

WADC TECHNICAL REPORT 53-373

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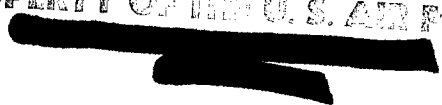
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A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM

DONNA J. TATE

DIRECTORATE OF MATERIALS & PROCESSES

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JUNE 1961

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DONNA J. TATE

JUNE 1961

DIRECTORATE OF MATERIALS & PROCESSES

**AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO**

FOREWORD

This report was prepared by Mrs. Donna J. Tate, Office of the Chief, Materials Central, Directorate of Advanced Systems Technology, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Technical reports published by the Materials Central during the period 1 July 1959 - 30 June 1960 are abstracted herein. Reports on research conducted by Materials Central personnel as well as that conducted on contract are included.

Qualified requestors may obtain copies of the reports abstracted herein direct from the Armed Services Technical Information Agency, (ASTIA), Arlington Hall Station, Arlington 12, Virginia. ASTIA's services for Department of Defense contractors are available through the Field of Interest Register on a Need To Know, certified by the cognizant military agency of their project or contract.

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ABSTRACT

These reports cover basic and applied research in the materials area being conducted by the Metals and Ceramics, Non-Metallic Materials, Physics and Applications Laboratories of the Materials Central.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



E. M. GLASS
Technical Director
Materials Central

TABLE OF CONTENTS

	Page
I TECHNICAL REPORTS AND TECHNICAL NOTES 1 JULY 1959 - 30 JUNE 1960	1
CERAMICS, CERMETS, AND GRAPHITE	1
CERAMICS AND CERMETS	1
GRAPHITE	9
HIGH STRENGTH METALS	9
HIGH TEMPERATURE METALS	20
PHYSICAL METALLURGY	25
MECHANICAL METALLURGY	30
JOINING	47
NON-DESTRUCTIVE TESTING	50
COATINGS	54
CERAMIC	54
ELECTRODEPOSITION	57
ORGANIC	60
CORROSION	61
POLYMERS AND SYNTHESIS STUDIES	68
ELASTOMERS	87
COMPOSITE MATERIALS	93
ADHESIVES	93
EROSION	100
PLASTICS	102
FIBROUS MATERIALS	113

TABLE OF CONTENTS (Continued)

	Page
FUELS, LUBRICANTS AND FLUIDS	120
FUELS	120
HYDRAULIC FLUIDS	121
LUBRICANTS	127
LUBRICANTS, SOLID FILM	140
ELECTRICAL AND ELECTRONIC MATERIALS	142
NUCLEAR MEASUREMENTS	145
THERMO PHYSICS	149
ANALYTICAL TECHNIQUES	154
DESIGN CRITERIA	159
APPLICATION STUDIES	165
ACOUSTIC MATERIALS AND STUDIES	168
II. CONTRACTOR INDEX	171
III. INVESTIGATOR INDEX	174

I. TECHNICAL REPORTS AND TECHNICAL NOTES 1 JULY 1959 - 30 JUNE 1960

CERAMICS, CERMETS AND GRAPHITE

CERAMICS & CERMETS

WADC TR 58-432, Part II, OTS Release

January 1960

SUBJECT: AN INVESTIGATION OF THE MECHANICAL PROPERTIES OF CERMETS AS RELATED TO THE MICROSTRUCTURES

INVESTIGATOR: Ira Binder

CONTRACT: AF 33(616)-5084, Firth Sterling, Inc.

ABSTRACT: 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.

WADC TR 58-452, Part II, OTS Release

January 1960

SUBJECT: METAL FIBER REINFORCED CERAMICS

INVESTIGATOR: J. J. Swica, W. R. Hoskyns, B. R. Goss, J. H. Conner, J. R. Tinklepaugh

CONTRACT: AF 33(616)-5298, College of Ceramics, Alfred Univ.

ABSTRACT: 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.

January 1960

SUBJECT: AN INVESTIGATION OF INTERMETALLIC COMPOUNDS FOR
VERY HIGH TEMPERATURE APPLICATIONS
INVESTIGATOR: R. M. Paine, A. James Stonehouse, W. W. Beaver
CONTRACT: AF 33(616)-56-12, Brush Beryllium Co.
ABSTRACT: 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 in-
cluded aluminides, beryllides, silicides, germanides, and zirconides,
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: NbAl₃, TaAl₃, CrBe₂,
MoBe₁₂, Nb₂Be₁₇, NbBe₁₂, TaBe₂, Ta₂Be₁₇, TaBe₁₂, TiBe₂, TiBe₁₂, "WBe₅",
ZrBe₁₃, Zr₂Be₁₇, Cr₃Si, Ti₅Si₃, "TiSi", TiSi₂, and TaCr₂.

A literature survey of intermetallic compounds is
included.

September 1959

SUBJECT: INFLUENCE OF SURFACE ON CERAMIC MECHANICAL
PROPERTIES
INVESTIGATOR: George R. Pulliam
CONTRACT: AF 33(616)-5875, Douglas Aircraft Co., Inc.
ABSTRACT: This study was devoted to establishing the im-
portance 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.

October 1959

SUBJECT: RESEARCH AND DEVELOPMENT SERVICES LEADING TO THE CONTROL OF ELECTRICAL PROPERTIES OF MATERIALS FOR HIGH TEMPERATURE RADOMES

INVESTIGATOR: Leon M. Atlas

CONTRACT: AF 33(616)-5929, Armour Research Foundation

ABSTRACT: 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.

October 1959

SUBJECT: MECHANISM OF WEAR OF NONMETALLIC MATERIALS

INVESTIGATOR: Edwin J. Latos

CONTRACT: AF 33(616)-5962, Armour Research Foundation

ABSTRACT: 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.

WADC TR 59-381, OTS Release

August 1959

SUBJECT: CERAMIC STUDIES SUMMARY TECHNICAL REPORT
INVESTIGATOR: P. Gibbs, G. S. Baker, E. K. Beauchamp, M. H. Miles
CONTRACT: AF 33(616)-5876, University of Utah
ABSTRACT: 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 $5 \cdot 10^{-5}$ 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.

WADC TR 59-432

February 1960

SUBJECT: REFRACTORY INORGANIC MATERIALS FOR STRUCTURAL APPLICATIONS
INVESTIGATOR: H. A. Pearl, J. M. Nowak, Joseph C. Conti, R. J. Urode
CONTRACT: AF 33(616)-5930, Bell Aircraft Corp.
ABSTRACT: 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 overall 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.

WADC TR 59-448, OTS Release

January 1960

SUBJECT: MECHANICAL PROPERTY SURVEY OF REFRACTORY NON-METALLIC CRYSTALLINE MATERIALS AND INTER-METALLIC COMPOUNDS

INVESTIGATOR: William D. Smiley, Leon E. Sobon, Frank M. Hruz, Eldon P. Farley, J. Emery Chilton, Eugene F. Poncelat, John J. Kelly

CONTRACT: AF 33(616)-5907, Stanford Research Institute

ABSTRACT: In this program, the literature on the mechanical properties of nonmetallic polycrystalline compounds and intermetallic compounds with melting points above 1500° 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.

February 1960

SUBJECT: SYNTHESIS OF NEW HIGH TEMPERATURE MATERIALS
INVESTIGATOR: J. L. Engelke, F. A. Halden, E. P. Farley
CONTRACT: AF 33(616)-5888, Stanford Research Institute
ABSTRACT: 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 $\text{HfC} \cdot 4\text{TaC}$ (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 make 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.

April 1960

SUBJECT: RESEARCH INVESTIGATION TO DETERMINE THE OPTIMUM CONDITIONS FOR GROWING SINGLE CRYSTALS OF SELECTED BORIDES, SILICIDES AND CARBIDES
INVESTIGATOR: A. D. Kiffer
CONTRACT: AF 33(616)-6326, Linde Co.
ABSTRACT: 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.

GRAPHITE

WADC TR 58-360, Part II, OTS Release

March 1960

SUBJECT: INVESTIGATION OF ELASTIC AND THERMAL PROPERTIES OF CARBON-BASE BODIES

INVESTIGATOR: S. Mrozowski, J. F. Andrew, N. Juul, J. Okada, H. E. Strauss, D. C. Wobschall

CONTRACT: AF 33(616)-5186, Carbon Research Laboratory, University of Buffalo

ABSTRACT: 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, 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 apparatus for direct determination of the heat diffusivity in the temperature range 100-700°C was set up and reasonably good data 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.

WADC TR 59-10, Part I

May 1959

SUBJECT: DEVELOPMENT AND EVALUATION OF MATERIALS FOR HIGH TEMPERATURE APPLICATIONS

INVESTIGATOR: Charles E. Schulze, Frank A. Saulino, William T. Adams, Gustave E. Mangsen, James C. Andersen, Howard W. Emmons

CONTRACT: AF 33(616)-5542, The Carborundum Company

ABSTRACT: 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.

WADC TR 59-706, OTS Release

April 1960

SUBJECT: INVESTIGATION OF GRAPHITE BODIES
INVESTIGATOR: S. W. Bradstreet, L. M. Atlas, S. A. Bortz, H.
H. Lund, H. H. Nakamura
CONTRACT: AF 33(616)-6143, Armour Research Foundation
ABSTRACT: 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.

HIGH STRENGTH METALS

WADC TR 58-457, Part I, OTS Release

February 1959

SUBJECT: RESEARCH ON TECHNIQUES FOR THE PRODUCTION OF
ULTRA-PURE BERYLLIUM
INVESTIGATOR: Joseph L. Lukesh, Laurence M. Schetky, Henry S.
Spacil, Malcolm Basche
CONTRACT: AF 33(616)-5300, Alloyd Research Corporation
ABSTRACT: The brittleness problem of beryllium is described
and the empirical and theoretical approaches to its solution discussed.
The Alloys Research Corporation program for the production of very high
purity beryllium is described in detail. Experimental results are out-
lined, conclusions are drawn, and recommendations for future work pre-
sented.

WADC TR 58-457, Part II, OTS Release

March 1960

SUBJECT: RESEARCH ON TECHNIQUES FOR THE PRODUCTION OF
ULTRA-PURE BERYLLIUM
INVESTIGATOR: Malcolm Basche, Laurence M. Schetky
CONTRACT: AF 33(616)-5300, Alloyd Research Corporation
ABSTRACT: 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:

- a. Zone purification in moderate and high vacuum
- b. Distillation under high vacuum
- c. 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.

WADC TR 58-653, OTS Release

June 1959

SUBJECT: A STUDY OF 17-7 PH STAINLESS STEEL
INVESTIGATOR: Nesbit L. Carwile, Samuel J. Rosenberg
CONTRACT: AF 33(616)-57-7, Metallurgy Division, National
Bureau of Standards

ABSTRACT: 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.

WADC TR 59-57

May 1959

SUBJECT: THE EFFECT OF SILICON ON THE TEMPERING OF HIGH STRENGTH STEELS

INVESTIGATOR: C. J. Altstetter, Morros Cohen, B. L. Averbach
CONTRACT: AF 33(616)-5161, Massachusetts Institute of Technology

ABSTRACT: 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 sub-stages 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.

WADC TR 59-63, OTS Release

June 1959

SUBJECT: DEVELOPMENT OF LOW ALLOY STEEL COMPOSITIONS
SUITABLE FOR HIGH STRENGTH STEEL CASTINGS

INVESTIGATOR: HUGO R. Larson, Ronald C. Campbell, Herbert W.
Lloyd

CONTRACT: AF 33(616)-5299, American Brake Shoe Company

ABSTRACT: 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.

WADC TR 59-86, OTS Release

June 1959

SUBJECT: DEVELOPMENT OF ULTRA-HIGH STRENGTH, TEMPER RESISTANT STEELS DESIGNED FOR IMPROVEMENT OF FATIGUE PROPERTIES THROUGH RELIEF OF RESIDUAL STRESS

INVESTIGATOR: Harvey B. Nudelman, John P. Sheehan

CONTRACT: AF 33(616)-3299, Armour Research Foundation

ABSTRACT: 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 per cent. 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.

WADC TR 59-223, OTS Release

September 1959

SUBJECT: RESEARCH ON STRAIN AGING EFFECTS IN TITANIUM

INVESTIGATOR: Harold S. Gurev, William M. Baldwin, Jr.

CONTRACT: AF 33(616)-5691, Case Institute of Technology

ABSTRACT: 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.

WADC TR 59-263, OTS Release

November 1959

SUBJECT: EFFECT OF HEAT TREATMENT ON THE METALLURGICAL AND MECHANICAL PROPERTIES OF 7AL-3MO TITANIUM ALLOY

INVESTIGATOR: Paul L. Hendricks

ABSTRACT: 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 up to 1000°F.

WADC TR 59-342, OTS Release

November 1959

SUBJECT: INVESTIGATION OF THE STRENGTH AND DUCTILITY RELATIONSHIPS IN TITANIUM-ALUMINUM ALLOYS BETWEEN 6 AND 15% ALUMINUM FOR APPLICATION AT ELEVATED TEMPERATURES

INVESTIGATOR: B. S. Lement, G. T. Hahn, K. Kreder

CONTRACT: AF 33(616)-5948, Manufacturing Laboratories, Inc.

ABSTRACT: 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 recrystallizing, 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.

WADC TR 59-353, OTS Release

October 1959

SUBJECT: DEVELOPMENT OF HIGH-TEMPERATURE IRON-BASE ALLOYS
INVESTIGATOR: A. Kasak, V. K. Chandhok, E. J. Dulis
CONTRACT: AF 33(616)-5428, Phase A, Crucible Steel Co. of America
ABSTRACT: 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, 5.71% 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 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.

October 1959

SUBJECT: DEVELOPMENT OF A CORROSION-RESISTANT BEARING STEEL FOR SERVICE IN AIRCRAFT AT TEMPERATURES UP TO 1000°F

INVESTIGATOR: G. Steven, T. V. Philip

CONTRACT: AF 33(616)-5428, Phase B, Crucible Steel Co. of America

ABSTRACT: 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 Designation	C	Mn	Si	Cr	V	W	Mo	Co
WADC 65	<u>1.10</u> 1.15	<u>0.15</u> max	<u>0.15</u> max	<u>13.5</u> 14.5	<u>2.5</u> 3.0	<u>2.0</u> 2.5	<u>3.75</u> 4.25	<u>5.0</u> 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.

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.

February 1960

SUBJECT: BERYLLIUM RESEARCH FOR DEVELOPMENT IN THE AREA OF CASTING

INVESTIGATOR: F. A. Crossley, A. G. Metcalfe, W. H. Graft

CONTRACT: AF 33(616)-5911, Armour Research Institute

ABSTRACT: 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.

March 1960

SUBJECT: METALLURGICAL AND MECHANICAL CHARACTERISTICS OF HIGH-PURITY TITANIUM-BASE ALLOYS

INVESTIGATOR: R. W. Douglass, F. C. Holden, Horace R. Ogden, R. I. Jaffee

CONTRACT: AF 33(616)-5462, Battelle Memorial Institute

ABSTRACT: 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.

March 1960

SUBJECT: FURTHER INVESTIGATIONS OF THE EFFECT OF PRIOR CREEP ON MECHANICAL PROPERTIES OF C110M TITANIUM WITH EMPHASIS ON THE BAUSCHINGER EFFECT

INVESTIGATOR: Jeremy V. Gluck, James W. Freeman

CONTRACT: AF 33(616)-3368, University of Michigan Research Institute

ABSTRACT: 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 more a minor factor.

WADC TR 59-695, Part II, OTS Release

April 1960

SUBJECT: BERYLLIUM JOINING WADC SPONSORED PROGRAM
INVESTIGATOR: E. M. Passmore
CONTRACT: AF 33(616)-5913, Avco Corporation
ABSTRACT: 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 rose rapidly with an increase in the pressure welding temperature up to about 1560°F (850°C), then decreased above 1830°C 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.

HIGH TEMPERATURE METALS

WADD TR 59-13, OTS Release

March 1960

SUBJECT: INVESTIGATION OF THE PROPERTIES OF TANTALUM AND ITS ALLOYS
INVESTIGATOR: F. F. Schmidt, W. D. Klopp, W. M. Albrecht, F. C. Holden, H. R. Ogden, R. I. Jaffee
CONTRACT: AF 33(616)-5668, Battelle Memorial Institute
ABSTRACT: 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.

WADC TR 59-280, OTS Release

October 1959

SUBJECT: DEVELOPMENT OF MOLYBDENUM-BASE ALLOYS
INVESTIGATOR: M. Semchyshen, Gordon D. McArdle, Robert Q. Barr
CONTRACT: AF 33(616)-5447, Climax Molybdenum Co. of Michigan
ABSTRACT: 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-in.-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 in. 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.

WADC TR 59-314, OTS Release

October 1959

SUBJECT: PREPARATION OF HIGH PURITY W, Mo, Ta, Cb, and Zr
INVESTIGATOR: George A. Moore, L. L. Wyman
CONTRACT: AF 33(616)-58-11, National Bureau of Standards
ABSTRACT: 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.

WADC TR 59-483, OTS Release

January 1960

SUBJECT: PHASE RELATIONSHIPS IN SELECTED BINARY AND TERNARY VANADIUM-BASE ALLOYS SYSTEMS
INVESTIGATOR: S. A. Komjathy, R. H. Read, W. Rostoker
CONTRACT: AF 33(616)-5771, Armour Research Foundation
ABSTRACT: In order to obtain information for the development of vanadium-base alloys, binary systems (V-Ja, 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°C,
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.

WADC TR 59-492, OTS Release

March 1960

SUBJECT: A STUDY OF TERNARY PHASE DIAGRAMS OF TUNGSTEN AND TANTALUM
INVESTIGATOR: W. Rostoker
CONTRACT: AF 33(616)-5678, Armour Research Foundation
ABSTRACT: 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.

February 1960

SUBJECT: OXIDATION OF TUNGSTEN AND TUNGSTEN BASED ALLOYS
INVESTIGATOR: E. A. Gulbransen, K. F. Andrew, P. E. Blackburn,
T. P. Copan, A. Merlin
CONTRACT: AF 33(616)-5770, Westinghouse Electric Corp.
ABSTRACT: 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°C 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.

February 1960

SUBJECT: STUDIES OF HEAT-RESISTANT ALLOYS
INVESTIGATOR: A. Philip Coldren, Jerry E. White, Ronald K. Bowen,
James W. Freeman
CONTRACT: AF 33(616)-5466, University of Michigan Research
Institute
ABSTRACT: 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 pre-
cipitation-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 solution 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.

PHYSICAL METALLURGY

WADC TR 55-243, Part II

September 1959

SUBJECT: DEVELOPMENT OF ALLOYS HAVING GOOD HIGH TEMPERATURE PROPERTIES THROUGH POWDER METALLURGY TECHNIQUES
INVESTIGATOR: Dr. R. Kieffer, Dr. F. Benesovsky
CONTRACT: AF 61(514)-1144, Metallwerk Plansee
ABSTRACT: Wet milled powder mixtures of Fe-Al-Mo 80-14-5-TiC/
Cr₃C₂ alloys with 0-30% of a 90-10 TiC/Cr₃C₂ 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.

WADC TN 58-369, OTS Release

May 1959

SUBJECT: OBSERVATIONS ON THE EFFECT OF SURFACE AND STRUCTURE ON THE TENSILE STRENGTH OF IRON WHISKERS
INVESTIGATOR: Helmut Weik
ABSTRACT: 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 con-

sidering the reasons for the extremely high tensile strength of very thin whiskers.

WADC TR 58-615, Part II, OTS Release

June 1959

SUBJECT: A COMPENDIUM OF CONSTITUTIONAL TERNARY DIAGRAMS OF THE METALLIC SYSTEM
CONTRACT: AF 61(052)-74, Dr. W. Guertler, Berlin, Germany
ABSTRACT: 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.

WADC TR 58-682

March 1959

SUBJECT: DEVELOPMENT OF TESTING PROCEDURES AND EVALUATION OF REFRACTORY MATERIALS
INVESTIGATOR: John M. Allen, James L. Harp
CONTRACT: AF 33(616)-5301, Battelle Memorial Institute
ABSTRACT: 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/(ft²) in a 24-second period when the maximum heat flux at the midpoint of the pulse is 822 Btu/(ft²)(sec). Similarly, nickel can absorb 7500 Btu/(ft²) with a maximum flux of 570 Btu/(ft²)(sec). Graphite protected by a coating of silicon carbide formed in place can absorb approximately 9000 Btu/(ft²) in a strongly oxidizing atmosphere

WADC TR 58-682 (Continued)

with a peak heat flux of 600 Btu/(ft²)(sec) before the protective coating is destroyed. More severe exposure caused drastic failure of the surface of each of these materials.

WADC TN 59-393, OTS Release

April 1960

SUBJECT: RESEARCHES ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS: SILVER AND LEAD
INVESTIGATOR: L. Peraldo Bicelli, B. Rivolta, Milano, Italy
CONTRACT: AF 61(052)-144
ABSTRACT: 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.

WADC TR 59-414

March 1960

SUBJECT: A SURVEY OF DISPERSION STRENGTHENING OF METALS AND ALLOYS
INVESTIGATOR: R. F. Bunshah, C. G. Goetzel
CONTRACT: AF 33(616)-5882, New York University
ABSTRACT: 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 interparticle 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

WADC TR 59-414 (Continued)

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.

WADC TR 59-441, Part I, OTS Release

January 1960

SUBJECT: THE INVESTIGATION OF THE MECHANISM OF SUBSTRUCTURAL FORMATION IN REFRACTORY METALS AND THE RELATION TO THE OBSERVED MECHANICAL PROPERTIES

INVESTIGATOR: A. Iannucci, G. Murray, J. Intrater, S. Weinig

CONTRACT: AF 33(616)-5908, Materials Research Corp.

ABSTRACT: 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.

WADC TR 59-730, OTS Release

March 1960

SUBJECT: LITERATURE SURVEY ON RESEARCH AND DEVELOPMENT ON HIGH PRESSURE TECHNOLOGY

INVESTIGATOR: Lawrence Berg, Harry Herman

CONTRACT: AF 33(616)-6729, Engineering Supervision Co.

ABSTRACT: 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 in-

WADC TR 59-730, OTS Release (Continued)

cludes, 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.

WADC TR 59-687, OTS Release

April 1960

SUBJECT: RESEARCHES ON HYDROGEN EVOLUTION
INVESTIGATOR: C. A. Knorr, Professor of Physical Chemistry
CONTRACT: AF 61(052)-142, Technische Hochschule Munich,
Germany
ABSTRACT: 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.

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.

MECHANICAL METALLURGY

WADC TR 57-755, OTS Release

June 1959

SUBJECT: CORRELATIONS BETWEEN TRANSVERSE AND TORSIONAL SHEAR CREEP DEFORMATIONS

INVESTIGATOR: Joseph Marin

CONTRACT: AF 33(616)-2729, Pennsylvania State University

ABSTRACT: 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.

WADC TR 57-756, OTS Release

June 1959

SUBJECT: STRESS DISTRIBUTION IN A PLATE WITH A HOLE SUBJECTED TO AN AXIAL LOAD AND CREEP

INVESTIGATOR: Joseph Marin

CONTRACT: AF 33(616)-2729, Pennsylvania State University

ABSTRACT: 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.

WADC TR 57-757

June 1959

SUBJECT: CREEP DEFORMATION IN A SINGLE RIVETED STRUCTURAL JOINT UNDER AXIAL TENSION

INVESTIGATOR: Joseph Marin

WADC TR 57-757 (Continued)

CONTRACT: AF 33(616)-2729, Pennsylvania State University
ABSTRACT: 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.

WADC TR 58-568, OTS Release

June 1959

SUBJECT: THE EFFECT OF CONFIGURATIONAL ADDITIONS USING VISCO-ELASTIC INTERFACES ON THE DAMPING OF A CANTILEVER BEAM

INVESTIGATOR: James S. Whittier

CONTRACT: AF 33(616)-5449, University of Minnesota

ABSTRACT: 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.

WADC TR 58-569, OTS Release

May 1959

SUBJECT: RESPONSE OF BARS(WITH INTERNAL AND BOUNDARY DAMPING) TO TRANSIENT AND RANDOM EXCITATION

INVESTIGATOR: Robert F. Lambert

CONTRACT: AF 33(616)-5426, University of Minnesota

ABSTRACT: 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 is 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 Brownian motion type excitation. Relationships showing the mode shape corrections for finite boundary impedances 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.

WADC TR 58-570, OTS Release May 1959

SUBJECT: THE RANDOM VIBRATION OF ELASTIC STRINGS-THEORETICAL
INVESTIGATOR: Richard H. Lyon
CONTRACT: AF 33(616)-5426, University of Minnesota
ABSTRACT: 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.

WADC TR 58-577, OTS Release June 1959

SUBJECT: PRELIMINARY SURVEY OF HIGH-SPEED IMPACT INFORMATION
INVESTIGATOR: Peter A. Franken
CONTRACT: AF 33(616)-5730, Bolt, Beranek and Newman, Inc.
ABSTRACT: 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

(Continued) , 20-52 10 0000
WADC TR 58-577 (Continued)

lists capabilities of existing propulsion systems. Methods of analyzing high-speed impact data are reviewed.

WADD TR 59-26, OTS Release

February 1960

SUBJECT: AN INVESTIGATION OF THE RELATIONSHIP OF HOT-HARDNESS TO THE ELEVATED TEMPERATURE EXTRUSION BEHAVIOR OF SELECTED ARC-CAST MOLYBDENUM BASE ALLOYS

INVESTIGATOR: Lt John R. Giancola

ABSTRACT: 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. Unalloyed 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.

WADC TR 59-76, OTS Release

November 1959

SUBJECT: BEAM VIBRATIONS WITH QUASI-ORTHOGONAL BOUNDARY CONDITIONS

INVESTIGATOR: Y. C. Das, L. E. Goodman, A. R. Robinson

CONTRACT: AF 33(616)-5426, University of Minnesota

ABSTRACT: 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 ross. 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.

WADC TR 59-78, Part I

July 1959

SUBJECT: MEASURING ACCURACY IN CREEP TESTS PART I. Influence of Eccentricity of Load
INVESTIGATOR: Arne Mellgren
CONTRACT: AF 61(052)-05, Royal Institute of Technology
ABSTRACT: 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.

October 1959

SUBJECT: MEASURING ACCURACY IN CREEP TESTS PART II. Influence of Thermal Stresses

INVESTIGATOR: Arne Mellgren

CONTRACT: AF 61(052)-05, Royal Institute of Technology

ABSTRACT: 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.

June 1959

SUBJECT: DAMPING ENERGY DISSIPATION AT SUPPORT INTERFACES OF SQUARE PLATES

INVESTIGATOR: T. J. Mentel, C. C. Fu

CONTRACT: AF 33(616)-5426, University of Minnesota

ABSTRACT: 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.

July 1959

SUBJECT: STEADY STATE RESPONSE OF A SIMPLE SYSTEM WITH A HYSTERETIC SPRING

INVESTIGATOR: Edward R. Rang

CONTRACT: AF 33(616)-5426, University of Minnesota

ABSTRACT: 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.

SUBJECT: THE EFFECT OF HIGH TEMPERATURE RECOVERY ON THE CREEP OF POLYCRYSTALLINE ALUMINUM IN THE DIS-
LOCATION CLIMB REGION OF TEMPERATURES

CONTRACT: AF 33(616)-3860, University of California

ABSTRACT: Recovery of the creep resistance of 99.99% pure polycrystalline 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 sub-boundaries.

SUBJECT: STEADY STATE UNDAMPED VIBRATIONS OF A CLASS OF NONLINEAR DISCRETE SYSTEMS

INVESTIGATOR: P. R. Sethna, B. E. Fristedt, V. G. Harvester

CONTRACT: AF 33(616)-5449, University of Minnesota

ABSTRACT: 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.

SUBJECT: RESEARCH ON THE MECHANISMS OF FATIGUE

INVESTIGATOR: J. C. Grosskreutz, Fred R. Rollins

CONTRACT: AF 33(616)-5065, Midwest Research Institute

ABSTRACT: 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.

WADC TR 59-227, OTS Release

September 1959

SUBJECT: INVESTIGATION OF FATIGUE PROPERTIES AT ROOM TEMPERATURE OF HIGH STRENGTH STEELS HAVING HIGH TEMPERING TEMPERATURES

INVESTIGATOR: Harold N. Cummings, Foster B. Stulen, William C. Schulte

CONTRACT: AF 33(616)-5182, Curtiss-Wright Corp.

ABSTRACT: 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)

WADC TR 59-227 (Continued)

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.

WADC TR 59-230, OTS Release

September 1959

SUBJECT: QUALITATIVE ASPECTS OF FATIGUE OF MATERIALS
INVESTIGATOR: Harold N. Cummings
CONTRACT: AF 33(616)-5182, Curtiss-Wright Corp.
ABSTRACT: 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.

WADC TR 59-339

March 1959

SUBJECT: EFFECT OF PRIOR CREEP ON SHORT-TIME MECHANICAL PROPERTIES OF 17-7 PH STAINLESS STEEL (RH 950 Condition Compared to TH 1050 Condition)
INVESTIGATOR: Jeremy V. Gluck, James W. Freeman
CONTRACT: AF 33(616)-3368, University of Michigan Research Institute
ABSTRACT: 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.

WADC TN 59-340, OTS Release

March 1960

SUBJECT: ELASTIC LIMIT BY THE RESIDUAL STRAIN METHOD USING SR4 AND TUCKERMAN GAGES ON ALUMINUM ALLOY
INVESTIGATOR: Alton W. Brisbane, R. F. Klinger
ABSTRACT: The "elastic limit" of 7075-T6 aluminum alloy was defined in terms of SR4 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.

WADC TR 59-389, OTS Release

November 1959

SUBJECT: THE USE OF ULTRASONIC METHODS TO DETERMINE FATIGUE EFFECTS IN METALS

WADC TR 59-389 (Continued)

INVESTIGATOR: John Truett, Bruce Chick, Amos Picker and George Anderson
CONTRACT: AF 33(616)-5884, Metals Research Laboratory, Brown University
ABSTRACT: 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.

WADC TR 59-399, OTS Release

March 1960

SUBJECT: CREEP AND PLASTIC DEFORMATION IN CYLINDRICAL SHELLS
INVESTIGATOR: Alfred M. Freudenthal, Maciej P. Bieniek
CONTRACT: AF 33(616)-6112, Columbia University
ABSTRACT: 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 $\dot{\epsilon} = K\sigma^n$. 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.

WADC TR 59-400, Part I, OTS Release

September 1959

SUBJECT: STATISTICAL EVALUATION OF DATA FROM FATIGUE AND CREEP-RUPTURE TESTS, Part I. Fundamental Concepts and General Methods
INVESTIGATOR: Waloddi Weibull
CONTRACT: AF 61(514)-1208, Bockamollan, Brosarps Station, Sweden
ABSTRACT: 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 para-

meters 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.

WADC TR 59-416, Part I, OTS Release

November 1959

SUBJECT: INVESTIGATION OF CREEP BUCKLING OF COLUMNS AND PLATES
INVESTIGATOR: Ralph Papirno, George Gerard
CONTRACT: AF 33(616)-4807, New York University
ABSTRACT: 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.

WADC TR 59-433, OTS Release

December 1959

SUBJECT: RAPID LOADING OF ALUMINUM ALLOY RIVETED JOINTS
INVESTIGATOR: Lt R. T. Ault
ABSTRACT: 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

WADC TR 59-433 (Continued)

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.

WADC TR 59-454, OTS Release

November 1959

SUBJECT: EFFECT OF PRIOR CREEP ON THE MECHANICAL PROPERTIES OF A HIGH-STRENGTH HEAT-TREATABLE TITANIUM ALLOY Ti-16V-2.5Al

INVESTIGATOR: Jeremy V. Gluck, James W. Freeman

CONTRACT: AF 33(616)-3368, University of Michigan Research Institute

ABSTRACT: 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 overaging 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.

WADC TR 59-470, OTS Release

March 1960

SUBJECT: NOTCH SENSITIVITY OF HIGH-TEMPERATURE ALLOYS

INVESTIGATOR: Howard R. Voorhees, James W. Freeman

WADC TR 59-470 (Continued)

CONTRACT: AF 33(616)-5775, University of Michigan Research Institute

ABSTRACT: 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 acuties, (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.

WADC TR 59-509, OTS Release

November 1959

SUBJECT: DAMPING OF FLEXURAL VIBRATIONS BY ALTERNATE VISCO-ELASTIC AND ELASTIC LAYERS

INVESTIGATOR: Eric E. Ungar, Donald Ross, Edward M. Kerwin, Jr.

CONTRACT: AF 33(616)-5426, Bolt Beranek and Newman, Inc.

ABSTRACT: 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.

March 1960

SUBJECT: STEADY STATE DAMPED VIBRATIONS AND STABILITY OF
A CLASS OF NONLINEAR DISCRETE SYSTEMS

INVESTIGATOR: S. T. Chow, P. R. Sethna

CONTRACT: AF 33(616)-5426, University of Minnesota

ABSTRACT: 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.

March 1960

SUBJECT: DAMPING OF RECTANGULAR PLATE VIBRATIONS

INVESTIGATOR: T. S. Lundgren, C. C. Chang, Y. C. Whang

CONTRACT: AF 33(616)-5426, University of Minnesota

ABSTRACT: 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 TR 59-545, OTS Release

February 1959

SUBJECT: ON THE UTILIZATION OF THE CONCEPT OF BOUNDARY IMPEDANCE IN VIBRATIONS OF BARS

INVESTIGATOR: F. J. Wilmers and R. F. Lambert

CONTRACT: AF 33(616)-5426, University of Minnesota

ABSTRACT: 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 a 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.

WADC TR 59-561, OTS Release

December 1959

SUBJECT: STUDY OF METHODS FOR NONDESTRUCTIVE MEASUREMENT OF RESIDUAL STRESS

INVESTIGATOR: Fred R. Rollins

CONTRACT: AF 33(616)-5932, Midwest Research Institute

ABSTRACT: 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.

WADC TR 59-591, OTS Release

January 1960

SUBJECT: FATIGUE BEHAVIOR OF 2014-T6, 7075-T6 and 7079-T6 ALUMINUM ALLOY REGULAR HAND FORGINGS

INVESTIGATOR: D. A. Paul, Kaiser Aluminum & Chemical Corp
D. Y. Wang, Materials Laboratory

ABSTRACT: 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.

WADC TR 59-591 (Continued)

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.

WADC TR 59-762, Part I, OTS Release

April 1960

SUBJECT: ULTRA-SHORT-TIME CREEP RUPTURE EQUIPMENT MANUAL
INVESTIGATOR: Joseph S. Ives, Jr.
CONTRACT: AF 33(616)-5557, American Machine & Foundry Co.
ABSTRACT: 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 material 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.

JOINING

WADC TR 58-674

October 1959

SUBJECT: THE CLADDING AND WELDING OF STAINLESS STEEL TO MOLYBDENUM AND NIOBIUM

INVESTIGATOR: Joseph Fugardi, John L. Zambrow

CONTRACT: AF 33(616)-3492, Sylvania-Corning Nuclear Corp.

ABSTRACT: 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 temperature 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.

WADC TR 59-404, OTS Release

December 1959

SUBJECT: DEVELOPMENT OF PARTIALLY VOLATILE BRAZING FILLER ALLOYS FOR HIGH-TEMPERATURE APPLICATION AND RESISTANCE OXIDATION

INVESTIGATOR: W. M. Lehrer, H. Schwartzbart

CONTRACT: AF 33(616)-5654, Armour Research Institute

ABSTRACT: 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 remelt temperature is increased have been studied for the range of alloys investigated which contained nickel, chromium, 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.

WADC TR 59-695, Part I, OTS Release

April 1960

SUBJECT: BERYLLIUM JOINING RAD SPONSORED PROGRAM
INVESTIGATOR: J. B. Cohen
CONTRACT: AF 33(616)-5913, Avco Corporation
ABSTRACT: 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.

NON-DESTRUCTIVE TESTING

WADC TR 54-231, Part VII

December 1959

SUBJECT: DEVELOPMENT OF NONDESTRUCTIVE TESTS FOR STRUCTURAL ADHESIVE BONDS
INVESTIGATOR: J. S. Arnold, C. T. Vincent
CONTRACT: AF 33(616)-5508, Stanford Research Institute
ABSTRACT: 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.

WADC TR 59-302, OTS Release

December 1959

SUBJECT: INVESTIGATION OF METHODS FOR DETERMINING ACTUAL FLAW SIZE IN MATERIALS BY NON-DESTRUCTIVE ULTRASONIC TECHNIQUES
INVESTIGATOR: Gerald J. Posakony, Merle R. Loetz
CONTRACT: AF 33(616)-5714, Automation Instruments, Inc.
ABSTRACT: 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.

WADC TR 59-412

June 1959

SUBJECT: NONDESTRUCTIVE TESTS FOR CERAMIC, CERMET AND GRAPHITE MATERIALS
INVESTIGATOR: Julian H. Lauchner, Dwight G. Bennett, George L. Morgan
CONTRACT: AF 33(616)-5687, University of Illinois

ABSTRACT: 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 by alumina bodies for the average particle ranges between one and ten microns.

February 1960

SUBJECT: RESEARCH AND DEVELOPMENT LEADING TO THE ESTABLISHMENT
OF ULTRASONIC TEST STANDARDS FOR AIRCRAFT MATERIALS
INVESTIGATOR: G. L. Cross, R. E. Kleint, R. D. McKown
CONTRACT: AF 33(616)-5877, Ultrasonic Testing & Research
Laboratory

ABSTRACT: 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.

COATINGS

CERAMIC

WADC TR 52-12, Part II, OTS Release

June 1959

SUBJECT: HIGH TEMPERATURE ELECTRICAL INSULATING INORGANIC COATINGS ON WIRE
INVESTIGATOR: Clifton G. Bergeron, Paul F. Schwarzlose, Arthur L. Friedberg, Robert L. Hallse, David L. Wilcox
CONTRACT: AF 33(616)-3943, University of Illinois
ABSTRACT: 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.

July 1959

SUBJECT: HIGH TEMPERATURE INSULATION FOR WIRE
INVESTIGATOR: J. N. Harris, J. D. Walton
CONTRACT: AF 33(616)-3944, Georgia Institute of Technology
ABSTRACT: 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 electrophoretically 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.

March 1960

SUBJECT: HIGH-TEMPERATURE INSULATION FOR WIRE
INVESTIGATOR: J. N. Harris, J. D. Walton, Jr.
CONTRACT: AF 33(616)-3944, Georgia Institute of Technology
ABSTRACT: 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.

WADC TN 58-13, (Continued)

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 per cent.

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.

WADC TR 59-102, OTS Release

November 1959

SUBJECT: DEVELOPMENT AND EVALUATION OF INSULATING TYPE CERAMIC COATINGS

INVESTIGATOR: E. W. Blocker, S. Sklarew, C. A. Hauck, A. V. Levy

CONTRACT: AF 33(616)-5441, Marquardt Aircraft Co.

ABSTRACT: 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.

WADC TR 59-526, OTS Release

January 1960

SUBJECT: PROTECTIVE COATINGS FOR REFRACTORY METALS

INVESTIGATOR: C. G. Bergeron, A. L. Friedberg, R. C. Anderson,
V. E. Bradford, D. M. Maroney, R. W. Boyl

WADC TR 59-526 (Continued)

CONTRACT: AF 33(616)-5734, University of Illinois
ABSTRACT: 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.

ELECTRODEPOSITION

WADC TR 58-5, Part II

May 1959

SUBJECT: COATINGS OF NICKEL-ALUMINUM ALLOYS PREPARED BY ELECTROLYSIS
INVESTIGATOR: Dwight E. Couch, Joan H. Conner
CONTRACT: AF 33(616)-57-10, National Bureau of Standards
ABSTRACT: 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.

June 1959

SUBJECT: DEVELOPMENT OF A METHOD TO ACCOMPLISH ALUMINUM DEPOSITION BY GAS PLATING
INVESTIGATOR: Malvern J. Hiler, William C. Jenkin
CONTRACT: AF 33(616)-5594, Commonwealth Engineering Co. of Ohio
ABSTRACT: 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 de-oxidation 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.

February 1960

SUBJECT: DEVELOPMENT OF PROTECTIVE COATINGS FOR REFRACTORY METALS
INVESTIGATOR: C. G. Goetzel, P. S. Venkatesan, R. F. Bunshah
CONTRACT: AF 33(616)-5735, New York University
ABSTRACT: 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).

WADC TR 59-405 (Continued)

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.

WADC TR 59-415, OTS Release

June 1959

SUBJECT: SUMMARY OF SECOND HIGH-TEMPERATURE INORGANIC REFRACTORY COATINGS WORKING GROUP MEETING

INVESTIGATOR: David Roller

ABSTRACT: 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 organizations 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.

WADC TR 59-465, OTS Release

November 1959

SUBJECT: ELECTRODEPOSITION OF ALUMINUM ON MAGNESIUM

INVESTIGATOR: E. W. Cooke, S. Kritzer

CONTRACT: AF 33(616)-5854, North American Aviation, Inc.

ABSTRACT: 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.

ORGANIC

WADC TR 59-354

January 1960

SUBJECT: STUDIES ON THE PROTECTIVE ULTRAVIOLET ABSORBERS IN
A HIGH VACUUM ENVIRONMENT

INVESTIGATOR: Richard G. Schmitt, Robert C. Hirt

CONTRACT: AF 33(616)-5945, American Cyanamid Co.

ABSTRACT: Compounds which are effective in protecting materials from terrestrial 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 2000-4000A 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 terrestrial 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.

CORROSION

WADC TR 57-542, OTS Release

September 1960

SUBJECT: A STUDY OF THE CORROSIVE EFFECTS OF THE COMBUSTION PRODUCTS OF BORON CONTAINING FUELS ON SELECTED HIGH TEMPERATURE MATERIALS

CONTRACT: AF 33(616)-3713, Reaction Motors Division

ABSTRACT: 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 symanic 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 fuel. 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 (T_c) 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 T_c has been determined. A characteristic value, A , representing this rate has been listed for many alloys and tantatively 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.

WADC TR 58-443, OTS Release

September 1959

SUBJECT: EFFECTS OF MOLTEN BORON OXIDE ON HIGH TEMPERATURE MATERIALS

INVESTIGATOR: Charles R. Andrews

WADC TR 58-443 (Continued)

CONTRACT: AF 33(616)-3898, University of Dayton Research
Institute

ABSTRACT: 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.

WADC TR 59-149, OTS Release

May 1959

SUBJECT: THIN METAL FILMS AS CORROSION INDICATORS
INVESTIGATOR: Richard B. Belser, Frank E. Hankinson
CONTRACT: AF 33(616)-3879, Georgia Institute of Technology
ABSTRACT: 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 of 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 undercoating 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.

WADC TR 59-205, OTS Release

January 1960

SUBJECT: FURTHER INVESTIGATION OF THE EFFECTS OF MOLTEN BORON OXIDE ON HIGH TEMPERATURE MATERIALS

INVESTIGATOR: Joseph W. Rosenbery

CONTRACT: AF 33(616)-3898, University of Dayton

ABSTRACT: 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 fuels (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.

WADC TR 59-205 (Continued)

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 $1750^{\circ}F$ - $2200^{\circ}F$ 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 $2100^{\circ}F$ and that siliconized SiC was the only material capable of resisting corrosion during 50 hours continuous exposure at $2200^{\circ}F$. 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.

WADC TR 59-317, OTS Release

January 1960

SUBJECT: EFFECT OF BORON CONTAINING HIGH ENERGY FUEL COMBUSTION PRODUCTS ON THE PROPERTIES OF STATICALLY STRESSED HIGH TEMPERATURE ALLOYS
INVESTIGATOR: George L. Vonnegut, William Mahler
CONTRACT: AF 33(616)-5753, Allison Division, General Motors Corp.

ABSTRACT: 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 are usually employed: Group I - J1570, Hastelloy C, Inconel, M252, Waspalloy - $1600^{\circ}F$; Group II - Hastelloy X, L 605, Inco 713, X40, GMR 235D - $1800^{\circ}F$; and Group III - Molybdenum with oxidation resistant coating - $2000^{\circ}F$.

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.

WADC TR 59-759, OTS Release

March 1960

SUBJECT: THIN METAL FILMS AS CORROSION INDICATORS
INVESTIGATOR: Richard B. Belser, Niels Engel
CONTRACT: AF 33(616)-3879, Georgia Institute of Technology
ABSTRACT: 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 NaCl 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 temperature 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.

March 1960

SUBJECT: CORROSION OF SUPERALLOYS BY SELECTED FUSED SALTS
INVESTIGATOR: A. Moskowitz, L. Redmerski
CONTRACT: AF 33(616)-6196, Crucible Steel Co. of America
ABSTRACT: 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/cm²) 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.

POLYMERS AND SYNTHESIS STUDIES

WADC TR 53-426, Part VIII

March 1960

SUBJECT: ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE LUBRICANTS AND RELATED MATERIALS
INVESTIGATOR: H. Gilman, W. Trepka, B. J. Gaj, O. L. Marrs, G. Schwebke
CONTRACT: AF 33(616)-6127, Iowa State University of Science and Technology
ABSTRACT: 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.

WADC TR 55-221, Part VI

April 1960

SUBJECT: FLUORINE-CONTAINING CONDENSATION POLYMERS AND RESINS
INVESTIGATOR: David Knutson, John S. Kolano, John E. Wier, Edward V. Gouinlock
CONTRACT: AF 33(616)-5548, Hooker Chemical Corp.
ABSTRACT: 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 perfluoroglutari-
midine and perfluoroadipamidine has been investigated.

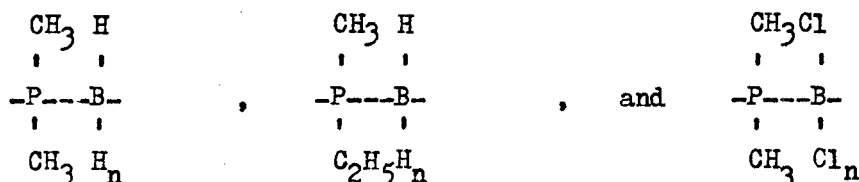
Research on the synthesis of certain novel or un-

usual 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.

WADC TR 57-126, Part III

September 1959

SUBJECT: PHOSPHINOBORINE POLYMERS
 INVESTIGATOR: Ross I. Wagner, Frederick F. Caserio, Jr.,
 Levern D. Freeman
 CONTRACT: AF 33(616)-5435, American Potash & Chemical Corp.
 ABSTRACT: 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 organophosphine 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.

February 1960

SUBJECT: DEVELOPMENT OF THERMALLY STABLE SILICON CONTAINING RESINS

INVESTIGATOR: L. W. Breed, William J. Haggerty, Jr.

CONTRACT: AF 33(616)-3675, Midwest Research Institute

ABSTRACT: 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.

June 1959

SUBJECT: ORGANOMETALLIC POLYMERS

INVESTIGATOR: Paul E. Koenig, Ronald D. Crain

CONTRACT: AF 33(616)-3848, Ethyl Corporation

ABSTRACT: 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.

WADC TR 58-51, Part II

May 1959

SUBJECT: HIGH POLYMERIC MATERIALS
INVESTIGATOR: Carl S. Marvel, Ludwig F. Audrieth, John C. Bailar, Jr.
CONTRACT: AF 33(616)-5486, University of Illinois
ABSTRACT: 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- α , 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 phosphonitrimides 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.

WADC TR 58-84, Part II

June 1959

SUBJECT: DEVELOPMENT OF HIGH-TEMPERATURE STABLE LIQUID UREAS AND AMIDES
INVESTIGATOR: Cecil C. Chappelow, Jr., Robert N. Clark
CONTRACT: AF 33(616)-5129, Midwest Research Institute
ABSTRACT: 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, sunymmetrical 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.

June 1959

SUBJECT: SYNTHESIS AND EVALUATION OF HIGH TEMPERATURE STABLE AND NUCLEAR RADIATION STABLE METAL-CYCLOPENTADIENYL FLUIDS

INVESTIGATOR: Robert L. Schaaf, Peter T. Kan

CONTRACT: AF 33(616)-5053, Wyandotte Chemicals Corporation

ABSTRACT: 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 siloxanylferrocenes 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'-(α -keto-trimethylene)-ferrocene and related compounds were investigated.

December 1959

SUBJECT: RELATIONS BETWEEN STRUCTURE AND RADIATION STABILITY OF VARIOUS ALKYL AROMATIC FLUIDS

INVESTIGATOR: Josef J. E. Schmidt, George E. Bohner

CONTRACT: AF 33(616)-5317, Denver Research Institute

ABSTRACT: 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 alkyl-polyphenyl 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.

WADC TR 58-377, Part II(Continued)

Additional data must be obtained in order to define completely the mode of radiolytic decomposition in these hydrocarbons.

WADC TR 58-589

May 1959

SUBJECT: THE SYNTHESIS OF SOME FLUORINE CONTAINING MONOMERS
INVESTIGATOR: William Durrell
ABSTRACT: 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 addition reactions were carried out and a novel mode dimerization of α, β -unsaturated fluoro-ketones discovered.

WADC TR 59-45, Part III

September 1959

SUBJECT: HIGH-TEMPERATURE STABLE SEMIORGANIC FLUIDS. PART III. PHOSPHINE OXIDES
INVESTIGATOR: Daniel Grafstein, Rita Dudak, Murray S. Cohen
CONTRACT: AF 33(616)-5653, Reaction Motors Division, Thiokol Chemical Corp.
ABSTRACT: 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-stop general synthesis of the phosphine oxides.

Benzenephosphosphenyl 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-*p*-tolylphosphine oxide and phenyl-dimethylphosphine 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.

WADC TR 59-61

June 1959

SUBJECT: RESEARCH ON POLYMERIC BONDING SYSTEMS AND THEIR DIELECTRIC BEHAVIOR
INVESTIGATOR: R. P. Anderson, M. M. Spring
CONTRACT: AF 33(616)-5535, General Electric Research Laboratory
ABSTRACT: 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 dimethyl-dichlorosilane. 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-butyl lithium 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. Coidrolysis of alkoxyboranes and alkoxyboranes does not produce reasonable yields of cocondensates.

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

WADC TR 59-61 (Continued)

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.

WADC TR 59-64

June 1959

SUBJECT: THERMAL DEGRADATION OF POLYMERS AT TEMPERATURES UP TO 850°C
INVESTIGATOR: Samuel L. Madorsky, Sidney Straus
CONTRACT: AF 33(616)-58-8, National Bureau of Standards
ABSTRACT: 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(tri vinylbenzene)) and 4 thermoset plastics (Vibron, 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(tri vinylbenzene).

WADC TR 59-95

June 1959

SUBJECT: SYNTHESIS OF 1000° STABLE BASE FLUIDS
INVESTIGATOR: James W. Dale, Elizabeth A. McElhill, Iral B. Johns, John Smith
CONTRACT: AF 33(616)-5553, Monsanto Chemical Co.
ABSTRACT: This report presents the results of a fairly basic multipronged investigation of the preparation of thermally stable fluids liquid at room temperature and useable 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

WADC TR 59-95 (Continued)

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, pyrimidine, 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.

WADC TR 59-129

July 1959

SUBJECT: MECHANISM OF CROSS-LINKING IN THE VULCANIZATION OF NEW ELASTOMERIC POLYMERS
INVESTIGATOR: Kay L. Paciorek, Raymond G. Spain, Eva P. Deck, Lawrence C. Mitchell
CONTRACT: AF 33(616)-5642, Wyandotte Chemicals Corp.
ABSTRACT: 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 monoamines 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.

WADC TR 59-129 (Continued)

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.

WADC TR 59-136

June 1959

SUBJECT: EVALUATION OF EXPERIMENTAL POLYMERS
INVESTIGATOR: Charles D. Doyle
CONTRACT: 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.

WADC TR 59-151, Part I

September 1959

SUBJECT: RESEARCH ON SYNTHESIS OF CHEMICAL INTERMEDIATES FOR HIGH TEMPERATURE FLUIDS AND POLYMERS
INVESTIGATOR: Robert E. Jones, John D. Garber, Samuel A. Robinson, Gustav A. Stein, George Gal
CONTRACT: AF 33(616)-5329, Merck Sharp & Dohme Research Laboratories
ABSTRACT: 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.

July 1959

SUBJECT: SYNTHESIS OF THERMALLY STABLE EPOXY RESINS FOR DIELECTRIC APPLICATIONS
INVESTIGATOR: Lee M. Kindley, Louis P. Glekas, Richard F. Marshall, Paul E. Ritt
CONTRACT: AF 33(616)-5518, Melpar, Incorporated
ABSTRACT: 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 tetrakis (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 quaternary 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.

September 1959

SUBJECT: SYNTHESIS OF THERMALLY STABLE INORGANIC AND SEMI-INORGANIC BASE FLUIDS

INVESTIGATOR: John A. Parkins

CONTRACT: AF 33(616)-5843, E. I. du Pont de Nemours & Co., Inc.

ABSTRACT: 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

September 1959

SUBJECT: VACUUM VOLATILITY OF ORGANIC RESINS

INVESTIGATOR: George F. Matacek, 1/Lt, USAF

ABSTRACT: 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 chlorotrifluoro-

WADC TR 59-268 (Continued)

ethylene 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, volatilized almost completely or were drastically charred during the course of the exposures.

WADC TR 59-272, Part I

September 1959

SUBJECT: SYNTHESIS OF SEMI-INORGANIC FLUORINE POLYMERS
INVESTIGATOR: Henry C. Brown
CONTRACT: AF 33(616)-5616, University of Florida
ABSTRACT: 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.

WADC TR 59-369

October 1959

SUBJECT: THE PREPARATION OF ORGANOMETALLIC DERIVATIVES OF INORGANIC "BENZENOID" COMPLEXES
INVESTIGATOR: Dietmar Seyferth, Hubert P. Kogler
CONTRACT: AF 33(616)-5582, Massachusetts Institute of Technology
ABSTRACT: 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-(ethyltrimethylsilylmethyl)-N-trimethyl-
borazene
B-tris-(n-butyldimethylsilylmethyl)-N-trimethyl-
borazene
B-tris-(pentamethyldisiloxanylmethyl)-N-trimethyl-
borazene
B-tris-(trimethylsiloxy)-N-trimethylborazene
N,N,N,N',N',N'-hexamethyl-B,B,B',B'-tetrakis-
(trimethylsilylmethyl)-B,B'-bis-borazene oxide)
B-tris-(trimethylsilylmethyl)-N-trimethylborazene

seems to have good thermal stability.

WADC TR 59-427

January 1960

SUBJECT: CONFERENCE ON HIGH TEMPERATURE POLYMER AND FLUID
RESEARCH

ABSTRACT: 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.

WADC TR 59-453

October 1959

SUBJECT: LINEAR POLYMERS CONTAINING THE TRIAZINE NUCLEUS

INVESTIGATOR: Herbert K. Reimschuessel, Edward Hagerman, Alan
M. Lovelace

ABSTRACT: 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 mono-
meric triazine derivatives are discussed, and the synthetic procedures
and properties of a number of these new triazine derivatives are de-
scribed. Furthermore, the syntheses of some prototype polymers are
given. Considerable interest is being afforded to polymers of these
type where the main polymer chain consists of alternating carbon and
nitrogen atoms. These polymers may be generally referred to as "semi
inorganic polymers".

October 1959

SUBJECT: THE PREPARATION OF CERTAIN HETEROCYCLIC POLYMERS
BY AN ALTERNATING INTRAMOLECULAR-INTERMOLECULAR
CHAIN PROPAGATION

INVESTIGATOR: George B. Butler, David L. Skinner, K. Darrell
Berlin, Robert W. Stackman

CONTRACT: AF 33(616)-5808, University of Florida

ABSTRACT: 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 intra-
molecular-intermolecular mechanism is implied for the polymerization
process.

The new organophosphorus monomers which have been prepared are: diallylphenylphosphine oxide, dimethallylphenylphosphine oxide and diallylmethylphenylphosphonium bromide. Diallylhydrogen-phenylphosphonium 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, diallylcyclohexamethylene-silane, diallylcyclopentamethylenesilane

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.

WADC TR 59-761, OTS Release

March 1960

SUBJECT: RESEARCH ON INORGANIC POLYMER SYSTEMS
INVESTIGATOR: 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
CONTRACT: AF 33(616)-5931, U. S. Borax Research Corp.
ABSTRACT: 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 proto-
type 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 tetraamino-
diborons 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 de-
composition 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.

ELASTOMERS

WADC TR 55-58, Part V

September 1959

SUBJECT: A STUDY OF THE EFFECTS OF NUCLEAR RADIATIONS ON ELASTOMERIC COMPOUNDS AND COMPOUNDING MATERIALS

INVESTIGATOR: Dale J. Harmon

CONTRACT: AF 33(616)-5646, B. F. Goodrich Co. Research Center

ABSTRACT: 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 (C₁₄-C₁₆) 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.

WADC TR 56-272, Part IV

October 1959

SUBJECT: DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS

INVESTIGATOR: George E. Trepus, Robert S. Roper, William R. Hickman

CONTRACT: AF 33(616)-5722, Boeing Airplane Co.

ABSTRACT: 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.

WADC TR 56-272, Part IV (Continued)

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.

WADC TR 56-272, Part V

March 1960

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ABSTRACT: 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.

WADC TR 57-651, Part II

June 1959

SUBJECT: DEVELOPMENT OF RUBBERLIKE MATERIALS FOR APPLICATIONS

INVOLVING CONTACT WITH LIQUID ROCKET PROPELLANTS

INVESTIGATOR: John H. Baldrige

WADC TR 57-651, Part II (Continued)

CONTRACT: AF 33(616)-5572, Connecticut Hard Rubber Company
ABSTRACT: 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.

WADC TR 58-89, Part II

September 1959

SUBJECT: DEVELOPMENT OF HIGH TEMPERATURE SEALANTS
INVESTIGATOR: Irvin P. Seegman, Paul A. Mallard, William Cheorvas, Ron E. Thompson, Francis H. Ingham, R. Loren Varner, Sarkis H. Kalfayan
CONTRACT: AF 33(616)-3976, Products Research Company
ABSTRACT: 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.

WADC TR 59-301

December 1959

SUBJECT: DEVELOPMENT OF MATERIALS FOR IMPROVED EAR PLUGS
AND FOR LIQUID-FILLED EAR PROTECTOR CUSHIONS
INVESTIGATOR: Edgar A. Verchot, Arthur E. Raeuber
CONTRACT: AF 33(616)-5673, Southern Research Institute
ABSTRACT: 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-protector 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.

October 1959

SUBJECT: COMPOSITE INORGANIC RESILIENT SEAL MATERIALS
INVESTIGATOR: M. Sabanas
CONTRACT: AF 33(616)-5793, Armour Research Foundation
ABSTRACT: 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.

April 1960

SUBJECT: COMPOSITE INORGANIC RESILIENT SEAL MATERIALS
INVESTIGATOR: M. Sabanas
CONTRACT: AF 33(616)-5793, Armour Research Foundation
ABSTRACT: 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

WADC TR 59-338, Part II (Continued)

seal air heated to 1000°F and retained pulsating pressures from zero to 5000 psi.

The relationship between the fiber structure, impregnation, and final composite were studied.

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

WADC TR 59-428

October 1959

SUBJECT: DESIGN HANDBOOK FOR O-RINGS AND SIMILAR ELASTIC SEALS

INVESTIGATOR: Frank W. Tipton

CONTRACT: AF 33(616)-5722, Boeing Airplane Company

ABSTRACT: 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.

COMPOSITE MATERIALS

ADHESIVES

WADC TR 54-447, Part III

June 1959

SUBJECT: WEATHERING OF ADHESIVE-BONDED LAP JOINTS OF CLAD ALUMINUM ALLOY
INVESTIGATOR: H. W. Eickner
CONTRACT: DO 33(616)-58-1, Forest Products Laboratory
ABSTRACT: 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,

WADC TR 54-447, Part III (Continued)

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.

WADC TR 55-491, Part IV

June 1959

SUBJECT: RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES

INVESTIGATOR: Gene H. Haertling, Kanaiyalal N. Parikh, Henry G. Lefort, Dwight G. Bennett

CONTRACT: AF 33(616)-5468, University of Illinois

ABSTRACT: 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. Infurthering 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 1067-1, 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 alkaline 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.

July 1959

SUBJECT: SURVEY OF ADHESION AND ADHESIVES
INVESTIGATOR: Frank W. Reinhart, Irma G. Callomon
CONTRACT: AF 33(616)-53-9, National Bureau of Standards
ABSTRACT: 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.

May 1959

SUBJECT: RESEARCH AND DEVELOPMENT ON ELEVATED TEMPERATURE RESISTANT STRUCTURAL METAL-TO-METAL ADHESIVES
INVESTIGATOR: Edward C. Janis, W. R. Boram, F. J. Riel, S. E. Susman
CONTRACT: AF 33(616)-5488, Narmco Industries, Inc.
ABSTRACT: This report describes research and development under Contract No. AF 33(616)-5488, "Research and Development on Elevated Temperature 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 at 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 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 condition.

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.

WADC TR 59-11, Sup 1

October 1959

SUBJECT: RESEARCH AND DEVELOPMENT ON ELEVATED TEMPERATURE
RESISTANT STRUCTURAL METAL-TO-METAL ADHESIVES
INVESTIGATOR: Edward C. Janis, Frank J. Riel, William R. Boram,
Samuel E. Susman
CONTRACT: AF 33(616)-5488, Narmco Industries, Inc.
ABSTRACT: 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 con-
ditions.

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.

WADC TR 59-82

June 1959

SUBJECT: INORGANIC HIGH TEMPERATURE ADHESIVES FOR METALS AND SANDWICH CONSTRUCTIONS
INVESTIGATOR: Roger A. Long, William Bassett
CONTRACT: AF 33(616)-5776, Narmco Industries, Inc.
ABSTRACT: 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 17-7 PH 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 contaminants which may prevent diffusion bonding.

WADC TR 59-113, Part I

July 1959

SUBJECT: RESEARCH AND DEVELOPMENT OF INORGANIC HIGH TEMPERATURE ADHESIVES FOR METALS AND COMPOSITE CONSTRUCTIONS
INVESTIGATOR: Joseph Bayer, William A. Patterson
CONTRACT: AF 33(616)-5538, Aeronca Manufacturing Corporation
ABSTRACT: 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.

July 1959

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.

WADC TR 59-152

July 1959

SUBJECT: STRENGTH PROPERTIES OF METAL-BONDING ADHESIVES AT TEMPERATURES FROM -100° TO 4800°F

INVESTIGATOR: H. W. Eickner

CONTRACT: DO 33(616)-58-1, Forest Products Laboratory

ABSTRACT: 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 4800°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.

WADC TR 59-260

November 1959

SUBJECT: STRENGTHS OF STRUCTURAL ADHESIVES AT TEMPERATURES DOWN TO MINUS 424°F

INVESTIGATOR: William M. Frost

CONTRACT: DO (33-616)-58-12, Cryogenic Engineering Laboratory

ABSTRACT: 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-phenolics but better bonding techniques could improve the performance of these epoxides.

February 1960

SUBJECT: ENVIRONMENTAL EXPOSURE OF ADHESIVE-BONDED METAL
LAP JOINTS

INVESTIGATOR: H. W. Eickner

CONTRACT: DO 33(616)-58-1, Forest Products Laboratory

ABSTRACT: 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.

EROSION

January 1960

SUBJECT: A STUDY OF RAIN EROSION TESTING METHODS FOR SUPER-SONIC SPEEDS

INVESTIGATOR: Donald E. Hurd, Roy F. Holmes

CONTRACT: AF 33(616)-3421, Thermodynamics Laboratories

ABSTRACTS: 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.

July 1959

SUBJECT: MECHANISM OF RAIN EROSION, PART XIII.
Mechanism Studies on Neoprene Coatings

INVESTIGATOR: Olive G. Engel

CONTRACT: AF 33(616)-58-12, National Bureau of Standards

ABSTRACT: 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.

May 1959

SUBJECT: RAIN EROSION FLIGHT TEST PROGRAMS

INVESTIGATOR: Samuel A. Marolo, George P. Peterson

ABSTRACT: 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 (3) 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.

PLASTICS

WADC TR 52-183, Sup 7

September 1959

SUBJECT: ANNUAL REPORT ON RESEARCH IN ANC-17 HANDBOOK
"PLASTICS FOR FLIGHT VEHICLES"
INVESTIGATOR: Donald G. Coleman
CONTRACT: AF 33(616)-58-1, Forest Products Laboratory
ABSTRACT: 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.

WADC TR 52-184, Sup 6

February 1960

SUBJECT: SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY
ON COMPOSITE CONSTRUCTION FOR FLIGHT VEHICLES
INVESTIGATOR: Donald G. Coleman
CONTRACT: AF 33(616)-58-1, Forest Products Laboratory
ABSTRACT: 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.

WADC TR 52-184, Sup 7

January 1960

SUBJECT: SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY
ON COMPOSITE CONSTRUCTION FOR FLIGHT VEHICLES
INVESTIGATOR: Donald G. Coleman
CONTRACT: AF 33(616)-58-1, Forest Products Laboratory
ABSTRACT: 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.

SUBJECT: NUCLEAR RADIATION OF REINFORCED PLASTIC MATERIALS
INVESTIGATOR: Richard L. Keller
ABSTRACT: The threshold 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 8.3×10^6 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.

SUBJECT: SOME PROPERTIES OF NOL-24 FINISHED FABRIC REINFORCED PLASTIC LAMINATES
INVESTIGATOR: Richard L. Keller
ABSTRACT: 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.

June 1959

SUBJECT: HEAT RESISTANT LOW PRESSURE PHENOLIC RESIN GLASS
INVESTIGATOR: Donald C. Biedenkapp
CONTRACT: AF 33(616)-3625, C. T. L., Inc.
ABSTRACT: 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.

May 1959

SUBJECT: EFFECT OF TYPE OF REINFORCEMENT ON FATIGUE PROPERTIES OF PLASTIC LAMINATES
INVESTIGATOR: G. H. Stevens, K. H. Boller
CONTRACT: DO 33(616)-58-1, Forest Products Laboratory
ABSTRACT: 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.

WADC TR 59-171

June 1959

SUBJECT: AN EVALUATION OF AN IMPROVED PROCEDURE FOR DETERMINING
THE COMPRESSIVE PROPERTIES OF REINFORCED PLASTICS

INVESTIGATOR: Richard J. McBride

CONTRACT: AF 33(616)-5500, University of Dayton

ABSTRACT: 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; Fuggenberger 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 resulted 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.

WADC TR 59-216

September 1959

SUBJECT: EFFECT OF ELEVATED TEMPERATURES ON WEIGHT LOSS AND FLEXURAL PROPERTIES OF THREE REINFORCED PLASTIC LAMINATES

INVESTIGATOR: Kenneth H. Boller, Kenneth E. Kimball

CONTRACT: D033(616)-58-1, Forest Products Laboratory

ABSTRACT: 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.

WADC TR 59-229

September 1959

SUBJECT: STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES AT ELEVATED TEMPERATURES DC 2106 Resin and 181 Heat-Cleaned Glass Fabric

INVESTIGATOR: Kenneth H. Boller, Kenneth E. Kimball

CONTRACT: D033(616)-58-1, Forest Products Laboratory

ABSTRACT: 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.

June 1959

SUBJECT: EXPLORATORY INVESTIGATION OF INORGANIC FIBER REINFORCED INORGANIC LAMINATE

INVESTIGATOR: George Margo

CONTRACT: AF 33(616)-5251, Goodyear Aircraft Corp.

ABSTRACT: 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 mat, 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 59-328

October 1959

SUBJECT: HEAT RESISTANT LAMINATING RESINS

INVESTIGATOR: Morton H. Gollis, John J. O'Connell

CONTRACT: AF 33(616)-5682, Monsanto Chemical Co.

ABSTRACT: This report describes the progress made toward the

October 1959

SUBJECT: HEAT RESISTANT LAMINATING RESINS
INVESTIGATOR: Morton H. Gollis, John J. O'Connell
CONTRACT: AF 33(616)-5682, Monsanto Chemical Co.
ABSTRACT: 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.

December 1959

SUBJECT: THE STUDY OF ABLATION OF STRUCTURAL PLASTIC MATERIALS
INVESTIGATOR: Franklin A. Vassalo, N. E. Wahl, G. A. Sterbutzel, J. Beal
CONTRACT: AF 33(616)-5683, Cornell Aeronautical Laboratory, Inc.
ABSTRACT: 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 de-

pendence 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/ft²-sec. are given for each material tested.

WADC TR 59-569

January 1960

SUBJECT: STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES
AT ELEVATED TEMPERATURES (CTL-91LD Phenolic Resin
and 181-Al100 Glass Fabric)

INVESTIGATOR: Kenneth H. Boller

CONTRACT: D033(616)-58-1, Forest Products Laboratory, Forest
Service

ABSTRACT: 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-Al100 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.

SUBJECT: BEHAVIOR OF PLASTIC MATERIALS IN HYPERTHERMAL ENVIRONMENTS

INVESTIGATOR: Donald L. Schmidt

ABSTRACT: 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.

SUBJECT: MECHANISM OF REINFORCEMENT OF FIBER-REINFORCED STRUCTURAL PLASTICS AND COMPOSITES

INVESTIGATOR: J. S. Islinger, K. Gutfreund, R. G. Maguire, O. H. Olson

CONTRACT: AF33(616)-5983, Armour Research Foundation

ABSTRACT: 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 59-604

February 1960

SUBJECT: AN EVALUATION OF MACRO-TEST PROCEDURES FOR DETERMINING
THE MECHANICAL PROPERTIES OF REINFORCED PLASTICS

INVESTIGATOR: Richard J. McBride

CONTRACT: AF 33(616)-5500, University of Dayton

ABSTRACT: 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.

WADC TR 59-668, Part I

February 1960

SUBJECT: A STUDY OF THE MECHANISM OF ABLATION OF REINFORCED
PLASTICS

INVESTIGATOR: R. Y. Mixer, C. W. Marynowski

CONTRACT: AF 33(616)-5964, Stanford Research Institute

ABSTRACT: 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 environments (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.

WADC TR 60-90

March 1960

SUBJECT: THERMAL PARAMETERS OF RE-ENTRY ABLATIVE PLASTICS

INVESTIGATOR: Donald L. Schmidt

ABSTRACT: The unique behavior of plastic materials in very

WADC TR 53-373 Sup 7

111

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.

SANDWICH CONSTRUCTION

WADD TR 60-133, OTS Release

February 1960

SUBJECT: CYLINDRICAL SANDWICH CONSTRUCTION DESIGN
INVESTIGATOR: Sidney Allinkov
ABSTRACT: 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.

FIBROUS MATERIALS

WADC TR 58-285, Part II, OTS Release

March 1960

SUBJECT: DEVELOPMENT OF HIGH MODULUS FIBERS FROM HEAT RESISTANT MATERIALS
INVESTIGATOR: J. Frees Brossy, J. D. Provance
CONTRACT: AF 33(616)-5263, Houze Glass Corp.
ABSTRACT: 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.

WADC TR 58-298, Part II

June 1959

SUBJECT: EXPLORATORY INVESTIGATION OF INORGANIC FIBER REINFORCED INORGANIC LAMINATE
INVESTIGATOR: George Margo
CONTRACT: AF 33(616)-5251, Goodyear Aircraft Corp.
ABSTRACT: 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 mat, 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 58-602, OTS Release

May 1959

SUBJECT: A SURVEY OF 18-OUNCE BLENDED SERGE FABRICS LABORATORY EVALUATION

INVESTIGATOR: John Menkart

CONTRACT: AF 33(616)-54-172; 55-82; 57-20, Harris Research Laboratories, Inc.

ABSTRACT: 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.

September 1959

SUBJECT: MASS TRANSFER COOLING OF PARACHUTE MATERIALS
INVESTIGATOR: Rodney H. Cornish, Charles W. Beadle, Franklin J. Ahimaz, Kenneth Foster
CONTRACT: AF 33(616)-5852, Department of Engineering Mechanics and Materials, Cornell University
ABSTRACT: 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, Chloro-anthraquinone, Methyl-anthraquinone, Hydroxy-anthraquinone, 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.

September 1959

SUBJECT: CANDIDATE MATERIALS FOR HIGH TEMPERATURE FABRICS
CONTRACT: AF 33(616)-5880, Arthur D. Little, Inc.
ABSTRACT: 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.

WADC TR 59-155 (Continued)

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.

WADC TR 59-242, OTS Release

October 1959

SUBJECT: A SURVEY OF 18-OZ. BLENDED SERGE FABRICS: STABILITY OF FABRICS TO MOISTURE AND R. H. MOVEMENT

INVESTIGATOR: Norman J. Abbott, Leo Barish, Milton M. Platt

CONTRACT: AF 33(616)-54-172; 55-82; 57-20, Fabric Research Laboratories, Inc.

ABSTRACT: 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.

WADC TR 59-250

June 1959

SUBJECT: INVESTIGATION OF A FUNGICIDAL TANNAGE

INVESTIGATOR: Lewellyn G. Picklesimer

ABSTRACT: A new tannage for leather has been developed. The

WADC TR 59-250 (Continued)

tanning material is the reaction product of tetrakis (hydroxymethyl) Phosphonium Chloride $(\text{HOCH}_2)_4 \text{PCl}$ 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.

WADC TR 59-374, OTS Release

March 1960

SUBJECT: AIR FLOW CHARACTERISTICS OF PARACHUTE FABRICS AT SIMULATED HIGH ALTITUDES
INVESTIGATOR: C. V. Seshadri, G. A. Brown, S. Backer, J. G. Krizik, D. M. Mellen
CONTRACT: AF 33(616)-5864, Textile Division, Massachusetts Institute of Technology
ABSTRACT: 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.

WADC TR 59-397, OTS Release

January 1960

SUBJECT: DEVELOPMENT OF SHADE STANDARD AND TOLERANCES FOR USAF BLUE 85 SERVICE OVERCOAT
INVESTIGATOR: John T. Walwood, Frank J. Rizzo, Constantine J. Megas, Aldo M. Crugnola

WADC TR 59-397 (Continued)

CONTRACT: AF 33(616)-53-221, Quartermaster Research & Engineering Command, U. S. Army
ABSTRACT: 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.

WADC TR 59-694, OTS Release

March 1960

SUBJECT: ANALYSIS OF WEBBING IMPACT DATA AND DETERMINATION OF OPTIMUM INSTRUMENTATION TO BE USED IN CONJUNCTION WITH THE IMPACTING OF WEBBING
INVESTIGATOR: R. B. Williams, R. J. Benjamin
CONTRACT: AF 33(616)-6440, Cook Research Laboratories
ABSTRACT: 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.

March 1960

SUBJECT: DEVELOPMENT OF TEXTILE TYPE VITREOUS SILICA YARNS
INVESTIGATOR: W. Wendell Drummond, Burns A. Cash
CONTRACT: AF 33(616)-6255, Bjorksten Research Laboratories, Inc.
ABSTRACT: 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.

WADC TR 59-703

January 1960

SUBJECT: TREATMENT OF COTTON FABRIC WITH FORMULATIONS OF 2,2'-DIHYDROXY-5,5'-DIFLUORODIPHENYL SULFIDE
INVESTIGATOR: Charles C. Yeager, Jay C. Chapin
CONTRACT: AF 33(616)-38797, Scientific Oil Compounding Co.
ABSTRACT: 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.

FUELS, LUBRICANTS AND FLUIDS

FUELS

WADC TR 59-108

September 1959

SUBJECT: RESEARCH ON THE EVALUATION OF NEUTRON AND GAMMA
IRRADIATION EFFECTS OF SIX HYDROCARBON-TYPE
FUELS

INVESTIGATOR: A. C. Nixon, T. R. Lusebrink

CONTRACT: AF 33(616)-6052, Shell Development Co.

ABSTRACT: 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 result, 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 59-663

February 1960

SUBJECT: FLAMMABILITY CHARACTERISTICS OF HIGH TEMPERATURE
HYDROCARBON FUELS

INVESTIGATOR: J. M. Kuchta, S. Lambiris, H. E. Perlee, D. R.
Halagan, M. G. Zabetakis

CONTRACT: DO 33(616)-57-4, Bureau of Mines

ABSTRACT: 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.

HYDRAULIC FLUIDS

WADC TR 57-294, Part II, OTS Release

May 1959

SUBJECT: RESEARCH ON LIQUID METALS AS POWER TRANSMISSION FLUIDS
INVESTIGATOR: Robert C. Kumpitsch
CONTRACT: AF 33(616)-3698, General Electric Company
ABSTRACT: The behavior of liquid metal NaK-77 (eutectic 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

WADC TR 57-294, Part II (Continued)

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. continuous flow, staged gear pump as a prime mover.

WADC TR 58-171, Part II

June 1959

SUBJECT: DEVELOPMENT OF HIGH TEMPERATURE OXIDATION-CORROSION INHIBITORS TO IMPROVE STABILITY OF HIGH TEMPERATURE HYDRAULIC FLUIDS AND LUBRICANTS

INVESTIGATOR: Robert Brunier

CONTRACT: AF 33(616)-5101, Armour Research Foundation

ABSTRACT: 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.

WADC TR 58-213

April 1959

SUBJECT: SILICONE FLUIDS AS HIGH TEMPERATURE JET ENGINE OILS
AND HYDRAULIC FLUIDS

INVESTIGATOR: George Baum, Harold W. Adams

ABSTRACT: 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 lubricating 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.

WADC TR 58-287, Part I

June 1959

SUBJECT: STORAGE STABILITY OF FLUIDS AND LUBRICANTS (DESERT
STORAGE OF AIRCRAFT OILS)

INVESTIGATOR: John B. Christian

ABSTRACT: 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.

WADC TR 53-373 Sup 7

123

WADC TR 58-287, Part I (Continued)

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 TR 58-407, Part I

June 1959

SUBJECT: FORMULATION OF SILANE BASE HIGH TEMPERATURE
HYDRAULIC FLUID
INVESTIGATOR: Arthur W. Sawyer, Edward E. Harris
CONTRACT: AF 33(616)-5292, Olin Mathieson Chemical Corp.
ABSTRACT: 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% N₂ - 5% O₂. 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. In general, corrosion of test metals has been negligible.

Antiwear additives were evaluated on a Modified Shell Four Ball Wear Tester. Tests were run at 500°F and 700°F, using M-10 steel balls. Several types of additives are effective at 500°F but a highly refined sodium petroleum sulfonate appears superior at 700°F. Test results show that approximate target properties are obtained.

Composition of, and physical data on fifty gallon samples of hydraulic fluid and engine oil formulations, supplied to Wright Air Development Center, are reported.

June 1959

SUBJECT: FORMULATION OF SILANE BASE HIGH TEMPERATURE
HYDRAULIC FLUID

INVESTIGATOR: Arthur W. Sawyer

CONTRACT: AF 33(616)-5292, Olin Mathieson Chemical Corporation

ABSTRACT: 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 anti-oxidants 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.

October 1959

SUBJECT: DEVELOPMENT OF NUCLEAR RADIATION-RESISTANT
HYDRAULIC FLUIDS

INVESTIGATOR: N. W. Furby, S. R. Calish, M. A. Pino, D. R.
Wilgus

CONTRACT: AF 33(616)-5583, California Research Corp.

ABSTRACT: 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 C₁₄₋₁₆ alkyl diphenyl ether-p-xylene copolymer was pre-
pared 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 oxi-
dative, 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 C₁₄₋₁₆-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 con-
tributing to stability against radiation, thermal, oxidative, shear,
and hydrolytic damage was gained. A radiation process which enhances
shear stability of thickened fluids was discovered.

LUBRICANTS

WADC TR 55-30, Part VII

June 1959

SUBJECT: FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS
INVESTIGATOR: E. Erwin Klaus, Merrell R. Fenske
CONTRACT: AF 33(616)-5460, Petroleum Refining Laboratory
ABSTRACT: 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.

WADC TR 57-36, Part IV

June 1959

SUBJECT: AIRFRAME LUBRICANTS, PART IV Status Report
CONTRACT: AF 33(616)-5684, Coordinating Research Council, Inc.
ABSTRACT: This report reviews the activities of the various

WADC TR 57-36, Part IV (Continued)

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 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.

WADC TR 57-61, Part III

July 1959

SUBJECT: LUBRICATION OF TITANIUM
INVESTIGATOR: Nicholas Fatica
CONTRACT: AF 33(616)-3350, Clevite Research Center
ABSTRACT: 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.

WADC TR 57-61, Part III (Continued)

Carburized iron-plated titanium was also investigated to a limited extent and found to give results approaching 52100 steel.

WADC TR 57-773, Part II

May 1959

SUBJECT: EVALUATION OF HIGH TEMPERATURE GEAR AND SPLINE LUBRICANTS
INVESTIGATOR: Donald J. Fessett
CONTRACT: AF 33(616)-5631, Western Gear Corporation
ABSTRACT: 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 Tetracaprate
MLO 58-431 -- 10% silicone, 90% pentaerythritol tetracaprate
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 to 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.

April 1960

SUBJECT: STORAGE STABILITY OF FLUIDS AND LUBRICANTS, PART
II. Room Temperature Storage of Aircraft Greases
INVESTIGATOR: John B. Christian
ABSTRACT: 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.

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.

May 1959

SUBJECT: DEVELOPMENT OF HIGH TEMPERATURE HEAVY LOAD-CARRYING GREASES
INVESTIGATOR: Robert K. Smith, William Eismann, Jr.
CONTRACT: AF 33(616)-5156, E. F. Houghton & Co.
ABSTRACT: 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.

WADC TR 58-288, Part II (Continued)

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.

WADC TR 58-297, Part II

June 1959

SUBJECT: DEVELOPMENT OF EVALUATION TECHNIQUES FOR DETERMINATION OF THE LUBRICITY AND STABILITY OF NEW HIGH-TEMPERATURE LUBRICANTS AND HYDRAULIC FLUIDS

INVESTIGATOR: Vernice Hopkins, Andrew D. St John

CONTRACT: AF 33(616)-5202, Midwest Research Institute

ABSTRACT: 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.

WADC TR 58-297, Part II (Continued)

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.

WADC TR 58-335, Part I

January 1959

SUBJECT: SYNTHESIS AND EVALUATION OF HIGH TEMPERATURE ANTI-OXIDANTS FOR SYNTHETIC HYDRAULIC FLUIDS AND LUBRICANTS

INVESTIGATOR: R. M. Silverstein

CONTRACT: AF 33(616)-5276, Stanford Research Institute

ABSTRACT: During the period covered by the current report, candidate anti-oxidants 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 warrant 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 anti-oxidant activity.

None of the additives evaluated to date showed any anti-oxidant activity in the silane fluids or in the mineral oils.

June 1959

SUBJECT: SYNTHESIS AND EVALUATION OF HIGH TEMPERATURE
ANTI-OXIDANTS FOR SYNTHETIC HYDRAULIC FLUIDS AND
LUBRICANTS

INVESTIGATOR: Robert M. Silverstein

CONTRACT: AF 33(616)-5276, Stanford Research Institute

ABSTRACT: 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 tetracaprate 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.

June 1959

SUBJECT: DEVELOPMENT OF GREASES FOR HIGH SPEED BALL AND
ROLLER BEARINGS

INVESTIGATOR: Paul R. McCarthy, Guy C. Blewett, Joseph J.
McGrath

CONTRACT: AF 33(616)-5020, Gulf Research & Development Co.

ABSTRACT: 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 experi-
mental 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.

WADC TR 58-350, Part II (Continued)

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.

WADC TR 59-48

June 1959

SUBJECT: MODIFICATION OF SHELL FOUR-BALL WEAR TESTER FOR 800°F OPERATION AND DEVELOPMENT OF A LUBRICANT EVALUATION TECHNIQUE

INVESTIGATOR: Vernice Hopkins

CONTRACT: AF 33(616)-5202, Midwest Research Institute

ABSTRACT: 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.

WADC TR 59-99

May 1959

SUBJECT: DEVELOPMENT OF GREASES AND SEALS FOR 275°F PNEUMATIC SYSTEMS

INVESTIGATOR: A. A. LePera, John Q. Griffith

ABSTRACT: 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.

WADC TR 59-99 (Continued)

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.

WADC TN 59-160, Part I, OTS Release

August 1959

SUBJECT: HIGH TEMPERATURE EVALUATION PROCEDURES FOR LUBRICANTS
INVESTIGATOR: Vernon A. Lauer, Donald C. Trop
ABSTRACT: 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 $\pm$ 5°F. This report also describes two high temperature viscosity baths capable of attaining and maintaining 400°F and 700°F respectively.

WADC TR 59-166, OTS Release

June 1959

SUBJECT: HEAT CAPACITY DETERMINATION OF MINERAL AND SYNTHETIC ENGINE OILS, LUBRICANTS, FUELS AND HYDRAULIC FLUIDS IN THE TEMPERATURE RANGE 70° - 500°F
INVESTIGATOR: T. M. Medved, C. E. Hensen, C. C. Bolze, J. W. Barger
CONTRACT: AF 33(616)-5269, Midwest Research Institute
ABSTRACT: The heat capacities of 33 mineral and synthetic engine oils, lubricants, fuels, and hydraulic fluids were measured over a temperature range of 80° 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.

September 1951

SUBJECT: NUCLEAR RADIATION RESISTANT HIGH TEMPERATURE LUBRICANTS

INVESTIGATOR: C. L. Mahoney, E. R. Barnum, W. S. Saari, K. J. Sax, W. W. Kerlin

CONTRACT: AF 33(616)-5617, Shell Development Co.

ABSTRACT: 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.

July 1959

SUBJECT: THERMAL CONDUCTIVITY OF LUBRICATING OILS AND HYDRAULIC FLUIDS

INVESTIGATOR: D. W. McCready

CONTRACT: AF 33(616)-5745, University of Michigan

ABSTRACT: 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".

WADC TR 59-191, Part I

September 1959

SUBJECT: RESEARCH AND DEVELOPMENT ON HIGH TEMPERATURE
ADDITIVES FOR LUBRICANTS AND HYDRAULIC FLUIDS
INVESTIGATOR: Kathleen Brown, Kenneth L. McHugh, Emery N. Wescott,
J. O. Smith
ABSTRACT: 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, silane 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 59-203

September 1959

SUBJECT: HIGH TEMPERATURE METALLO-ORGANIC AND INORGANIC BASE FLUIDS
INVESTIGATOR: L. W. Breed, F. V. Morriss
CONTRACT: AF 33(616)-5718, Midwest Research Institute
ABSTRACT: 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 59-244

October 1959

SUBJECT: PROCEEDINGS OF THE AIR FORCE-NAVY-INDUSTRY LUBRICANTS CONFERENCE
INVESTIGATOR: Robert J. Benzing
ABSTRACT: 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.

WADC TR 59-603, OTS Release

January 1960

SUBJECT: FRICTION AND WEAR AT ELEVATED TEMPERATURES
INVESTIGATOR: Ernest Rabinowicz
CONTRACT: AF 33(616)-5963, Massachusetts Institute of Technology
ABSTRACT: 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 and on an older machine. These and other results are discussed theoretically in terms of the surface energy--hardness ratio and of the wear

WADC TR 59-603 (Continued)

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 59-633, Part I

April 1960

SUBJECT: HIGH TEMPERATURE INSTRUMENT OIL
INVESTIGATOR: Albert A. Schwartz, S. Frank Murray, Harry R. Broadley, Jr., Robert S. Norman
CONTRACT: 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 re-

WADC TR 59-633, Part I (Continued)

designed 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 59-764, OTS Release

March 1960

SUBJECT: LUBRICATION BEHAVIOR OF LIQUID METALS
INVESTIGATOR: Patrick H. McDonald
CONTRACT: 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 macroscopic 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.

LUBRICANTS, SOLID FILM

WADC TR 57-455, Part III

June 1959

SUBJECT: HIGH TEMPERATURE SOLID DRY FILM LUBRICANTS
INVESTIGATOR: Melvin T. Lavik
CONTRACT: AF 33(616)-3684, Midwest Research Institute
ABSTRACT: 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.

ELECTRICAL AND ELECTRONIC MATERIALS

WADC TR 58-275, Part II, OTS Release

October 1959

SUBJECT: FABRICATION OF INFRARED TRANSMITTING MATERIALS BY HOT PRESSING TECHNIQUES
INVESTIGATOR: Norbert J. Kreidl, Eugene C. Letter, Harold C. Hafner, Dean A. Buckner, J. Raymond Hensler
CONTRACT: AF 33(616)-5123, Bausch & Lomb Optical Co.
ABSTRACT: 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
3
Ferric oxide, MgO , ZnO , Co_3O_4 , Cu_2O and $\text{CoO} \cdot \text{Al}_2\text{O}_3$ 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.

WADC TR 59-303, OTS Release

November 1959

SUBJECT: METHODS OF PURIFICATION OF METALS AND INTER-METALLIC COMPOUNDS
INVESTIGATOR: Sherman Susman
CONTRACT: AF 33(616)-5895, Armour Research Foundation
ABSTRACT: 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 ceria-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 calcium 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 $J_t S_t / C_{o1}$ ratios for copper, calcium and aluminum are found to be 113, 194, and 58 respectively.

October 1959

SUBJECT: SYNTHESIS AND PURIFICATION OF DIELECTRIC MATERIALS
INVESTIGATOR: T. W. Dakin, R. N. Wenzel, W. C. Divens, D. H.
Hogle, D. W. Lewis, P. A. Tierney
CONTRACT: AF 33(616)-5979, Westinghouse Electric Corp.
ABSTRACT: 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 fluorphlogopite. Work to improve the physical and electric strength characteristics of the paper and electrophoretic mica coatings is continuing.

NUCLEAR MEASUREMENTS

WADC TR 59-4, OTS Release

December 1959

SUBJECT: MEASUREMENT OF SCATTERED GAMMA RAYS BY InFOIL
ACTIVATION
INVESTIGATOR: Michael P. Charles
ABSTRACT: 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 have 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 59-79, OTS Release

May 1959

SUBJECT: INVESTIGATION OF A NEW DYE-GLASS GAMMA RADIATION
DOSIMETER
INVESTIGATOR: Denver Hale
ABSTRACT: 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 absorbancy 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 TR 59-119

April 1959

SUBJECT: REMOVAL DOSE AS AN ENVIRONMENTAL MEASUREMENT OF
X-RAYS AND GAMMA RAYS

INVESTIGATOR: R. L. Hickmott

ABSTRACT: 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 59-167

June 1959

SUBJECT: RADIATION INDUCED ULTRA-VIOLET ABSORPTION IN
METHYL METHACRYLATE AS A METHOD OF DOSIMETRY

INVESTIGATOR: D. R. Johnson

ABSTRACT: 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 59-372

January 1960

SUBJECT: CHEMICAL DOSIMETRY

INVESTIGATOR: Dennis R. Johnson, Lowell A. King, Gordon G. Wepfer

ABSTRACT: In radiation effects and radiation chemistry

January 1960

SUBJECT: CHEMICAL DOSIMETRY
INVESTIGATOR: Dennis R. Johnson, Lowell A. King, Gordon Wepfer
ABSTRACT: 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 TN 59-401, OTS Release

November 1959

SUBJECT: ATTEMPT TO EVALUATE CHLORINATED HYDROCARBON
DOSIMETERS BY CROSS CALIBRATING ELEVEN GAMMA RAY
SOURCES
INVESTIGATOR: R. L. Hickmott, Maurice J. Cote, Philip B. Hemmig
ABSTRACT: 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.

WADD TN 60-17

March 1960

SUBJECT: INCREASING THE DOSE RATE OF THE WADD KILO-CURIE
COBALT-60 FACILITY BY ALTERING ITS GEOMETRY
INVESTIGATOR: D. J. Dunbar, D. R. Johnson, R. E. Rondeau
ABSTRACT: 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 whose inside 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 configura-
tion 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, 4.0×10^7 ergs gm^{-1} (C) hr^{-1} .

WADC TN 60-48, OTS Release

March 1960

SUBJECT: DESIGN OF A DUAL SENSOR GAMMA RAY CALORIMETER
INVESTIGATOR: James R. Coss, Philip B. Hemmig
ABSTRACT: Design for a small, dimple, dual element calori-
meter 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.

THERMO PHYSICS

WADC TR 59-87, Part II

July 1959

SUBJECT: DETERMINATION OF FACTORS GOVERNING SELECTION AND APPLICATION OF MATERIALS FOR ABLATION COOLING OF HYPERVELOCITY VEHICLES

INVESTIGATOR: John H. Bonin, Channon F. Price, Donald E. Taylor

CONTRACT: AF 33(616)-5436, Chicago Midway Laboratories

ABSTRACT: 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.

WADC TR 59-87, Part III, OTS Release

February 1960

SUBJECT: DETERMINATION OF FACTORS GOVERNING SELECTION AND APPLICATION OF MATERIALS FOR ABLATION COOLING OF HYPERVELOCITY VEHICLES

INVESTIGATOR: J. H. Bonin, C. F. Price, H. Halle

CONTRACT: AF 33(616)-5436, Chicago Midway Laboratories

ABSTRACT: 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.

WADC TN 59-215, OTS Release

December 1959

SUBJECT: THERMAL PROPERTIES OF MATERIALS AT ELEVATED TEMPERATURES

INVESTIGATOR: Herbert W. Deem, Webster D. Wood, Charles F. Lucks

CONTRACT: AF 33(616)-5849, Battelle Memorial Institute

ABSTRACT: 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

WADC TN 59-215 (Continued)

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.

WADC TR 59-273, OTS Release

February 1960

SUBJECT: A RELAXATION TIME TECHNIQUE FOR MEASUREMENT OF THERMAL DIFFUSIVITY
INVESTIGATOR: George Sonnenschein, Robert A. Winn
ABSTRACT: 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 $1.0 \text{ cm}^2/\text{sec}$. and temperatures between 25 and 950°C .

WADC TR 59-276, Part I, OTS Release

December 1959

SUBJECT: THERMAL DIFFUSIVITY OF IRON. PART I. A Generator for Producing Sinusoidal Temperatures
INVESTIGATOR: Howard W. Flieger, Jr., Defoe C. Ginnings
CONTRACT: AF 33(616)-57-6, National Bureau of Standards
ABSTRACT: 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

WADC TR 59-276, Part I (Continued)

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.

WADC TR 59-346, OTS Release

February 1960

SUBJECT: PLASMA JET TEMPERATURE STUDY
INVESTIGATOR: Dr. Willard J. Pearce
CONTRACT: AF 33(616)-5848, General Electric Company
ABSTRACT: 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 $I = KgA \lambda \exp(-E/kT)$, 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

λ = 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.

January 1960

SUBJECT: THE REMANENCE OF ALNICO V AND VI MAGNETS BETWEEN ROOM TEMPERATURE AND 550°C
INVESTIGATOR: Rudolf K. Tenzer
CONTRACT: AF 33(616)-3385, Indiana Steel Products Co.
ABSTRACT: 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 closely 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 $\pm 1\%$ 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.

March 1960

SUBJECT: STANDARDIZATION OF THERMAL EMITTANCE MEASUREMENTS
INVESTIGATOR: W. N. Harrison, J. C. Richmond, E. K. Plyler, R. Stair, H. K. Skramstad
CONTRACT: AF 33(616)-58-20, National Bureau of Standards
ABSTRACT: 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.

ANALYTICAL TECHNIQUES

WADC TN 58-361, OTS Release

May 1959

SUBJECT: RAPID SEPARATION AND GRAVIMETRIC DETERMINATION OF ALUMINUM IN FERROUS METALS
INVESTIGATOR: Lois A. Keyser, Charles D. Houston
ABSTRACT: 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.

WADC TN 58-368

May 1959

SUBJECT: THE QUANTITATIVE ANALYSIS OF 5-ETHYL-10,10-DI-PHENYLPHENAZASILINE IN AROMATIC BASE STOCK FLUID PRIOR TO PERFORMANCE TESTING
INVESTIGATOR: Lt James T. Thompson
ABSTRACT: 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 TR 59-68, OTS Release

October 1959

SUBJECT: ELECTRON MICROSCOPY OF ETCH PITS
INVESTIGATOR: Richard E. Pawel, 1/Lt, USAF
ABSTRACT: 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 59-107, OTS Release

July 1959

SUBJECT: A MASS SPECTROMETER SYSTEM FOR MATERIALS RESEARCH SUMMARY REPORT, PHASE I
INVESTIGATOR: Charles F. Robinson, George D. Perkins, Norton W. Bell

WADC TR 59-68 (Continued)

CONTRACT: AF 33(616)-5571, Consolidated Electrodynamics Corp.
ABSTRACT:

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.

WADC TR 59-139, OTS Release

October 1959

SUBJECT: A HIGH TEMPERATURE X-RAY DIFFRACTOMETER SPECIMEN MOUNT

INVESTIGATOR: Lt William L. Baun

ABSTRACT: 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.

WADC TR 59-325, OTS Release

September 1959

SUBJECT: ION EXCHANGE AND OTHER CHEMICAL METHODS FOR BERYLLIUM BASE ALLOYS

INVESTIGATOR: Silve Kallmann, Robert Liu, Hans Oberthin

CONTRACT: AF 33(616)-5743, Ledoux & Co.

ABSTRACT: 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

WADC TR 59-325 (Continued)

covered in the range of 0.1 to 10% are copper, aluminum, iron, nickel, cobalt, cerium, silver, gold and palladium.

WADC TR 59-344, OTS Release

September 1959

SUBJECT: THE DETERMINATION OF NEAR INFRARED SPECTRA
INVESTIGATOR: Scott Anerson, Raymond Isaac, Myra Blankenship
CONTRACT: AF 33(616)-5283, Anderson Physical Laboratory
ABSTRACT: 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, on a Beckman DK-2 Ratio Recording Spectrophotometer. Modification of the instrument's recording, to accomodate 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.

WADC TR 59-431

December 1959

SUBJECT: SYNTHESIS, INFRARED SPECTRA, AND X-RAY POWDER DIFFRACTION DATA OF SOME 1, 3, 5, TRIAZINE DERIVATIVES
INVESTIGATOR: Herbert K. Reimschuessel, Neil T. McDevitt, William L. Baun
ABSTRACT: 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.

February 1960

SUBJECT: INVESTIGATION OF FAR INFRARED SPECTRA
INVESTIGATOR: Richard C. Lord
CONTRACT: AF 33(616)-5578, Massachusetts Institute of Technology
ABSTRACT: Applications of a small grating spectrometer to problems of molecular structure by investigation of infrared absorption spectra in the region $50 - 300 \text{ cm}^{-1}$ 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 ($50 - 600 \text{ cm}^{-1}$) 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.

January 1960

SUBJECT: THE QUANTITATIVE ANALYSIS OF N-PHENYL- α -NAPHTHYLAMINE AND 5-ETHYL-10, 10-DIPHENYLPHENAZASILINE IN ALIPHATIC TRIESTER BASE STOCK FLUID
INVESTIGATOR: Lee D. Smithson, James T. Thompson
ABSTRACT: A quantitative ultra-violet method has been developed for determining the quantity of two antioxidant additives (n-phenyl- α -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.

February 1960

SUBJECT: A NEW INSTRUMENT FOR THE DETERMINATION OF MOLECULAR WEIGHT BY DIFFERENTIAL VAPOR PRESSURE
INVESTIGATOR: N. M. Wiederhorn, J. H. Vreeland, R. H. Thompson

CONTRACT: AF 33(616)-5483, Arthur D. Little, Inc.

ABSTRACT: The differential vapor pressure molecular weight instrument developed previously was subjected to use and further evaluated with regard to sensitivity, reproducibility, and accuracy. To 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 $\pm 5\%$. 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.

WADD TR 60-55, OTS Release

March 1960

SUBJECT: ON THE FEASIBILITY OF USING X-RAY FLASH TECHNIQUES FOR DIFFRACTION STUDIES

INVESTIGATOR: werner R. Rambaske

CONTRACT: AF 33(616)-6607, university of Dayton

ABSTRACT: 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 diffractograms 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.

DESIGN CRITERIA

WADC TR 55-150, Part 8

September 1959

SUBJECT: 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

INVESTIGATOR: R. J. Favor, W. P. Achbach, H. J. Grover

CONTRACT: AF 33(616)-3965, Battelle Memorial Institute

ABSTRACT: 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 con-
ditions 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 ade-
quately the regions of interest specified. Reasons for this con-
clusion are discussed in this report. It should be emphasized that
for creep strain above about 1 per cent, both sources of data appear
adequate.

WADC TR 56-330, Part XI, OTS Release

September 1959

SUBJECT: THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE
TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE
FROM VARIOUS CLASSES OF METALS Part XI, Photo-
elastic Analysis of I-Beams With Elliptic-Type
Web Cutouts

INVESTIGATOR: Will J. Worley

CONTRACT: AF 33(616)-5658, University of Illinois

ABSTRACT: 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 AF33(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

WADC TR 56-330, Part XI (Continued)

web cutout. The method shows reasonable agreement when compared with available data on the actual aluminum alloy I-Beam.

WADC TR 56-330, Part XII, OTS Release

February 1960

SUBJECT: 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

INVESTIGATOR: O. M. Sidebottom, S. Dharmarajan, J. L. Gubser, J. D. Leasure

CONTRACT: AF 33(616)-5658, University of Illinois

ABSTRACT: 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 56-645, Part III

October 1959

SUBJECT: PROPERTIES OF GLASSES AT ELEVATED TEMPERATURES

INVESTIGATOR: M. J. Kerper, C. C. Diller, E. H. Eimer

CONTRACT: AF 33(616)-56-13, National Bureau of Standards

ABSTRACT: 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

WADC TR 56-645, Part III (Continued)

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.

WADC TR 58-69, Part II, OTS Release

January 1960

SUBJECT: ON STRESS INTERACTION IN FATIGUE AND A CUMULATIVE DAMAGE RULE PART II. 7075 Aluminum Alloy
INVESTIGATOR: Alfred M. Freudenthal, Robert A. Heller
CONTRACT: AF 33(616)-3982, Columbia University
ABSTRACT: 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.

WADC TR 59-25, OTS Release

February 1960

SUBJECT: INVESTIGATION OF THE UNNOTCHED AND NOTCHED FATIGUE BEHAVIOR OF SEVERAL HEAT RESISTANT MATERIALS FOR ENGINE BOLTS
INVESTIGATOR: D. M. Forney, Jr., Douglas Y. Wang
ABSTRACT: 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.

WADC TR 59-484, OTS Release

December 1959

SUBJECT: SHORT TIME, ELEVATED TEMPERATURE STRESS-STRAIN
BEHAVIOR OF TENSILE, COMPRESSIVE, AND COLUMN
MEMBERS

INVESTIGATOR: Eugene C. Bennett

CONTRACT: AF 33(616)-6043, Marquardt Corp.

ABSTRACT: 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.

WADC TR 59-572, OTS Release

March 1960

SUBJECT: INVESTIGATION OF THE COMPRESSIVE, BEARING, AND
SHEAR CREEP-RUPTURE PROPERTIES OF AIRCRAFT
STRUCTURAL METALS AND JOINTS AT ELEVATED TEMPERA-
TURES

INVESTIGATOR: Luke A. Yerkovich

CONTRACT: AF 33(616)-5772, Cornell Aeronautical Laboratory, Inc.

ABSTRACT: 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.

WADC TR 59-702, Part I, OTS Release

March 1960

SUBJECT: MECHANICAL PROPERTIES OF SELECTED ALLOYS AT ELEVATED TEMPERATURES
INVESTIGATOR: Harry A. Pearl, George F. Kappelt, Edmund J. King
CONTRACT: AF 33(616)-5760, Bell Aircraft Corp.
ABSTRACT: 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:

<u>Materials</u>	<u>Testing Temperature Range</u>
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

WADC TR 59-702, Part I (Continued)

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.

WADC TR 59-702, Part II, OTS Release

March 1960

SUBJECT: MECHANICAL PROPERTIES OF SELECTED ALLOYS AT
ELEVATED TEMPERATURES PART II. Design Criteria
of Silicon Carbide

INVESTIGATOR: Harry A. Pearl, John M. Nowak, Harry G. DeBan

CONTRACT: AF 33(616)-5760, Bell Aircraft Corporation

ABSTRACT: A study was made of nondestructively testing
silicon carbide by density and density uniformity, dynamic modulus
by sonic technique, x-ray diffraction under transverse load, electrical
resistivity, and internal friction. Dynamic modulus of silicon carbide
was experimentally determined at 80°F and 2200°F. Modulus 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.

APPLICATION STUDIES

WADC TR 58-455, Part I, OTS Release

June 1959

SUBJECT: PRESSURE-SENSITIVE TAPE SUSPENSION SYSTEM FOR
AIRCRAFT PARTS IN SHIPPING CONTAINERS PART I.
Laboratory Tests of Suspension Systems

INVESTIGATOR: Arnold W. Voss

CONTRACT: PO (33-600) 53-4023, Forest Products Laboratory,
Forest Service, U. S. Department of Agriculture

ABSTRACT: 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 58-455, Part II, OTS Release

October 1959

SUBJECT: PRESSURE-SENSITIVE TAPE SUSPENSION SYSTEMS FOR
AIRCRAFT PARTS IN SHIPPING CONTAINERS PART II.
Effects of Accelerated Exposure on Tape
Properties

INVESTIGATOR: Arnold W. Voss

Contract: PO (33-600) 53-4023, Forest Products Laboratory,
Forest Service, U. S. Department of Agriculture

ABSTRACT: 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.

September 1959

SUBJECT: THE EFFECTS OF SIMULATED SPACE ENVIRONMENTS
ON CUSHIONING MATERIALS

INVESTIGATOR: David N. Keast, Jordan J. Baruch

CONTRACT: AF 33(616)-5887, Bolt Beranek and Newman, Inc.

ABSTRACT: Measurements of some of the properties of several types of package cushioning materials at temperatures of $+75^{\circ}\text{F}$, -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 measurements 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.

September 1959

SUBJECT: DEVELOPMENT OF LIGHTWEIGHT ALUMINUM CRATES FOR
BULKY LOW DENSITY ITEMS

INVESTIGATOR: Warren D. Hypes

ABSTRACT: 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.

February 1960

SUBJECT: MEASURING FIELD HANDLING AND TRANSPORTATION CON-
DITIONS

INVESTIGATOR: Capt Kenneth W. Bull, Materials Central
Dr. Carl F. Kossack, Purdue University

WADD TR 60-4, OTS Release (Continued)

ABSTRACT: 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 height 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.

ACOUSTIC MATERIALS AND STUDIES

WADC TR 58-460, Part I, OTS Release

June 1959

SUBJECT: 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

INVESTIGATOR: William E. Lawrie

CONTRACT: AF 33(616)-5060, Armour Research Foundation

ABSTRACT: 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.

WADC TR 58-460, Part II, OTS Release

June 1959

SUBJECT: DEVELOPMENT OF NEW SOUND ABSORBING MATERIALS FOR NOISE SUPPRESSORS PART II. Evaluation of Commercially Available Materials

INVESTIGATOR: William A. Lawrie

CONTRACT: AF 33(616)-5060, Armour Research Foundation

ABSTRACT: 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

WADC TR 58-460, Part II (Continued)

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.

WADC TR 59-70, OTS Release

June 1959

SUBJECT: BIBLIOGRAPHICAL REVIEW OF PANEL FLUTTER AND EFFECTS OF AERODYNAMIC NOISE
INVESTIGATOR: J. V. Rattayya, L. E. Goodman
CONTRACT: AF 33(616)-5426, University of Minnesota
ABSTRACT: 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 reference 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.

WADC TR 59-231, OTS Release

September 1959

SUBJECT: DEVELOPMENT OF A FATIGUE TESTING APPARATUS FOR TESTING HONEYCOMB SANDWICH STRUCTURAL PANELS IN SERVICE-SIMULATED SONIC FATIGUE
INVESTIGATOR: Edmond F. E. Zeijdel, William H. Ashely
CONTRACT: AF33(616)-5172, Midwest Research Institute
ABSTRACT: 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.

WADC TR 59-231 (Continued)

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, OTS Release

January 1960

SUBJECT: ROLE OF STRUCTURAL DAMPING IN ACOUSTICAL FATIGUE

INVESTIGATOR: W. J. Trapp, B. J. Lazan

ABSTRACT: 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.

II. APPENDIX, CONTRACTOR INDEX

	Page
Aeronca Manufacturing Corporation.	98
Alfred University, College of Ceramics	1
Allison Division, General Motors Corporation	64
Alloyd Research Corporation.	9
American Brake Shoe Company.	11
American Cyanamid Company.	60
American Machine and Foundry Company	46
American Potash and Chemical Corporation	69
Anderson Physical Laboratory	156
Arthur D. Little, Incorporated	115, 158
Armour Research Foundation	3, 8, 12, 16, 21, 22 47, 91, 110, 122, 142, 168
Automation Instruments, Incorporated	50
Avco Corporation	18, 48
Battelle Memorial Institute.	17, 20, 26, 149, 159
Bausch and Lomb Optical Company	142
Bell Aircraft Corporation.	4, 163, 164
B. F. Goodrich Company	87
Bjorksten Research Laboratories, Incorporated.	119
Boeing Airplane Company.	87, 88, 92
Bockamollan, Brosarps Station.	40
Bolt, Beranek and Newman, Incorporated	32, 43, 166
Brown University, Metals Research Laboratory	40
Brush Beryllium Company.	2
Bureau of Mines	120
California Research Corporation.	126
California, University of.	36
Carbon Research Laboratory, University of Buffalo.	7
Carborundum Company.	7
Case Institute of Technology	12
Chicago Midway Laboratories	149
Clevite Research Center.	128
Climax Molybdenum Company of Michigan	20
Columbia University.	40, 161
Commonwealth Engineering Company of Ohio	58
Connecticut Hard Rubber Company.	89
Consolidated Electrodynamics Corporation	155
Cook Research Laboratories	118
Coordinating Research Council, Incorporated.	127
Cornell Aeronautical Laboratories, Incorporated.	108, 115, 162
Crucible Steel Co. of America.	14, 15, 67
Cryogenic Engineering Laboratory	99

CONTRACTOR INDEX (Continued)

	Page
C. T. L., Incorporated	104
Curtiss-Wright Corporation.	37, 38
Dayton, University of	62, 63, 105, 111, 158
Denver Research Institute	74
Douglas Aircraft Company, Incorporated.	2
E. F. Houghton and Company.	130
E. I. du Pont de Nemours and Company, Incorporated	81
Engineering Supervision Company	28
Ethyl Corporation	70
Fabric Research Laboratories, Incorporated.	116
Firth Sterling, Incorporated.	1
Florida, University of.	82, 84
Forest Products Laboratory.	93, 99, 100, 102 104, 106, 109, 165
General Electric Company	76, 121, 151
Georgia Institute of Technology	55, 62, 65
Goodyear Aircraft Corporation	107, 113
Gulf Research and Development Company	133
Harris Research Laboratories, Incorporated	114
Hooker Chemical Corporation	68
Houze Glass Corporation	113
Illinois, University of	50, 54, 57, 71, 94, 159, 160
Indiana Steel Products Company.	152
Kaiser Aluminum and Chemical Corporation.	45
Ledoux and Company.	155
Linde Company	6
Manufacturing Laboratories, Incorporated.	13
Marquardt Aircraft Company.	56, 162
Massachusetts Institute of Technology	10, 82, 117, 138, 157
Materials Research Corporation.	28
Melpar, Incorporated.	80
Merck Sharp and Dohme Research Laboratories	79
Metallwerk Plansee.	25
Michigan, University of	17, 23, 38, 42, 43, 136

CONTRACTOR INDEX (Continued)

	Page
Midwest Research Institute	36, 45, 70, 73, 131, 134, 135, 138, 140, 169
Minnesota, University of	31, 32, 33, 35, 36, 44, 45, 169
Monsanto Chemical Company	77, 107, 108
Narmco Industries, Incorporated	96, 97, 98
National Bureau of Standards.	9, 21, 57, 77, 96, 101 150, 152, 160
New York University	27, 41, 58
North American Aviation, Incorporated	59
Olin Mathieson Chemical Corporation	124, 125
Pennsylvania State University	30
Petroleum Refining Laboratory	127
Products Research Laboratory	89
Purdue University	59
Quartermaster Research & Engineering Command, U.S. Army	118
Reaction Motors Division	61, 75
Royal Institute of Technology	34, 35
Scientific Oil Compounding Company.	119
Shell Development Company	120, 136
Southern Research Institute	90
Stanford Research Institute	5, 6, 50, 111, 132, 133
Sylvania-Corning Nuclear Corporation	47
Technische Hochschule, Munich, Germany	29
Thermodynamics Laboratories	100
Ultrasonic Testing and Research Laboratory	52
U. S. Borax Research Corporation.	85
Utah, University of	4
Western Gear Corporation.	129
Westinghouse Electric Corporation	23, 144
Wyandotte Chemicals Corporation	74, 78

III. APPENDIX, INVESTIGATOR INDEX

	Page
Abbott, Norman J.	116
Achbach, W. P.	158
Adams, Harold W.	129
Adams, William T.	7
Ahima, Franklin J.	115
Albrecht, W. M.	20
Allen, John M.	26
Allinikov, Sidney	112
Altstetter, C. J.	10
Anderson, George	40
Anderson, James C.	7
Anderson, R. C.	56
Anderson, R. P.	76
Anerson, Scott	156
Andrew, J. F.	7
Andrew, K. F.	23
Andrews, Charles R.	61
Arnold, J. S.	50
Ashley, William H.	169
Atlas, Leon M.	3, 8
Audrieth, Ludwig F.	71
Ault, Robert T.	41
Averbach, B. L.	10
Backer, S.	117
Bailar, John C., Jr.	71
Baker, J. S.	4
Baldrige, John H.	88
Baldwin, William M., Jr.	12
Barger, J. W.	135
Barish, Leo	116
Barr, Robert Q.	20
Barnum, E. R.	136
Baruch, Jordan J.	166
Basche, Malcolm	9
Bassett, William	98
Baum, George	123
Baun, William L.	155, 156
Bayer, Joseph	98
Beadle, Charles W.	115
Beal, J.	108
Beauchamp, E. K.	4
Beaver, W. W.	2
Bell, Norton W.	154

INVESTIGATOR INDEX (Continued)

	Page
Belser, Richard B.	62, 65
Benesovsky, F.	25
Benjamin, R. J.	118
Bennett, Dwight G.	50, 94
Benzing, Robert J.	138
Berg, Lawrence	28
Bergeron, Clifton G.	54, 56
Berlin, K. Darrell	84
Bernett, Eugene C.	162
Bicelli, L. Peraldo	27
Biedenkapp, Donald C.	104
Bieniek, Marciej P.	40
Binder, Ira	1
Blackburn, P. E.	23
Blankenship, Myra	156
Blewett, Guy C.	133
Blocker, E. W.	56
Boyd, R. W.	56
Bohner, George E.	74
Boller, K. H.	104, 106, 109
Bolze, C. C.	135
Bonin, John H.	149
Boon, J. L.	149
Boram, W. R.	96, 97
Bortz, S. A.	8
Bowen, Ronald K.	23
Bradford, V. E.	56
Bradstreet, S. W.	8
Breed, L. W.	70, 138
Brisbane, Alton W.	39
Broadley, Harry R.	139
Brossy, J. Frees	113
Brotherton, R. J.	85
Brown, G. A.	117
Brown, Henry C.	82
Brown, Kathleen	137
Brunier, Robert	122
Buckner, Dean A.	142
Bull, Kenneth	166
Bunshah, R. F.	27, 58
Butler, George B.	84

INVESTIGATOR INDEX (Continued)

	Page
Cash, Burna S.	119
Calish, S. R.	126
Callomon, Irma G.	96
Campbell, G. W.	85
Campbell, Ronald C.	11
Carwile, Nesbit L.	9
Caserio, Frederick F., Jr.	69
Chandhok, V. K.	14
Chang, C. C.	44
Chapin, Jay C.	119
Chappelow, Cecil C., Jr.	73
Charles, Michael P.	145
Cheorvas, William	89
Chick, Bruce	40
Chilton, J. Emery	5
Christian, John B.	123, 130
Chow, S. T.	44
Clark, Robert N.	73
Cohen, J. B.	48
Cohen, Morros	10
Cohen, Murray S.	75
Coldren, A. Phillip.	23
Coleman, Donald G.	102
Conner, J. H.	1, 57
Cornish, Rodney H.	115
Copan, T. P.	23
Cote, Maurice J.	147
Cooke, E. W.	59
Conti, Joseph C.	4
Coss, James R.	148
Couch, Dwight E.	57
Crain, Ronald D.	70
Cross, G. L.	52
Crossley, F. A.	16
Crugnola, Aldo M.	117
Cummings, Harold W.	37, 38
Dakin, F. W.	144
Dale, James W.	77
Das, Y. C.	33
DeBan, Harry G.	164
Deck, Eva P.	149
Deem, Herbert W.	149

INVESTIGATOR INDEX (Continued)

	Page
Dharmarjan, S.	160
Diller, C. C.	160
Divens, W. C.	144
Douglass, R. W.	17
Doyle, Charles D.	79
Drummond, W. Wendell	119
Dudak, Rita	75
Dulis, E. J.	14
Dunbar, D. J.	148
Durrell, William	75
Eichner, H. W.	93, 99, 100
Eimer, E. H.	160
Eismann, William J.	130
Emmons, Howard W.	7
Engel, Niels	65
Engel, Olive G.	101
Engelke, J. L.	6
English, W. D.	6
Farley, Eldon P.	5, 6
Fessett, Donald J.	129
Fatica, Nicholas	128
Favor, R. J.	159
Fenske, Merrell R.	127
Flieger, Howard W.	150
Forney, D. M., Jr.	161
Foster, Kenneth	115
Franken, Peter A.	32
Freeman, Levern D.	69
Freeman, James W.	17, 23, 38, 42
Freudenthal, Alfred	40, 161
Friedberg, Arthur L.	54, 56
Fristedt, B. E.	36
Frost, William M.	99
Fu, C. C.	35
Fugardi, Joseph	47
Furby, N. W.	126
Gaj, B. J.	68
Gal, George	79
Garber, John D.	79
Gerald, George	41

INVESTIGATOR INDEX (Continued)

	Page
Giancola, J. R.	33
Gibbs, P.	4
Gilman, H.	68
Ginnings, Defoe C.	150
Glekas, Louis P.	80
Gluck, Jeremy V.	17, 38, 42
Goetzel, C. G.	27, 58
Goldsmith, H.	85
Gollis, Morton H.	107, 108
Gouinlock, V.	68
Goodman, L. E.	33, 169
Goss, B. R.	1
Grafstein, Daniel	75
Graft, W. H.	16
Griffith, John Q.	134
Grosskreutz, J. C.	36
Grover, H. J.	159
Gubser, J. L.	160
Guertler, W.	26
Gulbransen, E. A.	23
Gurev, Harold S.	12
Gutfreund, K.	110
Haertling, Gene H.	94
Hafner, Harold C.	142
Hagerman, Edward	83
Haggerty, William J., Jr.	70
Hahn, G. T.	13
Halagan, D. R.	120
Holden, F. A.	6
Hale, Denver	145
Hallse, Robert L.	54
Harp, James L.	26
Hankinson, Frank E.	62
Hansen, C. E.	100
Harmon, Dale J.	87
Harris, Edward E.	124
Harris, J. N.	55
Harrison, W. N.	152
Harvester, V. G.	36
Hauck, C. A.	56
Heller, Robert A.	161

INVESTIGATOR INDEX (Continued)

	Page
Hemmig, Phillip B.	147, 148
Hendricks, Paul L.	13
Hensen, C. E.	135
Hensler, J. Raymond.	142
Herman, Harry.	28
Hickman, William R.	87, 88
Hickmott, R. L.	146, 147
Hiler, Malvern, J.	58
Hirt, Robert C.	60
Hogle, D. H.	144
Holden, F. C.	17, 20
Holmes, Roy F.	100
Hopkins, Vernice	131, 134
Hoskyns, W. R.	1
Houston, Charles D.	154
Hruz, Frank M.	5
Hurd, Donald E.	100
Hypes, Warren D.	166
Iannucci, A.	28
Ingham, Francis H.	89
Intrater, J.	28
Isaac, Raymond	156
Islinger, J. S.	85
Ives, Joseph S., Jr.	46
Jaffee, R. I.	17, 20
Janis, Edward C.	96, 97
Jenkin, William C.	58
Johns, Iral B.	77
Johnson, D. R.	146, 147, 148
Jones, Robert E.	79
Juul, N.	7
Kalfayan, Sarkis H.	89
Kallmann, Silve	155
Kappelt, George F.	163
Kasak, A.	14
Kan, Peter T.	74
Keast, David N.	166
Keller, Richard L.	103
Kelly, John J.	5
Kerlin, W. W.	136

INVESTIGATOR INDEX (Continued)

	Page
Kerper, M. J.	160
Kerwin, Edward M., Jr.	43
Keyser, Lois A.	154
Kieffer, R.	25
Kiffer, A. D.	6
Kimball, Kenneth E.	106
Kindley, Lee M.	80
King, Lowell A.	146, 147, 163
Klaus, Erwin E.	127
Kleint, R. E.	52
Klinger, R. F.	39
Klopp, W. D.	20
Knorr, C. A.	29
Knutson, David	68
Koenig, Paul B.	70
Kogler, Hubert P.	82
Kolano, John S.	68
Komjathy, S. A.	21
Kossack, Carl F.	166
Kreder, K.	13
Kreidl, Norbert J.	142
Kritzer, S.	59
Krizik, J. J.	69
Kuchta, J. M.	120
Kumpitsch, Robert C.	121
Lambert, Robert F.	31, 45
Lambiris, S.	120
Larson, Hugo R.	11
Latos, Edwin J.	3
Lauchner, Julian H.	50
Lauer, Vernon A.	135
Lavik, Melvin T.	140
Lawrie, William E.	168
Lazan, B. J.	170
Leasure, J. D.	160
Lefort, Henry G.	94
Lehrer, W. M.	47
Lement, B. S.	13
LePara, A. A.	134
Letter, Eugene C.	142
Levy, A. V.	56
Lewis, D. W.	144

INVESTIGATOR INDEX (Continued)

	Page
Liu, Robert	155
Lloyd, Herbert W.	11
Loetz, Merle R.	50
Long, Roger A.	98
Lord, Richard C.	157
Lovelace, Alan M.	83
Lucks, Charles F.	149
Lukish, Joseph L.	9
Lund, H. H.	8
Lundgren, T.	44
Lusebrink, T. R.	120
Lyon, Richard H.	32
Madorsky, Samuel L.	77
Maguire, R. G.	110
Mahler, William	64
Mahoney, C. L.	136
Mallard, Paul A.	89
Margo, George	107, 113
Marvel, Carl S.	71
Manasevit, H. M.	85
Mangsen, Gustave E.	7
Marin, Joseph	30
Marolo, Samuel A.	101
Maroney, D. J.	56
Marshall, Richard F.	80
Marrs, O. L.	68
Marynowski, C. V.	111
Matacek, George F.	81
McArdle, Jordan D.	20
McBride, Richard J.	105, 111
McCarthy, Paul R.	133
McCloskey, A. L.	85
McCready, D. W.	136
McDevitt, Neil T.	156
McDonald, Patrick H.	140
McElhill, Elizabeth A.	77
McGrath, Joseph J.	133
McHugh, Kenneth L.	137
McKown, R. D.	52
Medved, T. M.	135
Megas, Constantine J.	117
Mellen, D. M.	117

INVESTIGATOR INDEX (Continued)

	Page
Melgren, Arne	34, 35
Menkart, John	114
Mentel, T. J.	35
Merlin, A.	23
Metcalfe, A. G.	16
Miles, M. H.	78
Mixer, R. Y.	111
Moore, George A.	21
Morgan, George L.	50
Morriss, F. V.	138
Moskowitz, A.	67
Mrozowski, S.	7
Murray, G.	28
Murray, S. Frank	28, 139
Nakamura, H. H.	8
Newsom, H. C.	85
Nixon, A. C.	120
Norman, Robert S.	139
Nowak, J. M.	4, 164
Nudelman, Harvey B.	12
Oberthin, Hans	155
O'Connell, John J.	107, 108
Ogden, H. R.	7
Olson, O. H.	110
Paciorek, Kay L.	78
Paine, R. M.	2
Parikh, Kanaiejalal W.	94
Papirno, Ralph	41
Parkins, John A.	81
Passmore, E. M.	18
Patterson, William A.	98
Paul, D. A.	45
Pawel, Richard E.	154
Pearce, Willard J.	151
Pearl, H. A.	4, 163, 164
Perkins, George D.	154
Perlee, H. E.	120
Peterson, George P.	101
Petterson, L. L.	85
Philip, T. V.	151

INVESTIGATOR INDEX (Continued)

	Page
Picker, Amos	15
Picklesimer, Lewellyn G.	116
Platt, Milton M.	116
Pino, M. A.	126
Plyler, E. K.	152
Poncelet, Eugene F.	5
Posakony, Gerald J.	50
Price, Channon F.	149
Provance, J. D.	113
Pulliam, George R.	2
Rabinowicz, Ernest	138
Raeuber, Arthur E.	90
Rambauske, Werner	158
Rang, Edward R.	35
Rattayya, J. V.	169
Read, R. H.	21
Redmerski, L.	67
Reimschuessel, Herbert K.	83, 156
Reinhart, Frank W.	96
Richmond, J. C.	152
Riel, F. J.	96, 97
Ritt, Paul E.	27
Rizzo, Frank J.	117
Robinson, A. R.	119
Robinson, Charles F.	154
Robinson, Samuel A.	33, 79
Roller, David	58
Rollins, Fred R.	36, 45
Rondeau, R. E.	148
Roper, Robert S.	87, 88
Rosenberg, Samuel J.	9
Rosenberg, Joseph W.	63
Ross, Donald	43
Rostaker, W.	21, 22
Saari, W. S.	136
Sabanas, M.	91
Saulino, Frank A.	7
Sawyer, Arthur W.	124, 125
Sax, K. J.	136
Schaaf, Robert L.	74
Schetky, Laurence M.	9

INVESTIGATOR INDEX (Continued)

	Page
Schmidt, Donald L.	110, 111
Schmidt, F. F.	20
Schmidt, Josef J. E.	74
Schmitt, Richard G.	60
Schulte, William C.	37
Schultz, Charles E.	7
Schwartz, Albert A.	139
Schwartzbart, H.	47
Schwarzlose, Paul F.	54
Schwelke, G.	68
Seegman, Irvin P.	89
Semchyshen, M.	20
Seshadri, C. V.	117
Sethna, P. R.	36, 44
Seyferth, Dietmar	82
Sheehan, John P.	12
Sidebottom, O. M.	160
Silverstein, R. M.	132, 133
Skinner, David L.	84
Sklarew, S.	56
Skramstad, H. K.	152
Smiley, William D.	5
Smith, John	77, 137
Smith, Robert K.	130
Smithson, Lee D.	157
Sobon, Leon E.	5
Sonnenschein, George	150
Spain, Raymond G.	78
Spacil, Henry S.	9
Spring, M. M.	76
Stackman, Robert W.	84
Stair, R.	152
Stein, Gustav A.	79
Steven, G.	15
Stevens, G. H.	104
St. John, Andrew D.	131
Stonehouse, A. James	2
Straus, Sidney	77
Strauss, H. E.	7
Sterbutzel, G. A.	108
Stulen, Foster B.	37
Susman, S. E.	96, 97, 142
Swica, J. J.	1

INVESTIGATOR INDEX (Continued)

Page

Taylor, Donald E.	149
Thompson, James T.	154, 157
Thompson, Ron E.	89
Thompson, R. H.	157
Tenzer, Rudolph K.	152
Tierney, P. A.	144
Tinklepaugh, J. R.	1
Tipton, Frank W.	92
Trapp, W. J.	170
Trepas, George E.	87, 88
Trepka, W.	68
Trop, Donald C.	135
Truell, John	40
Ungar, Eric E.	43
Urode, R. J.	4
Varner, R. Loren	89
Vassalo, Franklin A.	108
Venkalesan, P. S.	58
Verchot, Edgar A.	90
Vincent, C. T.	50
Vonnegut, George L.	64
Vreeland, J. H.	157
Voorhees, Howard R.	42
Voss, Arnold W.	165
Wagner, Ross I.	69
Wahl, N. E.	108
Walton, J. D.	55
Walwood, John T.	117
Wang, Douglas Y.	45, 161
Weibull, Waloddi	40
Weik, Helmut	25
Weinig, S.	28
Wenzel, R. N.	144
Wepfer, Gordan G.	146, 147
Wescott, Emery N.	137
Whang, Y. C.	44
White, Jerry E.	23
Whittier, James S.	31
Wiederhorn, N. M.	157
Wier, John E.	68

INVESTIGATOR INDEX (Continued)

	Page
Wilcox, David L.	54
Wilgus, D. R.	100
Williams, R. B.	118
Wilmers, F. J.	45
Winn, Robert A.	150
Wobschall, D. C.	7
Wood, Webster	149
Woods, W. G.	149
Yeager, Charles C.	119
Yerkovich, Luke A.	162
Zabetakis, M. G.	120
Zambrow, John L.	47
Zeijdel, Edmond F. E.	169

DIRECTORATE OF MATERIALS & PROCESSES, AERONAUTICAL SYSTEMS DIVISION, Wright-Patterson Air Force Base, Ohio, A REVIEW OF THE AIR FORCE MATERIALS RESEARCH AND DEVELOPMENT PROGRAM, by Donna J. Tate, June 1961. 186p. (WADC TR 53-373 Supplement 7) Unclassified report These reports cover basic and applied research in the materials area being conducted by the Metals and Ceramics, Non-Metallic Materials, Physics and Applications Laboratories of the Materials Central.	UNCLASSIFIED
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