD 150954. PB 131 718.

The objective of this program was the measurement of the high temperature thermal properties of materials. The materials investigated were Hastelloy B, Hastelloy C, Stellite 21, Stainless Steel type 17-7 P.H., Stainless Steel type 446, Silicon Carbide, 60-15 Cr (ASTM B83-46), and beryllium. The thermal conductivity, specific heat, and linear coefficient of thermal expansion were measured from 1000°F to 3000°F, or the melting point of the material, whichever was lower. All measurements were made in the Heat Transfer Laboratory of the Propulsion and Fluid Mechanics Research Department, Armour Research Foundation of Illinois Institute of Technology. Both the experimental measurements and the results of the conversion of these measurements to the desired physical properties are given.

Denver Research Institute. WADC TR 57-468. THERMAL PROPERTIES OF HIGH TEMPERATURE MATERIALS. Richard D. Seibel, George L. Mason. AF 33 (616)-3696. June 1958. ASTIA Document No. AD 155605.

Equipment has been designed and built for the measurement of the specific heat, thermal conductivity and thermal expansion of a series of ten metals and ceramics. These measurements are from about 1000°F. to the melting point or 3000°F., whichever is lower. A vacuum dilatometer using a linear variable differential transformer as the sensing element has been used. A comparative method for measuring thermal conductivity has been used. The heat capacity was determined with an ice calorimeter and its associated high temperature furnace designed to operate up to 5000°F.

Data are presented for the expansion of all of the test materials. Enthalpy, specific heat and thermal conductivity data were determined for only part of the materials. All data are presented graphically.

Southern Research Institute. WADC TR 58-129. MEASUREMENT OF SOME THERMAL PROPERTIES OF THREE GLASSES. John V. Melonas, Perry C. Covington, Coultas D. Pears. AF 33(616)-5264. August 1958. ASTIA Document No. AD 155816. PB 151419. Order from OTS $0.50.

The thermal conductivity and heat capacity were determined for the following three glass materials: (1) Soda-Lime Silica (Libbey-Owen-Ford 9330), (2)Borosilicate (Pittsburgh Plate Glass 3235), and (3) Alumina-Silicate Glass (Corning Glass Works 1723). This investigation covered the temperature range from -100°F to the strain point of the material.

Library of Congress. WADC TR 56-423 Pt II. THERMAL PROPERTIES OF CERTAIN METALS. PART II - Iron, Beryllium, Iridium, Palladium, Platinum, and Tungsten. Thomas C. Goodwin, Jr., Maurce W. Ayton. DO 33(616)55-10. September 1958. ASTIA Document No. AD 157169. PB 151679. Order from OTS $5.00.

The bibliography on the thermal properties of certain metals consists of references, with abstracts, to pertinent open literature published from 1920 to 1957, and to unclassified reports issued from about 1944 to 1957. These references were obtained by a comprehensive search of the sources listed in the Introduction. The material included in the bibliography pertains to various thermal properties, namely, heat capacity, thermal conductivity, emissivity, thermal diffusivity, and thermal expansion of iron (pure), beryllium, iridium, rhodium, palladium, platinum, and tungsten.

Southern Research Institute. WADC TR 58-179. MEASUREMENT OF THE THERMAL PROPERTIES OF VARIOUS AIRCRAFT STRUCTURAL MATERIALS. John V. Melonas, Perry C. Covington, Coultas D. Pears. AF 33(616)-3328. November 1958. ASTIA Document No. AD 204795. PB 151546.

Thermal expansion, specific heat, and thermal conductivity properties were measured for six structural panels and cores through a temperature range from -100 to 600°F. In addition, some physical tests were made on these materials. The materials covered in the investigation consisted of one asbestos-resin laminate, three fiberglass-resin laminates, and two fiberglass-resin fluted core sandwich panels.

The test procedures and equipment used in the thermal property evaluations are modifications of the procedure described in WADC TR 54-306 according to MIL and Federal specifications.

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Armour Research Foundation. WADC TR 58-274. MEASUREMENTS OF THERMAL PROPERTIES. I. B. Fieldhouse, J. C. Hedge, J. I. Lang. AF 33(616)-3701. November 1958. ASTIA Document No. AD 206892. PB 151 583. Order from OTS $2.50.

The objective of this program was the measurement of the high temperature thermal properties of materials. The materials investigated were Stainless Steel type 316, Stainless Steel type 347, Hastelloy R-235, Aluminum Oxide, Niobium, Lithium Hydride and Synthetic Sapphire. The thermal conductivity, specific heat, and linear thermal expansion were measured from 100°F to 3000°F, or to the melting point of the material, whichever was lower. All measurements were made in the Heat Transfer Laboratory of the Propulsion and Fluids Department, Armour Research Foundation of Illinois Institute of Technology. Both the experimental measurements and the results of the conversion of these measurements to the desired physical properties are given.

Armour Research Foundation. WADC TR 58-476. THERMOPHYSICAL PROPERTIES OF SOLID MATERIALS. Alexander Goldsmith, Thomas E. Waterman. AF 33(616)-5212. January 1959. ASTIA Document No. AD 207905. PB 151 715.

Thermophysical property data for a number of solid materials are presented in both graphical and tabular form, with all entries annotated. Materials included are generally those with melting points in excess of 1000°F.

This volume is the partial result of the first year's effort in a program whose objective is the compilation, evaluation and consolidation of all primary test data published since 1940. Although this report is incomplete, it is representative of the end item. The reasons for its early distribution are to disseminate usable information on many important materials and to elicit comments and suggestions for improvement of the final product.

Chicago Midway Laboratories. WADC TR 59-87, Part II. DETERMINATION OF FACTORS GOVERNING SELECTION AND APPLICATION OF MATERIALS FOR ABLATION COOLING OF HYPERVELOCITY VEHICLES. John H. Bonin, Channon F. Price, Donald E. Taylor. AF 33(616)-5436. July 1959. ASTIA Document No. AD 226368. PB 161049. Order from OTS $1.25.

The results obtained from tests of samples of twenty-two different materials are presented. The samples were exposed to the high temperature plasma discharge produced in an air-stabilized electric arc. The sample shape, test conditions and test procedure, and the material behavior are reported upon.

National Bureau of Standards. WADC TR 59-276, Part I. THERMAL DIFFUSIVITY OF IRON. PART I. A Generator for Producing Sinusoidal Temperatures. Howard W. Flieger, Jr., Defoe C. Ginnings. AF 33(616)-57-6. December 1959. ASTIA Document No. AD 234 615.

There is described a generator for producing sinusoidal temperatures in an apparatus for measuring thermal diffusivities. Using sine potentiometers, sinusoidal temperature waves have been obtained with distortions comparable with the inaccuracies of the sine potentiometers. The system using pulse time modulation has the advantages that (1) a simple relay is used to turn on and off a fixed current to give a sinusoidal power variation in an electric heater, (2) an additional fixed power can be added to the sinusoidal power without producing distortion, and (3) the generator can be used easily with servo-control either to improve wave form or to introduce known distortion.

Battelle Memorial Institute. WADC TN 59-215. THERMAL PROPERTIES OF MATERIALS AT ELEVATED TEMPERATURES. Herbert W. Deem, Webster D. Wood, Charles F. Lucks. AF 33(616)-5849. December 1959.

Apparatus was designed and assembled for making linear-thermal-expansion, specific-heat, and thermal conductivity measurements to 500°F or above on metals and ceramic-type materials.

Linear-thermal-expansion and specific-heat measurements are made in the same apparatus using the same specimen. Expansion measurements are made by directly viewing fiducial marks on a cylindrical specimen with two telemicroscopes. Specific-heat measurements are made by measuring the heat input to an inner heater which is required to heat the specimen of known mass over a measured temperature range. Thermal-conductivity measurements are made by a radial heat-flow method using a thick-walled-cylindrical specimen with an encircling graphite heater.

No apparatus are essentially completed and are ready for calibration. No measurements are reported.

Indiana Steel Products Co. WADC TR 59-438. THE REMANENCE OF ALNICO V AND VI MAGNETS BETWEEN ROOM TEMPERATURE AND 550°C. Rudolf K. Tenzer. AF 33(616)-3385. January 1960. PB 161 708. Order from OTS $0.75.

Because Alnico V and VI magnets have been found suited for use at temperatures up to 550°C, the behavior of remanence in such magnets was studied closly at elevated temperatures.

Temperature effects on the remanence of a magnet are clearly classified and defined: a) reversible, b) irreversible, and c) effects due to changes in the material. These effects were determined on Alnico V and VI magnets at temperatures up to 570°C for periods as long as 1000 hours.

Material effects of a few percent can be minimized for magnet applications between 350°C and 550°C by heat treating at the highest operating temperature for at least 24 hours. Irreversible effects can be eliminated by cycling the magnets between room temperature and the highest operating temperature. After stabilization, remanence varies less than ±1% at elevated temperatures during exposures up to 1000 hours. Reversible effects vary according to the type of material and the temperature range in which a magnet is stabilized.

Chicago Midway Laboratories. WADC TR 59-87, Part III. DETERMINATION OF FACTORS GOVERNING SELECTION AND APPLICATION OF MATERIALS FOR ABLATION COOLING OF HYPERVELOCITY VEHICLES. J. H. Bonin, C. F. Price, H. Halle. AF 33(616)-5436. February 1960. PB 161 702. Order from OTS $3.00.

The results obtained from tests of samples of fifty-two different materials are presented. The samples were exposed to the high-temperature plasma discharge produced in an air-stabilized electric arc. The sample shape, test conditions and test procedure, and the material behavior are reported upon.

Chicago Midway Laboratories. WADC TR 59-366, Part I.
THERMAL PROTECTION OF STRUCTURAL, PROPULSION AND TEMPERATURE SENSITIVE MATERIALS FOR HYPERSONIC AND SPACE FLIGHT - PART I. Relative Performance of Ablating Materials Exposed to Low and High Heat Flux Environments. John H. Bonin, Channon F. Price. AF 33(616)-6006. May 1960. PB 161 956. Order from OTS $1.75.

This report includes the results of an investigation for which the prime objective was the comparison of the relative performance of ablating materials exposed to thermal environments associated with recoverable space capsule trajectories. The experimental equipment used is briefly described, and test results for 19 different test specimens are presented. The data are used to compare the relative performance of the ablating materials with heat sink materials on the weight-per-unit-area basis.

WADD TN 60-88.
ANALYSIS OF RELATIVE PERFORMANCE OF ABLATING MATERIALS COMPARED TO A HEAT SINK MATERIAL. R. E. Otto. June 1960. PB 171 054. Order from OTS $0.50.

This report analyzes the data presented in WADC TR 59-366 generated by Chicago Midway Laboratories, University of Chicago, under contract AF 33(616)-6006. Data are presented comparing relative performance of ablating materials with heat sink materials in terms of inside surface temperature rise and on a weight per unit area basis. Structural characteristics of available materials and analysis of re-entry deceleration loads are indicated.

Armour Research Foundation. WADC TR 58-476.
THERMOPHYSICAL PROPERTIES OF SOLID MATERIALS VOLUME I. ELEMENTS (Melting Temperature Above 1000°F) REVISED EDITION. Alexander Goldsmith, Thomas E. Waterman, Harry J. Hirschhorn. AF 33(616)-5212. August 1960.

Thermophysical property data, and their variation with temperature, are presented for a great number of solid materials, based on literature published during the period 1940-1957. Each reported value is shown and annotated, and recommended "most probable value" curves are given.

Materials covered include Elements, Alloys, Ceramics, Cermets, Intermetallics, Polymerics, and Composite Materials. Except for materials in the last two categories, only those melting above 1000°F are included.

Properties covered include the following: Melting point, density, latent heats, specific heat, thermal conductivity, thermal diffusivity, emissivity, reflectivity, thermal expansion, vapor pressure, and electric resistivity.

Each of the four volumes is designed to be expansible, and it is expected that additional or revised data sheets for inclusion of these volumes will be forthcoming.

Armour Research Foundation. WADC TR 58-476, Volume II.
THERMOPHYSICAL PROPERTIES OF SOLID MATERIALS-VOLUME II. ALLOYS (Melting Temperature above 1000°F). Alexander Goldsmith, Harry J. Hirschhorn, Thomas E. Waterman. AF 33(616)-5212. November 1960.

Thermophysical property data, and their variation with temperature, are presented for a great number of solid materials, based on literature published during the period 1940-1957. Each reported value is shown and annotated, and recommended "most probable value" curves are given.

Materials covered include: Elements, Alloys, Ceramics, Cermets, Intermetallics, Polymerics, and Composite Materials. Except for materials in the last two categories, only those melting above 1000°F are included.

Properties covered include the following: Melting point, density, latent heats, specific heat, thermal conductivity, thermal diffusivity, emissivity, reflectivity, thermal expansion, vapor pressure, and electric resistivity.

Each of the four volumes is designed to be expansible, and it is expected that additional or revised data sheets for inclusion in these volumes will be forthcoming.

Atomics International. WADD TR 60-581.
THERMAL PROPERTIES OF REFRACTORY MATERIALS. Guy W. Lehman. AF 33(616)-6794. December 1960. ASTIA Document No. AD 247411. PB 160 804. Order from OTS $2.60.

A pulse heating method for measuring the specific heat of conductors from near absolute zero to their melting point is described. Results obtained on copper, iron, molybdenum, tantalum, and rhenium are reported here and indicate that the pulsed heating method gives specific heat data accurate to a few percent. Our measurements show that the heat capacities of molybdenum and tantalum gradually rise above their Dulong and Petit values of 0.06253 and 0.03316 cal/gm/°C, respectively, at high temperatures. At 2800°K, cp(Mo) = 0.1350 cal/gm/°C and at 3200°K, cp(Ta) = 0.0667 cal/gm/°C. These anomalies have been noted in other transition metals.

The percentage elongation, relative to room temperature, of zirconium carbide has been measured 1000 and 2000°C and was found to vary linearly from 0.6% at 1000°C to 1.6% at 2300°C with a permanent set occurring above 2300°C.

A transient method for measuring thermal diffusivity, specific heat, and thermal conductivity of insulators as well as conductors has been under development for the temperature range 20°C to 2500°C. A theoretical analysis of the transient thermal response of this system is presented and basic equations are derived for obtaining the above mentioned thermal properties. Theoretical and experimental results on the thermal response of a tantalum heater compare quite favorably. A cooling curve of beryllium oxide enclosed in a tantalum heater is presented and shows that heat conduction losses are completely negligible above 900°C. Experimental diffusivity results are presented on Armco iron below the Curie point.

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