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REIC Report No. 34
June 30, 1964

# RADIATION-EFFECTS STATE OF THE ART
# 1963-1964

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RADIATION EFFECTS INFORMATION CENTER
Battelle Memorial Institute
Columbus 1, Ohio

The Radiation Effects Information Center has been established at Battelle Memorial Institute by the United States Air Force to provide a means of placing radiation-effects data in the hands of designers and those conducting research and development. Access to the Center and to its reports is obtained through the Air Force. This report has been prepared pursuant to the provisions of Contract No. AF 33(615)-1124 [continuation of AF 33(657-10085], BPSN Nos. 64-6899-7381 738103 and 64-6399-7634.

REIC Report No. 34
June 30, 1964

Report on

# RADIATION-EFFECTS STATE OF THE ART

# 1963-1964

to

AIR FORCE SYSTEMS COMMAND
RESEARCH AND TECHNOLOGY DIVISION

RADIATION EFFECTS INFORMATION CENTER
Battelle Memorial Institute
Columbus, Ohio 43201

# TABLE OF CONTENTS

## APPENDIX I

## APPENDIX II

Note: This report was prepared by Battelle Memorial Institute under Contract No. AF 33(615)-1124 [continuation of AF 33(657)-10085] for use of the Air Force. The following Battelle staff members contributed to this report: D. J. Hamman, E. N. Wyler, R. K. Thatcher, W. H. Veazie, Jr., F. R. Shober, N. J. Broadway, B. P. Fairand, L. H. Bettenhausen, Mrs. H. C. Gillette, and Mrs. L. M. Nelson.

# Report on
# Radiation Effects State of the Art
# 1963-1964

## SUMMARY

### Electronic Components and Equipment

During the 1963-1964 interval, reports available at the REIC indicate that radiation-effects studies in the transient- and space-radiation environments are growing rapidly (see the Programs in Progress portion of the electronic section).

A very large portion of the electronics work is in the semiconductor field, as this constitutes the most sensitive area outside of organic insulation.

The best summary of the radiation-effects state of the art in electronics can be found in reviewing the various bar graphs of the electronics section, although Figures A and B provide a capsule digest.

More work is needed in the area of correlation of damage from various radiation forms and also in the transient area; in both cases the concentration should be on providing higher grade data.

### Polymeric Materials

The major emphasis in current radiation studies continues to be in the areas of space environment, that is, combined environments of radiation, extreme temperatures, and vacuum. In general, materials having a high degree of cure, high molecular weight, good heat resistance, and little or no additive, such as a plasticizer, show promise for use in space environment. In many cases, the radiation resistance of a material is improved in vacuum because of the lack of oxygen, which is generally the major factor contributing to polymer deterioration.

Polyimide and phosphonitrillic chloride polymers have been reported as having improved radiation stability over presently used polymers and merit consideration for application in a radiation environment provided suitable components can be fabricated. O-rings manufactured from rubber compositions containing antirads have shown improved service life in a radiation environment. This O-ring, although substantially improved, still does not meet the requirements for many applications. Several structural adhesives and laminates have been found to be satisfactory in radiation exposures at cryogenic temperatures. These include polyurethane, epoxy and modified epoxy, phenolic, and polyester materials.

Continued work is needed in fundamental studies directed toward new and improved polymers having greater resistance to radiation damage. Also, damage mechanisms

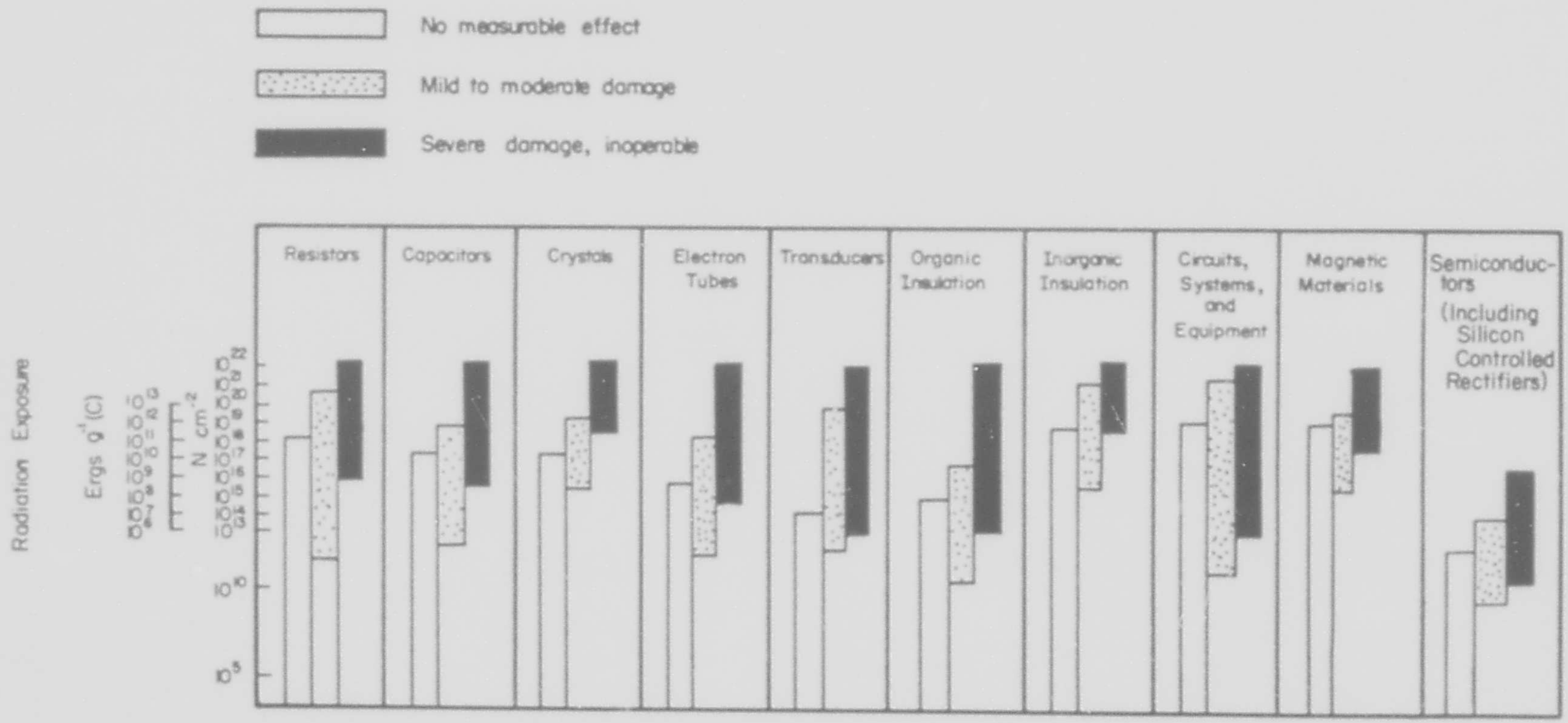

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FIGURE A. STATE-OF-THE-ART ASSESSMENT FOR ELECTRONIC PARTS AND SYSTEMS FROM A GENERIC STANDPOINT AS TO CAPABILITIES FOR RESISTING NUCLEAR-RADIATION DAMAGE

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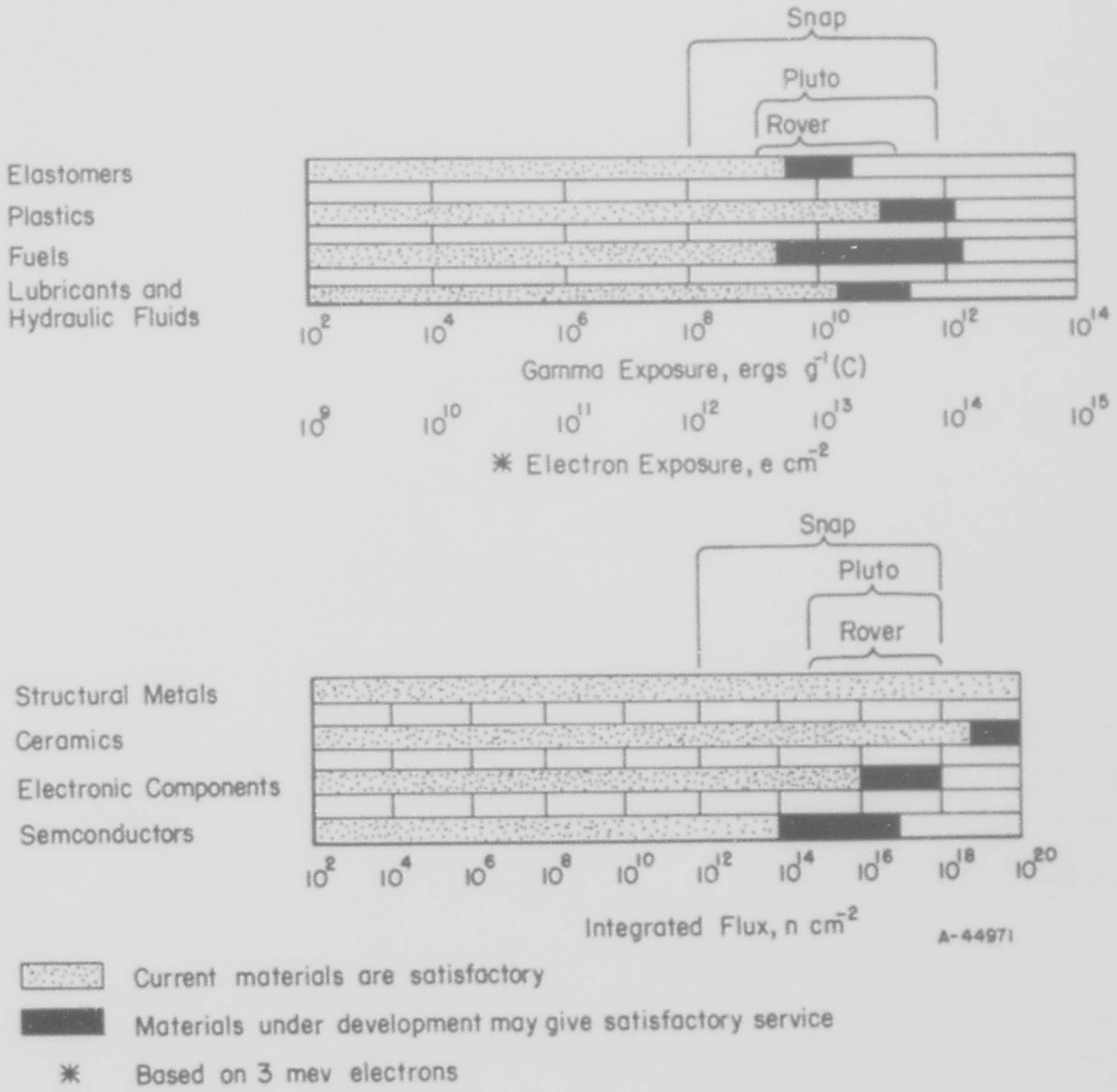

FIGURE B. STATUS OF MATERIALS IN RELATION TO RADIATION ENVIRONMENTS should be determined. More information is needed concerning the effect of high-energy impulse on polymers and the effect of exposure rate on various components. In addition, more data are needed regarding the amount of damage which may be sustained by elastomeric and plastic materials before failure occurs in the operation of the fabricated component.

## Lubricants, Flotation and Hydraulic Fluids

There was very little work in this area during the past year, and very few programs are in progress. Recent inquiries at the REIC indicate that there may be some increase in this area. This is particularly true in the space-radiation area.

Solid-film lubricants and hexafluorobenzene appear promising, although the solid-films are not usable in some cases and the current cost of hexafluorobenzene may be a deterrent.

## Ceramics

The rate of effort in the ceramics area was lower than for the previous year, and the number of current contracts indicates a further decline.

More information is needed on combined environmental effects, particularly in the space areas of temperature, vacuum, ultraviolet, protons, and electrons.

## Space-Radiation Environment

This section reports on more up-to-date measurements of the trapped radiation environments including the data from Explorers 15 and 18, Injun 3, and some Soviet data.

## Dosimetry

Some of the newer dosimetry practices are discussed. Atomic-displacement techniques and activation procedures are usual for neutron dosimetry, while chemical effects are discussed for gamma fields.

Recommendations for reporting procedures are also given.

## Structural Metals and Alloys

The radiation-induced property changes in structural metals and alloys continue to be of importance to reactor designers and operators. The summarization of data in this section reflects the increased use of structural materials in high-temperature applications.

This section also discusses the mechanism of radiation-induced property changes in ferritic and austenitic stainless steel, and high-temperature alloys.

## General

Figure B is a crude approximation of where we stand with respect to materials development for use in various radiation environments. The bracketed areas give some estimate of the radiation levels to be expected from various systems.

# INTRODUCTION

This is the seventh state-of-the-art summary of radiation-effects activities prepared by the Radiation Effects Information Center for the Air Force and NASA. The purpose of this report, based upon the data processed by the REIC, is to summarize progress and review problem areas in radiation-effects activities. This review should be helpful to those responsible for planning and executing radiation-effects programs. It is a revision and updating of the 1962-1963 summary, based on new information obtained during the past year.

This report summarizes briefly (1) the current state of the art, (2) recent results of radiation-effects studies, (3) programs in progress, and (4) Battelle's conclusions and recommendations in the following areas:

(A) Electronic Components and Equipment (Including Semiconductors)

(B) Polymeric Materials

(C) Lubricants, Flotation and Hydraulic Fluids

(D) Ceramics

(E) Space-Radiation Environment

(F) Dosimetry

(G) Structural Metals and Alloys.

This report is oriented around steady-state nuclear, pulse nuclear, and space-radiation environments.

Information on reactor-core materials, propulsion systems, shielding, and biological effects is not included in this report.

Appendix I summarizes the operation of the Radiation Effects Information Center from June 1, 1963, through May 31, 1964.

Appendix II lists the available reports and memoranda issued by the REIC.

# SECTION A

## ELECTRONIC COMPONENTS AND EQUIPMENT (INCLUDING SEMICONDUCTORS)

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# ELECTRONIC COMPONENTS AND EQUIPMENT (INCLUDING SEMICONDUCTORS)

## Current State of the Art

There was an increase in the amount of material received during the past year over that received during the previous report period. This increase, particularly apparent in semiconductor research, is possibly due to the need for radiation-effects information as applied to space applications.

The steady-state information for the 1963 period seems to follow a trend in which the sensitive components are being investigated as individual parts while the more-radiation-resistant components are being tested only in circuits. The pulsed-radiation studies seem to be devoted to obtaining data and explaining the effects of radiation on some of the odd components such as Shockley diodes, field-effect transistors, etc. A possible reason is that theories explaining the effects of pulsed radiation on the more common components have already been accepted. As far as space-radiation studies are concerned, semiconductors (particularly solar cells) are a big problem.

In view of the large amount of information received this year and last, it was decided to revise and rewrite REIC Report No. 18. This report is entitled "The Effects of Nuclear Radiation on Electronic Components". The rewritten report will contain a detailed discussion of radiation effects on component parts and will include a semiconductor section. The new report should be published before October of this year. As a result, this section of the state-of-the-art report will not contain any detailed discussions, but will be a brief updating of information.

## Resistors

The early experiments conducted to determine resistor susceptibility to radiation damage produced sufficient data to allow the electronics engineers and circuit designers to use these parts with some confidence. At least their concern over the probability of failure is much less than it is for the more-radiation-sensitive components.

During 1963 few reports were received that contained resistor data. The information that did come in was either the result of a manufacturer's inquiry as to susceptibility of his particular parts or the result of system studies in which it was desired to reassure the designer that the specified resistors would not exceed the criterion of failure.

In general the state of the art for resistors has not changed pronouncedly during the last year. Figure A-1 is an illustration of the state of the art on resistors. It should be remembered that many factors influence the susceptibility of the resistors to radiation damage. Much of the broadness indicated for each category of damage is caused by variation in insulation materials, structural design, body enclosure, and winding schemes. One of the most critical characteristics that appears to show a direct relationship to radiation is the resistance of the element. Usually, the element with the highest value of resistance for a group of units of one type from one manufacturer is the most susceptible of that group. This is due mainly to the higher ratio of conductive particles to organic binder materials for carbon-composition resistors; extremely thin films in carbon- and metal-film resistors; and small-diameter wires for wirewound resistors and potentiometers. Hence, only nominal values that were less than their respective critical values were used for the estimates in Figure A-1.

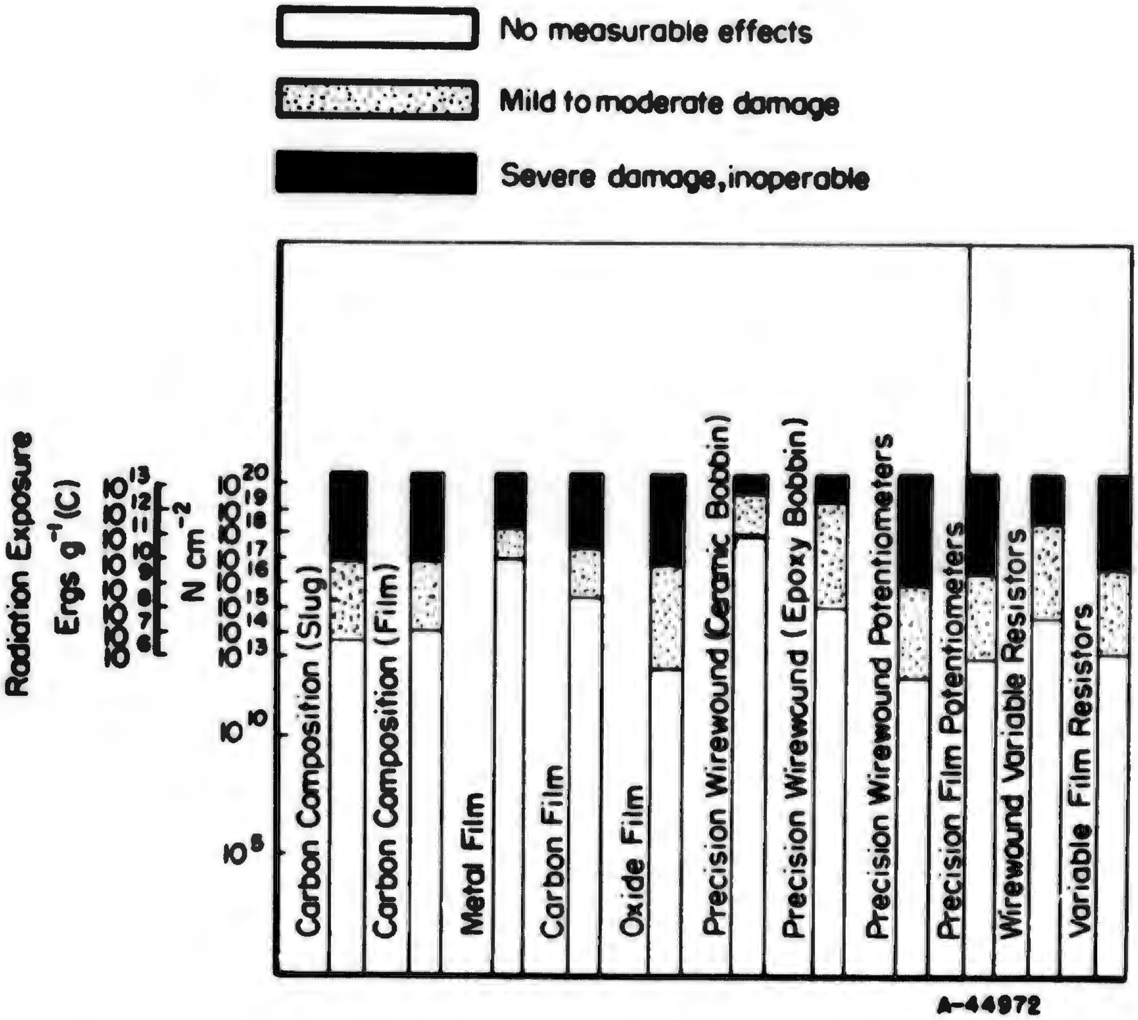

FIGURE A-1. STATE-OF-THE-ART APPRAISAL OF COMMONLY USED RESISTIVE ELEMENTS WITH RESPECT TO ESTIMATED PERFORMANCE IN A RADIATION ENVIRONMENT

All the types of resistors presented in Figure A-1 show thresholds of radiation damage between $10^{12}$ and $10^{16}$ n cm$^{-2}$ total fast-neutron exposure and from $10^6$ to roughly $10^{10}$ ergs g$^{-1}$ (C) of total gamma exposure. Severe damage can be expected for resistors of this group between $10^{15}$ and $10^{19}$ n cm$^{-2}$ total fast-neutron exposure and $10^8$ and $10^{12}$ ergs g$^{-1}$ (C) total gamma exposure. Although it may seem that resistors can be selected for most levels of radiation, this is not always an easy task. One reason is that not all types of resistors cover all ranges of resistance. Hence it may happen that, to get the value of resistance necessary, the designer will have to select a more radiation-sensitive type of resistor. Another area which needs studying is the damage mechanism(s) in resistors. Hopefully, studies of this sort would lead to a good empirical prediction for radiation damage.

## Capacitors

Capacitors in general show the same trend as the resistors in that some of the types are well explored from the radiation-exposure standpoint. The capacitors differ, however, in that there is only a slight overlap in nominal values. This offers the designer only a limited range for selection.

Capacitor data obtained from steady-state and space-radiation studies were definitely lacking during this period. The information which did come in was usually the result of a circuit or system study. Pulsed-radiation reports, however, contained a large majority of the capacitor information. This information covered practically all types equally and reported both individual and system capacitor studies.

In conclusion, the present state of the art for capacitors is illustrated by the bar graph of Figure A-2. Moderate changes can be expected for from $10^{12}$ to $10^{18}$ n cm$^{-2}$ fast-neutron exposure depending on capacitor type. Severe damage can be expected for various types between $10^{14}$ and $10^{19}$ n cm$^{-2}$.

## Piezoelectric and Quartz Crystals

During this survey period only a small amount of information has been received on crystals, and most of it is concerned with a system or a circuit. One investigation, however, studied the effects of various types of radiation on lead zirconium titanate and barium titanate. The data were erratic, but did indicate that the crystals may be useful in relatively high levels of radiation (but not above 1 x $10^{18}$ n cm$^{-2}$).

This area of study still lacks much information on radiation effects and damage mechanisms. A good comparison study of many individual crystals is needed.

## Electron Tubes

It has been found in the past that the primary damage mechanism which limits the life of electron tubes in high-intensity radiation environments continues to be the damaging effects to the glass envelopes and glass-to-metal seals. Recently the use of low-boron-content glass has greatly increased the life of tubes. A problem remains,

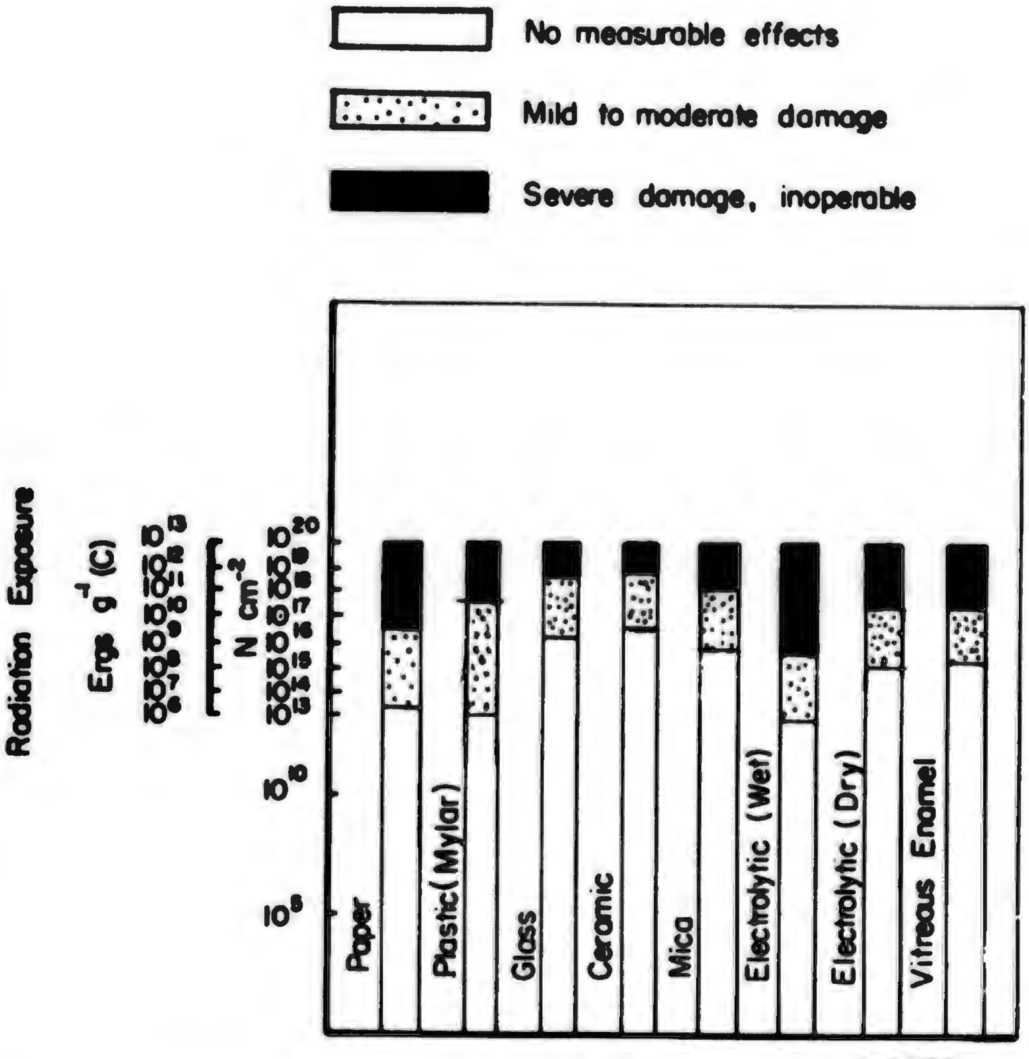

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FIGURE A-2. STATE-OF-THE-ART ASSESSMENT OF COMMONLY USED FIXED CAPACITORS WITH RESPECT TO ESTIMATED PERFORMANCE IN A RADIATION ENVIRONMENT however, because irradiated components are still susceptible to shock and vibration. Several companies have been trying to solve these problems. The General Electric Company has been the most successful with its TIMM tubes that function well up to $10^{18}$ n cm$^{-2}$ fast neutrons.

Figure A-3 indicates the sensitivity of various types of tubes to a radiation environment. It is possible the state of the art for electron tubes will be showing some interesting advances in the future. The radiation environment aboard a nuclear-powered spacecraft coupled with the radiation vulnerability of semiconductor devices may promote the use of vacuum tubes in such vehicles.

## Transducers

The information received during the report period on transducers shows that this area still is lacking data. It is evident from some of the developments, however, that work is being done in this area and that the technology is progressing rapidly. This growth can largely be attributed to various aeronautical and space programs that have emphasized the extreme need for transducers capable of withstanding damage when exposed to elements of nuclear and high-temperature environments. It should therefore be only a short time before the area of transducer information begins to fill out.

The new information indicates that both pressure and displacement transducers have functioned with only small deviation up to a fast-neutron exposure of $10^{17}$ n cm$^{-2}$. One device was a Colvin pressure transducer, and the other was a constantan foil strain gage.

In order to offer some aid for the selection of transducers for a nuclear environment, Figures A-4 and A-5 are presented. Some of the work is the result of appraisals of radiation-damage thresholds while the rest is the result of experimentation.

## Magnetic Materials

The amount of information received on magnetic materials during the past year was about the same as in 1962. In looking over this information it is seen that, from a technological view, the thresholds of permanent damage due to steady-state nuclear radiation has been established for most types of soft and hard magnetic materials. Permanent effects as a result of bomb burst on some typical soft materials also have been covered. Very little, however, has been reported to date on transient effects resulting from pulsed neutron radiation. Also, information concerning charged-particle effects on magnetic properties is lacking.

Magnetic materials which have been incorporated into devices comprise one of the more radiation-resistant electronic components. Magnetic materials show no change in the important characteristics when exposed to radiation fluxes in the range $10^{15}$ to $10^{20}$ n cm$^{-2}$ fast neutrons. They have also been shown to withstand pulsed gamma radiation to a total exposure of $10^9$ ergs g$^{-1}$ (C).

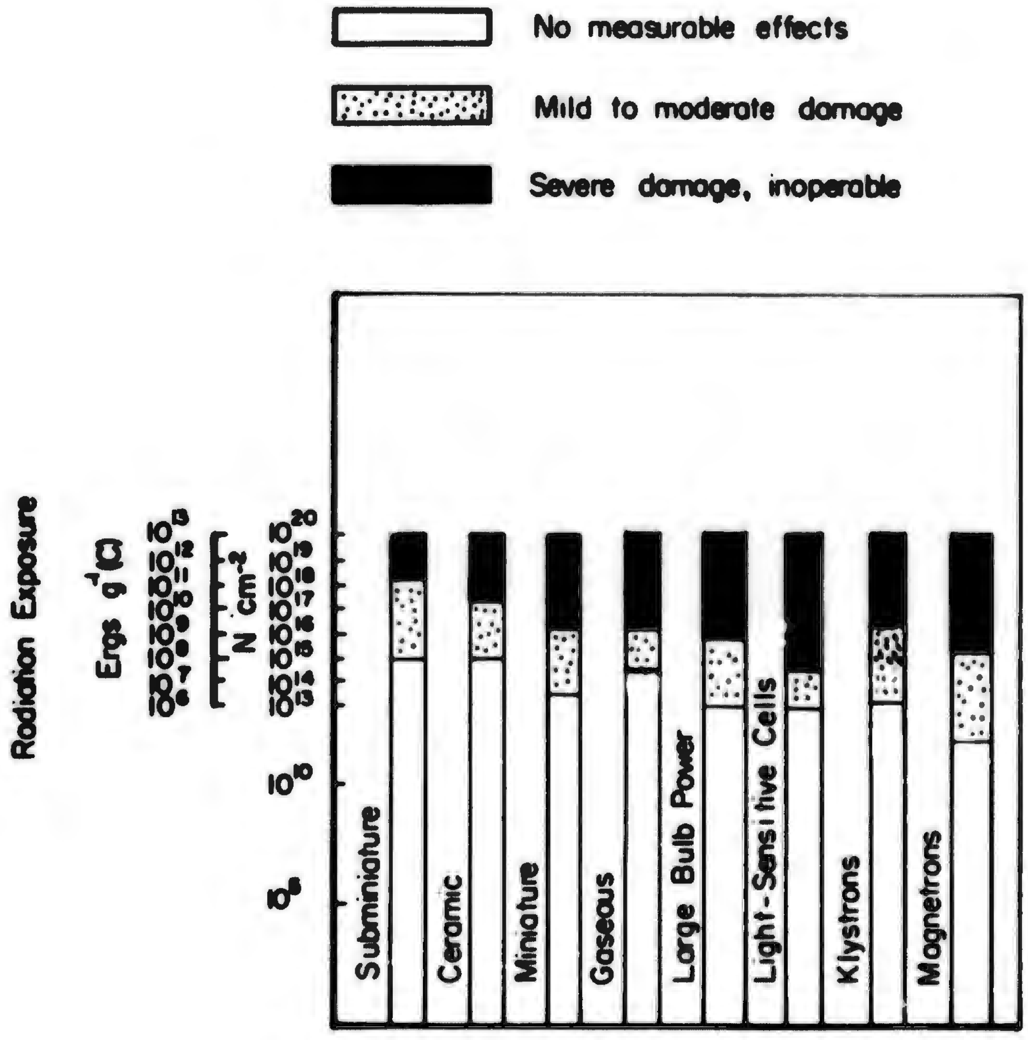

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FIGURE A-3. STATE-OF-THE-ART ASSESSMENT OF RADIATION-DAMAGE RESISTANCE FOR VARIOUS ELECTRON TUBES

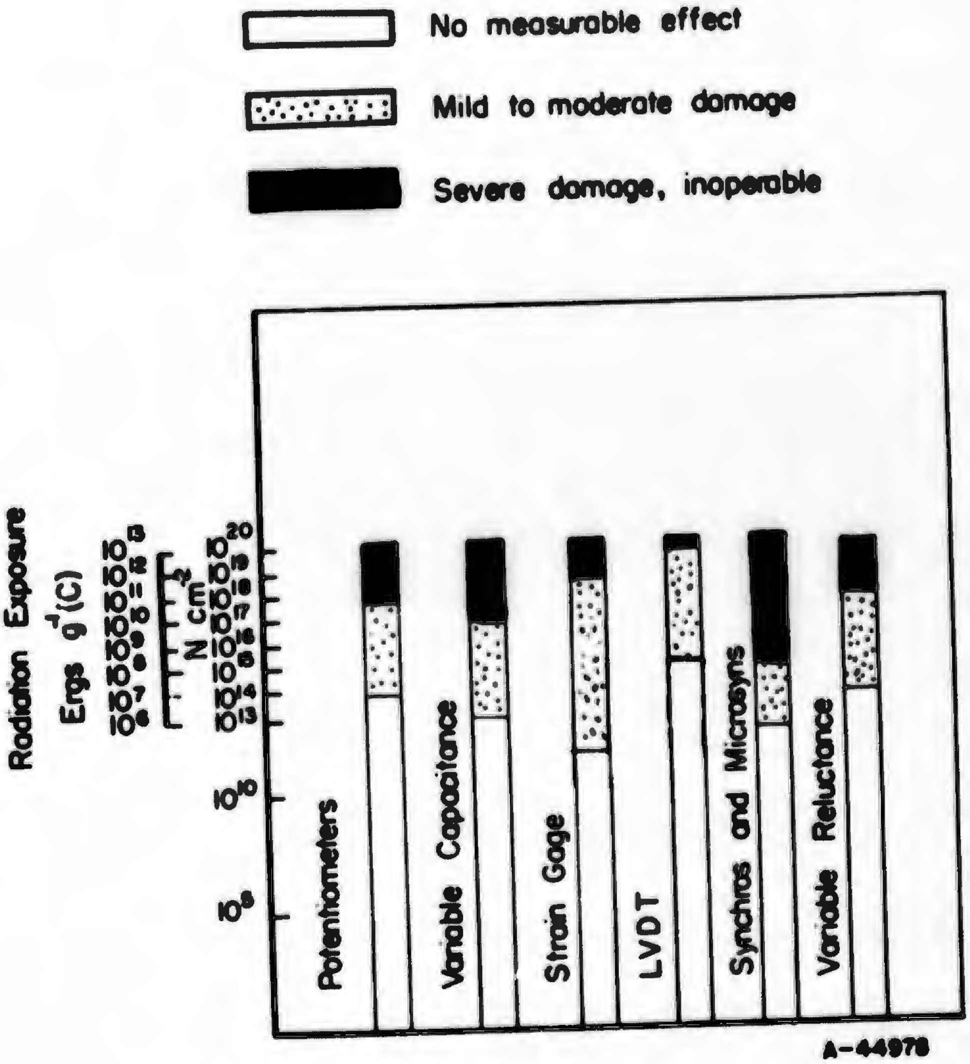

FIGURE A-4. ESTIMATED EFFECTS OF RADIATION ON DISPLACEMENT TRANSDUCERS AS A FUNCTION OF FAST-NEUTRON AND GAMMA RADIATION EXPOSURE

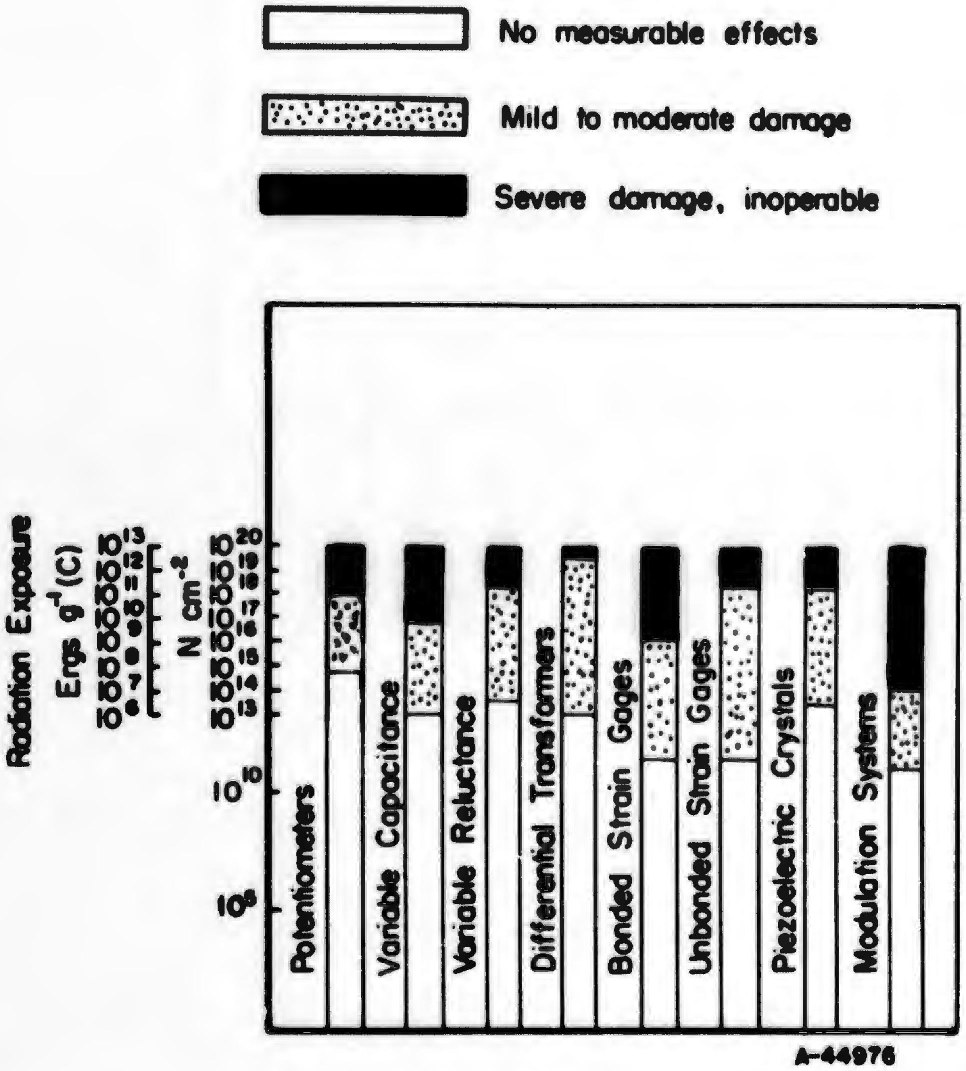

FIGURE A-5. ESTIMATED EFFECTS OF RADIATION ON PRESSURE TRANSDUCERS AS A FUNCTION OF FAST-NEUTRON AND GAMMA RADIATION EXPOSURE

## Electrical Insulation

Electrical insulation has presented definite problems since the beginning of radiation studies. Much of the knowledge now available has come about from the preparation of measurement circuits for basic components being irradiated, although in the past 2 years much more information has been coming in as a result of radiation studies of particular insulations which might be used in systems.

Pulsed radiation has probably caused the more difficult problems due to the transient effects induced in the insulation. The permanent effects can generally be avoided by the proper selection of insulation. There are insulations which function well up to fast-neutron exposures of $10^{17}$ n $cm^{-2}$.

Figures A-6 and A-7 show the radiation damage thresholds for various common types of organic and inorganic insulation. From these figures it is seen that more investigation is needed in order to improve the radiation resistance of the organic insulations.

## Semiconductors

The area of semiconductor investigation has produced a large amount of information during the past year. There is, however, one difficulty with this work. The majority of the information is on tests conducted to find the threshold of particular types of devices. The studies are not actually advancing the state of the art, but only adding to the number of different types of units tested.

It has been known for some time that nuclear radiation causes p-n junction devices to degrade. Diodes show decreases in forward voltage with a constant voltage across the junction. Also, the reverse leakage current and reverse breakdown voltage show increases. These are results of radiation-induced defects and decreases in minority-carrier lifetimes. In solar cells these changes show up as a decrease in output. In transistors the changes are manifested as an increase in the saturation current, $I_{CBO}$, and a reduction in common-emitter current gain, $\beta$.

The bar chart in Figure A-8 illustrates the thresholds of some of the common semiconductor devices. As inferred above, the area of radiation effects on semiconductors needs basic studies to advance the state of the art, particularly from the device standpoint.

## Circuits and Systems

Electronics engineers and circuit designers have developed several successful methods directed toward obtaining radiation-resistant circuits. One method used particularly with pulsed radiation is programming a computer with component equations and then having the computer perform a circuit analysis for each level of radiation. A method most popular for steady-state circuitry is to design the circuit with radiation-hardened components and/or components which have been shown to withstand the specified

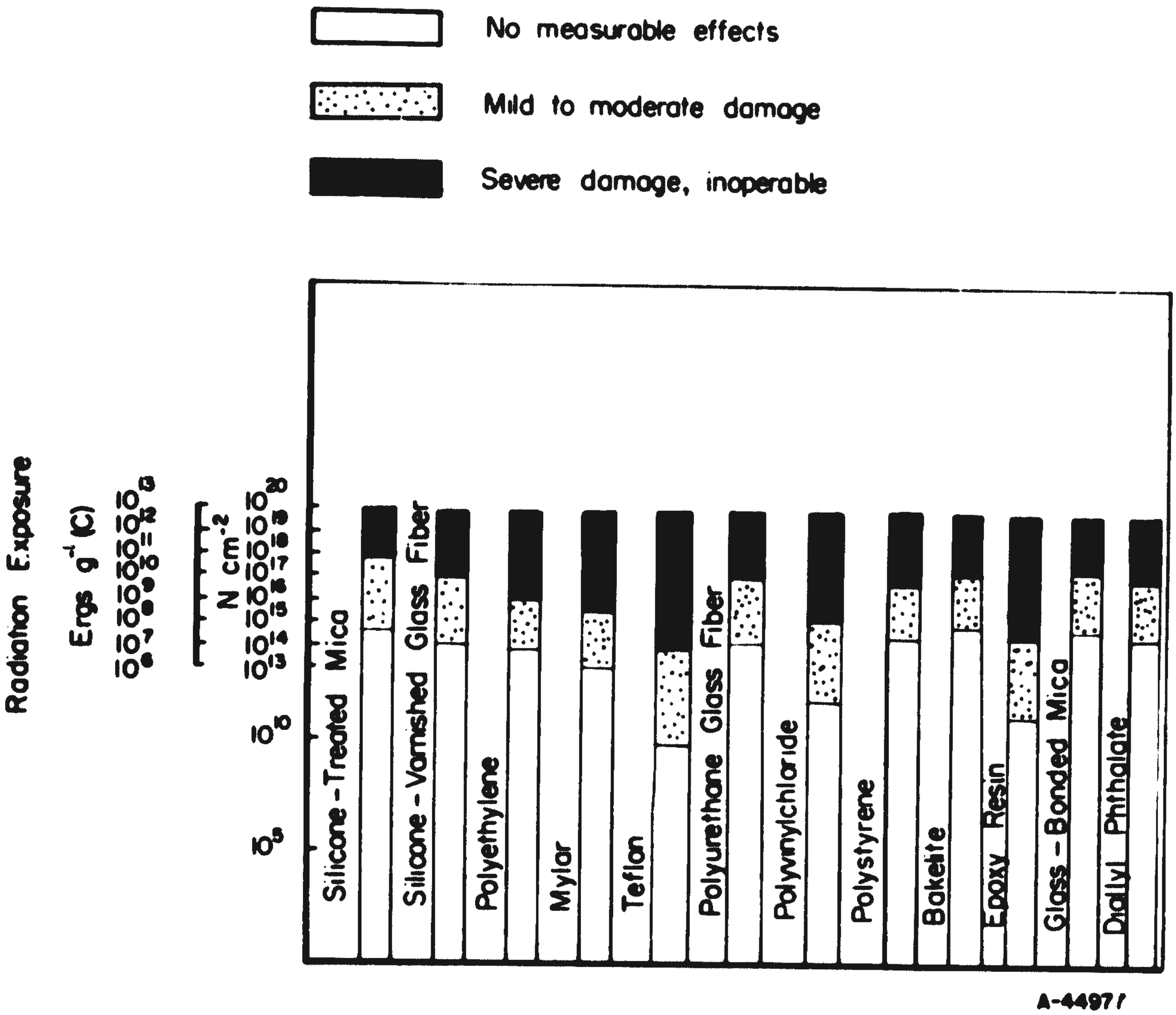

FIGURE A-6. STATE-OF-THE-ART ESTIMATES OF RESISTANCE TO RADIATION DAMAGE FOR SOME COMMONLY USED ORGANIC INSULATION MATERIALS

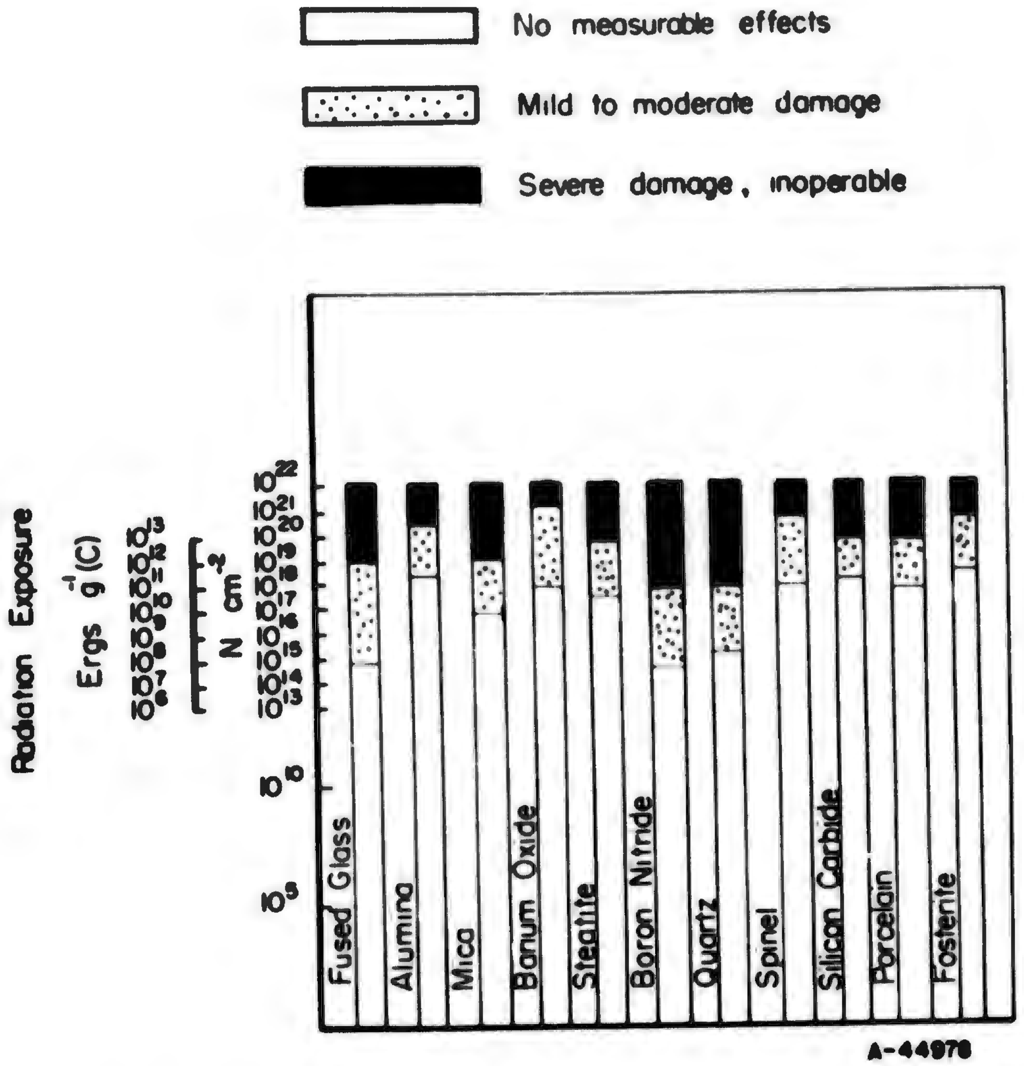

FIGURE A-7. STATE-OF-THE-ART ESTIMATES OF RESISTANCE TO RADIATION DAMAGE FOR SOME COMMONLY USED INORGANIC INSULATION MATERIALS

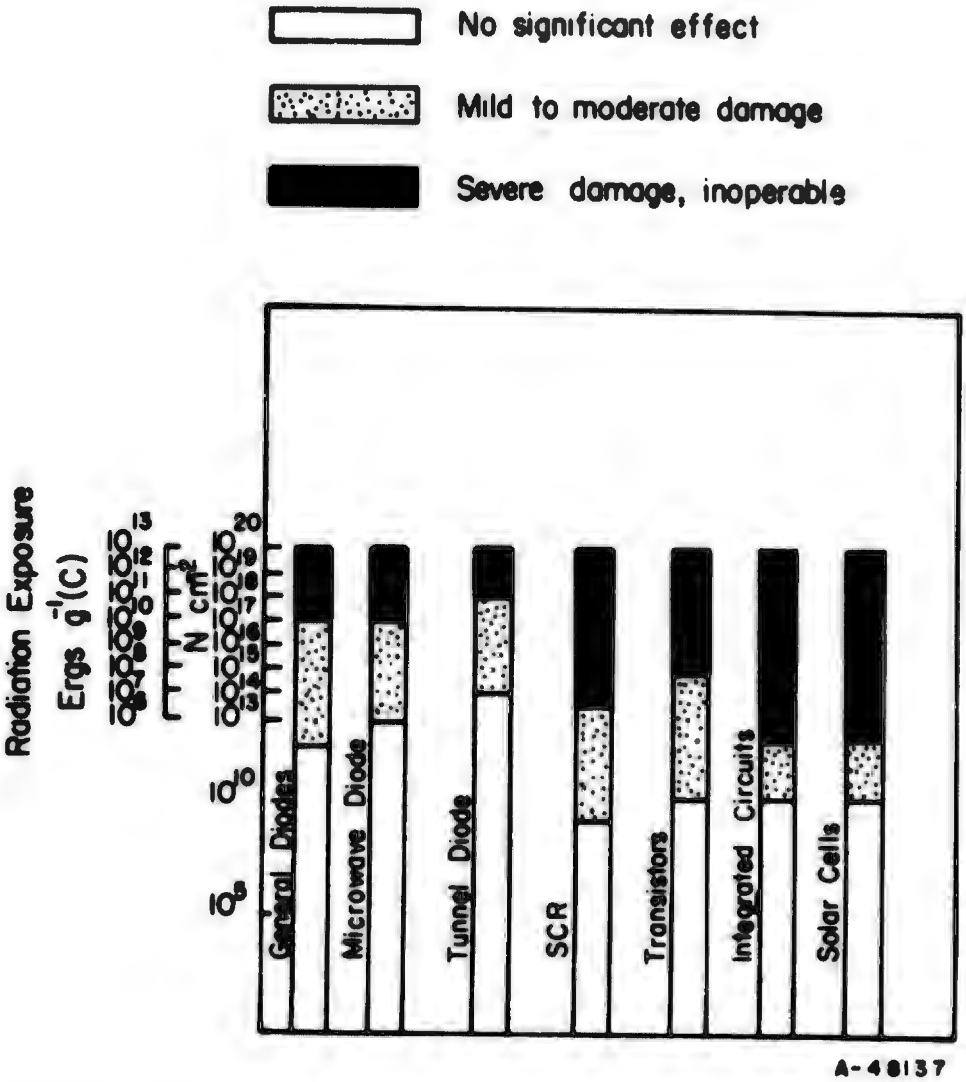

FIGURE A-8. STATE-OF-THE-ART ASSESSMENT OF RADIATION-DAMAGE RESISTANCE FOR VARIOUS SEMICONDUCTORS level of radiation. Once designed, the circuit is tested to at least the necessary level and usually somewhat beyond. Of course, the designer in all cases tries to apply techniques of design which will compensate for the radiation effects.

In many of the reports the indications are that even though these techniques are not good they get results which in most cases are acceptable or slightly better. These techniques definitely require improvement. The level of radiation to which most of the circuits are being irradiated is in the range of $10^{13-15}$ n cm$^{-2}$ fast-neutron exposure. With the need for circuitry that will operate well to high radiation exposures, there is a great need for a good empirical or statistical approach to designing radiation-resistant circuits.

## Programs in Progress*

Current programs on radiation effects on electronic components and materials are summarized below.

Admiral Corporation is determining the effects of nuclear radiation on frequency-control devices.

Aerospace Corporation has an internal program to study radiation damage in selected electronic materials and piece parts.

American Bosch Arma Corporation is studying radiation effects on guidance and navigation equipment.

American Concertone, Inc., is conducting research to develop a radiation-hardened telemetry tape recorder.

Battelle Memorial Institute is planning to test several thousand electronic parts to study their reliability in vacuum, at two radiation rates and at two temperatures.

Bell Telephone Laboratories, Inc., has an internal program to study the radiation environment of Telstar, and the radiation effects on solar cells and radiation-sensing semiconductor devices.

The Bendix Corporation is designing, fabricating, and qualification testing electronic subsystems for start-up control of the SNAP 10A reactor.

The Bendix Corporation is studying the Q-factor technique as a means of predicting radiation damage to the gain of minority-carrier, bipolar transistors.

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*Detailed information on these programs is given in REIC Memorandum 18-C, "Compilation of Nuclear, Pulsed, and Space Radiation Effects Projects" (U). Confidential.

Bendix Corporation is to establish criteria for the design of microwave amplifiers and selection of constituent materials compatible with the space environment.

Bendix Systems Division is studying the relationship of neutron-energy spectrum and neutron-to-gamma ratio to bulk radiation damage for space-vehicle components.

Bendix Systems Division is concerned with the effects of radiation on transducers at cryogenic temperatures.

The Boeing Company is making flash X-ray, Linac, Dynametron and Triga reactor tests to improve the capability for predicting radiation-induced transient behavior of electronic circuits.

The Boeing Company is determining the response of a thin-film memory system to transient nuclear radiation.

The Boeing Company is conducting radiation-effects studies on microcircuits.

Brookhaven National Laboratory is conducting radiation-effects studies on optical, ultrasonic, and magnetic resonance properties of insulators, and on solid-state reactions.

Burroughs Laboratories is determining the threshold of susceptibility and limits of usefulness of electronic components for advanced computers in nuclear environments.

Burroughs Laboratories is to develop and test a computer model which will result in a guidance computer with an MTBF of 1500 hours while exposed to $5 \times 10^{15}$ Nvt and $9 \times 10^9$ ergs $g^{-1}$ (C).

Burroughs Laboratories is obtaining and interpreting data of the parameter response of silicon transistors exposed to pulsed nuclear radiation.

Controls for Radiation, Inc., is conducting investigations of fundamental mechanisms in organic semiconductors of permanent and transient radiation effects.

Douglas Aircraft Company is continuing its internal program to study the performance of microminiaturized electronic systems and thin-film resistors in a radiation-vacuum environment.

Electro-Optical Systems, Inc., is developing fabrication techniques to produce radiation-resistant solar cells.

General Dynamics/Fort Worth is studying the effects of irradiation in air and in vacuum on electrical insulation, potting compounds, and dielectric materials.

General Dynamics/Fort Worth is to conduct a series of irradiation tests on NERVA components.

General Atomic Division of General Dynamics is using optical techniques and the electron linear accelerator to study radiation effects on semiconductors and insulators.

General Atomic Division of General Dynamics is making transient-radiation-effects mechanism studies on electronic parts.

General Atomic Division of General Dynamics is studying defects responsible for the degradation suffered by silicon solar cells when exposed to space radiation.

General Atomic Division of General Dynamics is investigating displacement radiation effects in semiconductors as related to energy of radiation, impurities, and annealing behavior.

General Atomic Division of General Dynamics is studying the effects of pulsed gamma radiation on dynamic electronic components.

General Electric Company is studying the radiation effects on survivable radio guidance systems.

General Electric Company is to develop a portable, radiation-hardened recorder to record structure vibration.

General Electric Company is to develop radiation-hardened, portable equipment to measure the yield of nuclear detonations.

General Electric Company has an internal program to design solid and secondary emission detectors for pulsed-radiation environments.

General Electric Company is to design new techniques of instrumentation and data acquisition in pulsed nuclear environments.

General Electric Company is investigating the effects of pulsed and steady-state radiation on microelectric components and circuits.

General Electric Company is evaluating the effects of radiation on high-temperature, electrical switchgear for space nuclear electrical systems.

General Electric Company is conducting radiation-effects tests on Nimbus stabilization and control subsystem.

General Electric Company is studying the radiation effects on electronics for space nuclear auxiliary-power systems.

General Electric Company is studying basic transient-radiation-effects mechanism on electronic tubes.

Receiving Tube Department of General Electric is continuing radiation tests on high-temperature thermionic circuitry.

Re-Entry Systems Department of General Electric Company is studying the re-entry vehicle heating, signature, and effects on electrical systems.

Re-Entry Systems Department of General Electric Company is designing a TIMM instrumentation recorder and TIMM amplifiers which will be cosmitronic (nuclear hard).

The Tube Department of General Electric is investigating microwave tubes in a pulsed-nuclear-radiation environment.

General Precision, Inc., is making determinations of radiation effects and means for hardening guidance and control systems.

Georgia Institute of Technology is determining the effects of time, temperature, and radiation of the aging characteristics of natural and synthetic quartz-crystal resonators.

Hughes Aircraft Company is studying transient radiation effects on passive parts.

Hughes Aircraft Company is studying basic mechanisms of transient radiation effects on electronic piece parts.

Hughes Aircraft Company has a program to develop radiation-hardened readout circuits.

Hughes Aircraft Company is determining transistor-design effects on radiation resistance of silicon planar transistors.

Hughes Aircraft Company has an internal program to study surface effects on space radiation on semiconductor devices and radiation damage to insulators.

Hughes Aircraft Company is studying the effects of pulsed radiation on guided-missile electronic equipment.

Infrared Industries Inc. , is studying the long-term effects of proton and electron irradiation on PbS infrared detectors at 300 K and 196 K and InSb infrared detectors at 77 K.

International Business Machines Corporation is determining the vulnerability of guidance and control systems to nuclear-weapon environment.

International Business Machines Corporation is contracted to study the effect of pulsed nuclear radiation on selected electronic parts and materials.

International Business Machines Corporation is to determine the effects of Van Allen belt radiation on the primary processor and data-storage unit of the Orbiting Astronomical Observatory.

International Business Machines Corporation is studying pulsed-radiation effects on aerospace digital computers.

Ion Physics Corporation is conducting p-n junctions formation technique evaluations aimed at improving electron-radiation resistance of cells.

The Johns Hopkins University is investigating the effect of geomagnetically trapped radiation on spacecraft components.

Ling-Temco-Vought, Inc. , is studying nuclear ramjet missile and subsystems in radiation environments.

Lockheed Aircraft Corporation is screening and obtaining performance data on SNAP-8 electrical-generating-system materials, controls, and components in a radiation environment.

Lockheed Aircraft Corporation is designing and installing vacuum-chamber and radiation-effects testing equipment for the testing of exposed transistors at controlled temperatures, and the testing of organic potting compounds and electronic equipment subassemblies for Saturn vehicles.

Lockheed Missiles and Space Company is developing nuclear-radiation-hardened spacecraft subsystems.

Massachusetts Institute of Technology is interested in space-oriented radiation effects studies on electronic components and circuitry in active communication satellites.

McDonnell Aircraft Company is developing solid-state radiation detectors and is studying energy levels in semiconductors using gamma radiation.

Melpar, Inc., is conducting research on 2000 F power wire for launch, space, and re-entry conditions.

Atomics International Division of North American Aviation, Inc., is studying the pulsed-radiation effects on selected semiconductor compounds and devices.

Atomics International Division of North American Aviation, Inc., is evaluating the effects of reactor radiation on instrument compartment components such as semiconductor modules, electromagnetic control devices, and reactor control actuators, sensors and insulation systems.

Atomics International Division of North American Aviation, Inc., is investigating the behavior of battery electrodes in a variety of radiation fields.

North Carolina State College is making theoretical and experimental studies of radiation-induced damage to semiconductor surfaces and the effects of this damage on semiconductor device performance.

Northrop Corporation is studying the transient radiation effects on specific circuits.

Northrop Corporation is establishing and refining techniques for predicting the performance of typical circuits for nuclear environments.

Northrop Corporation is determining physical effects of particular radiation on p-n junctions as a means for neutron-proton damage correlations.

Oak Ridge National Laboratory is studying radiation effects in semiconductors, superconductors, and insulating crystals.

Old Diminion College is investigating the theory of defect annealing in irradiated semiconductors.

Parametrics, Inc., is developing a device to measure high temperatures in a nuclear rocket engine.

Purdue University is conducting basic radiation-damage studies on semiconductors, particularly germanium.

Radio Corporation of America is conducting experimental and theoretical studies of the effects of radiation from natural and man-made sources on electronic-systems materials and components.

Radio Corporation of America will evaluate Nuvistors in the DORF reactor.

Radio Corporation of America is making extensive irradiation of n-p solar cells, diodes, and insulation for the Nimbus program.

Rand Corporation is continuing to investigate the effects of space radiation on the lifetime of communication satellites.

Rensselaer Polytechnic Institute is investigating transient radiation damage in semiconductor materials.

Rensselaer Polytechnic Institute is determining the effects of electron, proton, and neutron-gamma radiation on semiconductor materials and thin films.

Republic Aviation Corporation is studying the performance of microminiaturized electronic systems and thin-film resistors in a radiation-vacuum environment.

Sandia Corporation is continuing a study to determine the effect of secondary electrons produced in intense gamma-ray fields on electronic-circuit components.

Sandia Corporation is using analytical approaches to predict the effect that permanent damage in semiconductor devices has upon systems or circuits.

Sandia Corporation is investigating the radiation-induced secondary photocurrents in transistors.

Space Technology Laboratories, Inc., is to irradiate different types of solar cells with 1 Mev electrons.

Sperry Gyroscope Company plans to conduct steady-state radiation and simulated space environmental tests on components to establish tolerance levels for designs of electronic devices.

Sperry Gyroscope Company is conducting investigations on digital computers systems capable of withstanding reactor radiation levels approaching $10^{21} nv_f t$.

UNIVAC Division of Sperry Rand Corporation is studying the effects of nuclear radiation on the MRV Computer.

Sperry Rand Corporation is studying the pulsed-radiation effects on microwave ferrite duplexers.

Sylvania Electric Products, Inc., is studying the effect of reactor radiation on nonoperating vacuum tubes for reactor irradiation of evacuated active circuit components.

Texas Instruments Inc., is investigating high-energy electromagnetic radiation effects on semiconductor devices.

University of Michigan is analyzing the magnetic properties of the radiation-damage sites in semiconductors to determine the nature of the damage.

The University of New Mexico is carrying on experimental and theoretical research in support of the AFSWC transient radiation effects on electronics program.

Westinghouse Electric Corporation is investigating structure and fabrication procedures to maximize the resistance of webbed dendritic silicon solar cells to space-radiation damage.

Westinghouse Electric Corporation is conducting extensive radiation-damage studies to develop solar-cell structures.

Westinghouse Electric Corporation is investigating the mechanisms of current flow in superconductors by determining the effects of radiation on their properties. The experiments are to yield information on the radiation resistance of superconductors.

U. S. Air Force Aeronautical Systems Division is determining the effect of 14.1 Mev neutron radiation on the critical temperatures of superconductors NbZr and $Nb_3SN$.

U. S. Air Force Aerospace Research Laboratories is to determine the energy band structure of cadmium sulfide utilizing electron irradiation.

U. S. Air Force Cambridge Research Laboratories is determining the density of vacancy interstitial parts and defect energy levels produced by neutron bombardment and gamma irradiation.

U. S. Air Force Cambridge Research Laboratories is determining the atomic displacement thresholds and annealing behavior of semiconductor materials exposed to electron bombardment at low temperatures.

U. S. Army Electronics Research and Development Laboratories is continuing its in-house program to develop communications and surveillance equipment which will function in a pulsed-radiation environment.

U. S. Army Frankford Arsenal is determining the effects of transient radiation on the electronic components of fire-control assemblies.

Harry Diamond Laboratories of the U. S. Army Materiel Command is studying nuclear-weapons effects on electronic material.

NASA, Goddard Space Flight Center is comparing Hughes diodes, In 2591 with In 3887 and diodes HMIN with HFIE under 2 Mev electron irradiation.

NASA, Langley Research Center is studying the electron-radiation damage to solar cells and protective windows.

NASA, Langley Research Center is making experimental in-beam measurements of electron-charge storage and discharge in dielectric materials. Electron energies of 20 Kev to 1.25 Mev are being utilized.

U. S. Naval Ordnance Laboratory is determining the electron-radiation effects on magnetic materials.

U. S. Naval Radiological Defense Laboratory is studying the effects of nuclear radiation on shipboard equipment.

U. S. Naval Research Laboratory is investigating the effects of electrons and protons on solar cells and other components.

U. S. Naval Research Laboratory is studying the effects of fast neutrons on ceramic bonded strain gages and is evaluating the electrical resistance of lead-wire insulation.

## Conclusions and Recommendations

A review of the state of the art for radiation-effects research on electronic parts and systems indicates that there is a general increase in activity in each area, and particularly in the area of semiconductors. Although the largest percentage, about 43 per cent, of work being done is in a pulse-radiation environment, space radiation is becoming much more important. Space-radiation studies account for 24 per cent of the total work, and the remaining 33 per cent is steady-state radiation studies. Semiconductor devices such as diodes, transistors, solar cells, and integrated circuits are of the most concern to the researcher. Considering only the electronic parts, semiconductor-device studies comprise roughly 56 per cent of the total investigations. In looking at both the electronic parts and the systems, 42 per cent is related to semiconductor devices, 32 per cent is related to other electronic parts, and 26 per cent to circuits and systems. These figures represent increases (in comparison with the 1962-63 period) for both semiconductors and other electronic parts.

A study of the groups sponsoring research in this area shows that the U. S. Air Force is conducting or sponsoring 37 per cent; U. S. Army, 19 per cent; NASA, 15 per cent; DASA, 5 per cent; U. S. Navy, 5 per cent; AEC, 8 per cent; and other nongovernment organizations, 11 per cent.

All percentages are in terms of number of contracts and not dollar value.

Even though the study of radiation effects on electronic parts has shown a definite increase in activity over the previous report period, there still is much work to be done to improve the devices and our knowledge of radiation effects. Devices such as transducers still have not been studied to any large extent in a radiation environment. While resistors, capacitors, and tubes have been studied in the nuclear environment, they still lack the explanatory theories and equations for predicting radiation effects. The area of semiconductors, although investigated more than any other area, still needs device and fundamental studies. Of course, with the increased interest in space-radiation effects, there are studies to be made in all areas of the electronic-parts field.

# SECTION B

## POLYMERIC MATERIALS

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# POLYMERIC MATERIALS

## Current State of the Art

The major emphasis in current radiation studies continues to be in the areas of space environment and combined environments of radiation, elevated temperatures, and vacuum. Also, radiation effects at cryogenic temperatures have been investigated. This latter work has been performed by only a limited number of organizations because of the type of equipment involved.

There has been a noticeable decrease in the rate of effort in radiation-effects studies. Part of this is due to the amount of information already collected, but much of it is due to a cutback in Government funding and no doubt to a change in overall objectives. However, there is a need for work to be continued on determining the effects of radiation at high exposures, the effects of exposure rate on components, high-impulse effects, and the mechanisms of degradation of various elastomers and plastics. In the latter area, the amount of degradation which can be tolerated in various component parts before failure in operation needs to be determined.

Theoretical studies are continuing. Some of these will result in a better understanding of mechanisms of degradation which in turn should lead to the development of more-radiation-resistant materials. However, a more concentrated effort is needed to speed up the development of polymeric materials which will meet present and future requirements.

Most of the information collected during the report interim was on the effects of radiation on various components and products. These data are presented first on components and then on elastomeric and plastic materials. Presentations are made alphabetically.

## Radiation Effects on Polymeric Components

### Adhesives

The behavior of structural adhesives in space environment has been studied at General Dynamics/Fort Worth by Kerlin and associates, at Lockheed-Georgia by Yasui, at Martin-Marietta by Podlaseck, and at Aerojet-General by Gray and associates. In general, the studies point out that space environment has little effect on shear strength of structural adhesives. Materials are available that are serviceable after exposure to space environment. Ultraviolet radiation will be a factor in considering transparent adhesives which are used to bond transparent materials such as polymethyl methacrylate.

General Dynamics(1,2)* reported the shear strength of nine structural adhesives before irradiation, after irradiation in air or in vacuum, and, for two adhesives, after irradiation at cryogenic temperatures. The average shear strengths are given in Tables B-1 and B-2. The strengths of unirradiated samples and of those irradiated in air and in vacuum at room temperature show, in most cases, little difference. Exceptions are FM-47 and Narmco C. The strength of FM-47 decreased considerably after

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*References are at the end of Section B.

TABLE B-1. AVERAGE ULTIMATE SHEAR STRENGTHS OF ADHESIVES BEFORE AND AFTER IRRADIATION(1, 2)

| Adhesive | Type | Shear Strength Before Irradiation, psi | Shear Strength After Irradiation in Air to a Gamma Exposure of 3.9 x $10^{10}$ Ergs $G^{-1}$(C), psi | Shear Strength After Irradiation in Vacuum to a Gamma Exposure of 2.9 x $10^{10}$ Ergs $G^{-1}$(C), psi |
|---|---|---|---|---|
| Shell 929 | Epoxy | 2264 | 2510 | 1973 |
| Shell 934 | Epoxy | 2562 | 2840 | 2320 |
| FM-1000 | Epoxy polyamide | 6283 | 6086 | 6117 |
| HT-424 | Epoxy phenolic | 3605 | 2599 | 3303 |
| Narmco A | Modified epoxy | 4157 | 3634 | 4959 |
| FM-47 | Vinyl phenolic | 4315 | 3716 | 2561 |
| Metlbond 4021 | Nitrile phenolic | 4370 | 3234 | 3250 |
| Metlbond 406 | Epoxy polyamide | 5560 | 752[a] | -- |
| APCO 1252 | Polyurethane | 2743 | 3262[b] | 3181 |
| Narmco C | Polyurethane | 883 | 20.8 | 454 |

(a) Gamma exposure = 4.9 x $10^{10}$ ergs $g^{-1}$(C). At a gamma exposure of 1 x $10^{10}$ ergs $g^{-1}$(C), shear strength = 5940.

(b) At a gamma exposure of 1 x $10^{10}$ ergs $g^{-1}$(C), shear strength given as 3416 and 4212 in two different tests. At a gamma exposure of 6 x $10^{10}$ ergs $g^{-1}$(C), shear strength = 5028.

## TABLE B-2. AVERAGE ULTIMATE SHEAR STRENGTH OF ADHESIVES BEFORE AND AFTER IRRADIATION AT CRYOGENIC TEMPERATURES(2)

| Adhesive | Type | Shear Strength Before Irradiation, psi | | | Shear Strength After Irradiation at Ambient Temperature (110 F to 143 F), psi(b) | | Shear Strength After Irradiation in Liquid Nitrogen (-320 F), psi(a) | | Shear Strength After Irradiation in Liquid Hydrogen (-423 F), psi(a) |
|---|---|---|---|---|---|---|---|---|---|
| | | Room Temp | -320 F | -423 F | Gamma Exposure, $1.0 \times 10^{10}$ Ergs $G^{-1}$(C) | Gamma Exposure, $6.0 \times 10^{10}$ Ergs $G^{-1}$(C) | Gamma Exposure, $1.3 \times 10^{10}$ Ergs $G^{-1}$(C) | Gamma Exposure, $5.2 \times 10^{10}$ Ergs $G^{-1}$(C) | Gamma Exposure, $1.3 \times 10^{10}$ Ergs $G^{-1}$(C) |
| Metlbond 406 | Epoxy polyamide | 5560 | 2990 | 3250 | 5940 | 752(c) | 2292 | 1600 | 1978 |
| Apco 1252 | Poly-urethane | 2240 | 5040 | 5928 | 4212 | 5028 | 4592 | 4248 | 4536 |

(a) Samples irradiated at cryogenic temperatures. Tested on Instron before warming up.

(b) Tested at room temperature.

(c) Gamma exposure = $4.9 \times 10^{10}$ ergs $g^{-1}$(C).

irradiation in vacuum. Narmco C lost its strength almost completely after air irradiation, whereas under vacuum irradiation, only a 50 per cent loss in strength was experienced. However, this latter adhesive has low shear strength before irradiation.

Gray, et al.,(3) at Aerojet-General irradiated lap shear specimens prepared with epoxy, epoxy phenolic, vinyl phenolic, nitrile-phenolic, and glass-supported epoxy-film adhesives. These were irradiated in air and in vacuum ($10^{-6}$ torr) to a gamma exposure of $10^9$ ergs $g^{-1}$(C) at a temperature of 100 F maximum. These specimens were then tested for shear strength at a temperature of -300 F. In all cases, loss in shear strength was small and the original strength of the adhesive bond specimens could be considered in the design of parts for the above conditions.

Yasui(4) at Lockheed irradiated three epoxy adhesives and one polyurethane adhesive-bonded test specimens immersed in liquid nitrogen. They were then tested at liquid nitrogen temperatures. No loss in shear strength of single-lap shear or in ultimate strength of flatwise specimens was noted.

Hexcell 422-J adhesive was tested in the form of lap-shear specimens at room temperature and at elevated temperatures.(5) Shear-strength tests were conducted at laboratory temperature (75 F) for samples irradiated at ambient temperatures (110-130 F) and at 350 F for the samples irradiated at an elevated temperature. Shear strengths of the samples irradiated at ambient temperature to gamma exposures up to $1.7 \times 10^{11}$ ergs $g^{-1}$(C) were not greatly different from the shear strength of the control samples. Samples irradiated to $6.8 \times 10^{10}$ ergs $g^{-1}$(C) and $2.2 \times 10^{11}$ ergs $g^{-1}$(C) at 250 and 310 F respectively, stored for 7 days at 350 F, and then tested at 350 F lost approximately 15 per cent and 10 per cent of their shear strengths. In contrast, controls which were not irradiated lost approximately 70 per cent of their shear strength. The shear strength of specimens exposed to gamma radiation [$6 \times 10^{10}$ ergs $g^{-1}$(C)] at 450 F decreased from 2500 psi to about 800 psi whereas the controls decreased to 900 psi. Apparently heat alone affected the lap shear strength almost as much as the combined radiation-heat environment.

Koehler and Pefhany(6) irradiated Epon 815, an epoxy cement, and Contact Cement No. 910 (Eastman Chemical Products Inc.) in air to a radiation exposure of $2 \times 10^{10}$ and $1.2 \times 10^{10}$ ergs $g^{-1}$(C), respectively. Both adhesives were reported as being satisfactory after this irradiation.

## Coatings

General Dynamics(7) and Lockheed Missiles and Space Company(8) are currently engaged in determining the effects of nuclear radiation on the optical characteristics of thermal control coatings, but only limited data are available at this time. Preliminary results indicate that negligible change in $\alpha_s/\epsilon$ was experienced by the materials listed below as a result of exposure to $10^9$ ergs $g^{-1}$(C) of Cobalt-60 gamma radiation:

Kemacryl White Lacquer No. M49WC17 (Sherwin-Williams)
Kemacryl Black Lacquer No. M49BC12 (Sherwin-Williams)
Leafing aluminum pigment in the Kemacryl acrylic vehicle (Sherwin-Williams)
Nonleafing aluminum pigment in the Kemacryl acrylic vehicle (Sherwin-Williams)
Fuller 517-W-1 Gloss White Silicone (W. P. Fuller Co.)

Fuller 517-B-2 Flat Black Silicone (W. P. Fuller Co.)
Fuller 172-A-1 Aluminum Silicone (W. P. Fuller Co.)
Fuller 171-A-152 Aluminum Silicone (W. P. Fuller Co.)
Dull Black Micobond L6X962 (Midland Industrial Finisher Co.)
LMSC White Silicate Paint on Al 1100 aluminum alloy.

The irradiations are to continue to $10^{11}$ ergs $g^{-1}$(C) gamma radiation and with other types of penetrating radiation.

Carroll(9) reported the evaluation of materials used on early Mariner spacecraft. An aluminized FEP Teflon and two zinc sulfide silicone paints showed the least degradation when exposed to ultraviolet light. These, however, were not exposed to gamma radiation.

Hormann(10) exposed tailored coatings, some of which contained UV absorbers, to ultraviolet and vacuum. He found that a flat-white titanium dioxide pigmented, silicone-alkyd coating showed good vacuum-thermal and ultraviolet radiation stability. A wide range of $\alpha/\epsilon$ values (0.20 - 0.85) based on this coating are available for various temperature-control conditions. Dispersion of an ultraviolet absorber in a clear film over the basic coating exhibited a protective action in reducing the weight loss through 500 F and in reducing $\Delta\alpha$ at 300, 400, and 500 F.

The black leafing aluminum system would provide high $\alpha/\epsilon$ values (0.90 - 1.40) due to decreased emittance values with increased leafing aluminum content. Hormann also indicates that above 400 F, the polyurethane systems are inadequate in a vacuum-thermal environment.

## Laminates

Epoxy, polyester, phenolic, melamine, and silicone laminates have been investigated as to their behavior in space environment. These do not appear to be adversely affected by nuclear-radiation exposure of $10^{10}$ ergs $g^{-1}$(C) and UV exposures of 2 pyrons for 500 hours. The polyesters were found to be the more sensitive to UV irradiation, but may be improved with the incorporation of UV stabilizers. Phenolics appear to be least sensitive to UV exposure. Epoxy laminates show improved strengths when tested in a vacuum environment. Present information indicates that cryogenic temperatures will not be a serious problem with structural laminates.

Kerlin and Smith(1, 11) tested nine glass-fabric laminates and one honeycomb laminate for effects of radiation-vacuum environment. These included Mobiloy AH-81, CTL-91-LD, and Conolon 506 (phenolic), Paraplex P-43 and Selectron 5003 (polyester), DC-2104 and DC-2106 (silicone), Epon 828 (epoxy), and HRP Honeycomb (phenolic). Tests indicated that the combined effects of radiation and vacuum (approximately $10^{10}$ ergs $g^{-1}$(C) and $10^{-7}$ torr) have no deleterious effect on the strength of the laminates except for P-43 and DC-2104. Paraplex P-43 lost tensile strength rapidly after $10^{10}$ ergs $g^{-1}$(C). At an exposure of $3.1 \times 10^{10}$ ergs $g^{-1}$(C) in vacuum, tensile strength decreased by approximately 20 per cent as compared with a loss of 6 per cent when irradiated in air to $3.9 \times 10^{10}$ ergs $g^{-1}$(C). Similarly Silicone DC-2104, after $10^{10}$ ergs $g^{-1}$(C) in vacuum, decreased in tensile strength. At $2.9 \times 10^{10}$ ergs $g^{-1}$(C), tensile strength decreased by 15 per cent. In air no loss was observed after $3.9 \times 10^{10}$ ergs $g^{-1}$(C).

Gray(3) irradiated phenolic, polyester, epoxy, and silicone laminates in air and in vacuum ($10^{-6}$ torr) to $10^{9}$ ergs $g^{-1}$(C). These were then tested at a temperature of -300 F. The environmental conditioning improved the strength of epoxy with unidirectional glass fibers and phenolic with glass fabric. However, phenolic with high silica exhibited a slight degradation in ultimate strength. No trend of improvement or degradation due to environmental exposure was found for the remaining materials.

Kerlin and Smith(1) irradiated Conolon 506 (phenolic) and Paraplex P-43 (polyester) at liquid nitrogen and liquid hydrogen temperatures to a gamma exposure of $6 \times 10^{10}$ ergs $g^{-1}$(C). No significant change in ultimate tensile strength occurred at the liquid nitrogen temperatures. Although tensile strength increased somewhat at the liquid hydrogen temperature, this was believed to be due to chemical reactions between the ionized hydrogen and components of the adhesive rather than to the lower temperature.

A laminate consisting of 181 glass-cloth (A-1100 finish) impregnated with AP-Resin-XHU (a phosphonitrillic chloride polymer) called Dynalam was irradiated for 55 hours in an ambient temperature (120-130 F) and at 450 F.(5) Some of the laminates contained a curing agent and some did not. The samples were tested at room temperature. After irradiation at 455 F to $6 \times 10^{10}$ ergs $g^{-1}$(C), tensile strength of a laminate containing a curing agent dropped from 39,400 psi to 30,600 psi while the laminate with no curing agent decreased from 31,000 to 21,000 psi.

## Potting Compounds

Several potting compounds have been found serviceable for space applications. Cure is an important factor for stability in space, a higher temperature cure being preferred to a room-temperature cure. Solvent systems are generally not satisfactory, as they tend to dissolve the insulation of imbedded wire.

Clauss reported at the 1961 Proceedings of the Institute of Environmental Sciences(12) that Epocast 202/9615 showed excellent stability to both radiation [$10^{10}$ ergs $g^{-1}$(C)] and vacuum exposure. Hysol 12-007A/B also showed excellent stability, although cure shrinkage was greater than 2 per cent and shrinkage during exposure to vacuum at 170 F was 4.5 per cent. Three materials were found satisfactory after exposure to vacuum at 170 F. These were not subjected to radiation exposure. They were manufactured by Products Research Company and included PRC 1535 A/B, Dow-Corning Corporation's DC 502/501 and Furane Plastics Epibond 1210/9615 epoxy-polyamide materials.

Kerlin and Smith(1) studied the effect of radiation and vacuum on several silicone and epoxy potting compounds. These were:

| | |
|---|---|
| RTV-501 | Silicone |
| RTV-60 | Silicone elastomer |
| EC-2273 | A fluorinated elastomer |
| Scotchcast 212 | Epoxy |
| Epon 828/Z | Epoxy |

Epon 828/Z and Silicone DC-R7521 did not change significantly in weight or compressive strength (compression-deflection) when irradiated to $10^{10}$ ergs $g^{-1}$(C) at a pressure of $2 \times 10^{-6}$ torr. Both materials changed in color to a dark brown. Compressive strength of RTV-60 silicone rubber increased considerably. Compression deflection at 25 per cent increased 667 per cent, while at 0.02-inch compression this property increased 405 per cent. Irradiation in air of Scotchcast 212 and EC-2273 had little effect on their properties. Irradiation in vacuum caused compressive strength of the Scotchcast to increase by almost 50 per cent. A small increase was noted for EC-2273. With Silicone RTV-501, radiation both in air and in vacuum increased compressive strength. This increase was more noticeable in vacuum.

Several potting compounds were investigated by Armstrong(13). These included:

| | |
|---|---|
| Scotchcast No. 3 | Minnesota Mining and Manufacturing Co. |
| Stycast 2651 MM | Emerson and Cuming |
| RTV-501 | Dow Corning |
| Epon 828/D | Shell Chemical Co. |
| Insulating Lacquer 1162 A/B | Dennis Chemical Co. |
| 12-007 | Hysol Corp. |
| Stayfoam AA402 | American Latex |
| EB 758 T | MICA Corp. |
| Scotchcast Foam Resin No. 603 | Minnesota Mining and Manufacturing Co. |

Insulation-resistance measurements were taken before, during, and after irradiation. Resistances of the samples were found to be dependent on the exposure rate. The greatest change occurred in the mica reference sample; the potting materials served to decrease the rate effects in the other samples. RTV-501 showed an appreciable change in insulation resistance at the higher exposure rates, as did Dennis Insulating Lacquer 1162 and American Latex Stayfoam AA-402.

## Seals, O-Rings, and Gaskets

Gray, et al.(3) determined the effect of gamma radiation on seals by subjecting to radiation components containing the seals, such as solenoid valves, check valves, relief valves, actuators, ball valves, and regulators. These were subjected to 2 weeks in a vacuum with the temperature cycled daily from -175 to +50 C, then subjected to radiation exposure in air, and finally to a repetition of the vacuum exposure. Although the effects of environment on the seals in these components were not given directly and are not strictly comparable, it would appear that Neoprene, Viton-A, Kel-F, and some Teflon seals were satisfactory. Butyl rubber, nitrile rubber and other Teflon seals were adversely affected.

The elastomer sealing materials within a solenoid changed in hardness or size, thereby increasing the mechanical forces required to actuate the valve. This is illustrative of the type of problems encountered. Leakage rates of components with elastomeric seals and seats generally increased as a result of the combined environmental-exposure testing.

Gray(3) also determined the effect of radiation and vacuum exposure on the compressive and tensile strength of Fluorobestos, a mixture of Teflon and random asbestos fibers. The material remained useful as a gasket material after exposure to $10^9$ ergs $g^{-1}$(C) in vacuum.

Kerlin and Smith(1, 11) continued their evaluation of Viton-B, nitrile rubber, Neoprene, and natural rubber as O-rings. These materials were subjected to nuclear radiation and vacuum environments and tested in air and in vacuum. Nitrile rubber (Parker Compound 66-581) was not seriously affected by radiation either in air or vacuum to an exposure of $10^9$ ergs $g^{-1}$(C). At $10^{10}$ ergs $g^{-1}$(C), the effect of irradiation was about the same whether or not air was present. Tensile strength of the rubber was somewhat lower in vacuum than in air. Weight loss was approximately 1 per cent in vacuum.

Tensile strength and elongation of natural rubber O-rings changed considerably when irradiated in vacuum to $9 \times 10^9$ ergs $g^{-1}$(C). However, up to $5 \times 10^9$ ergs $g^{-1}$(C), there was little difference between irradiation in air and in vacuum. No significant change in weight occurred.

Irradiation to $10^9$ ergs $g^{-1}$(C) had practically no effect on Neoprene either in air or in vacuum. Tests in vacuum showed little difference from tests in air. Radiation effects on Viton-B also appeared to be similar, whether irradiated in air or in vacuum. Elongation decreased considerably after $10^9$ ergs $g^{-1}$(C).

A nitrile rubber (PRP 737-70) containing an antirad showed better tensile strength when irradiated in air than in vacuum. However, after $10^9$ ergs $g^{-1}$(C), elongation decreased considerably.

All of the materials except Viton-B showed good stability to radiation both in air and in vacuum to about $10^9$ ergs $g^{-1}$(C), but above this exposure changes in physical properties were large. Viton-B showed considerable change at $10^9$ ergs $g^{-1}$(C). At the highest exposure, natural and nitrile rubber appeared to be damaged more by exposure to gamma radiation in vacuum than by exposure in air. The Neoprene and Viton-B were not tested to sufficiently high radiation levels in vacuum to determine the effect of vacuum on gamma-radiation damage.

General Dynamics(5) irradiated four O-ring formulations manufactured by Precision Rubber Products Corporation (PRP). Three of the formulations were developed in a cooperative program by B. F. Goodrich Co. and PRP to develop radiation-resistant O-ring compounds. The fourth was a standard PRP Viton-B formulation. Data were given for a Neoprene rubber containing 5 parts Antiox 4010, and for Viton-B. These materials, when irradiated at 375 F in air and in fluid, maintained considerable tensile strength and elongation. Data are given in Table B-3.

Lewis(7), at General Dynamics/Fort Worth irradiated two elastomers as O-rings. These were also compositions containing antirads and were developed by B. F. Goodrich and Precision Rubber Products Companies. A summary of the effects of irradiation is given for these materials in Tables B-4 and B-5. These appear to be serviceable to a radiation exposure of $10^{10}$ ergs $g^{-1}$(C).

Koehler and Pefhany(6) tested a gaging system for reactor pressure tubes designed to measure diameter, surface defects, wall thickness, and straightness in a defuelled, drained channel during periods of reactor shutdown. The ultrasonic crystal used to trace the wall contour required water coupling to the tube wall, and therefore some of the O-rings were wet or immersed in water during the testing of the gaging system. As a result, some of the O-ring materials were tested wet and some dry, depending on the location. Neoprene O-rings were found to be satisfactory to a radiation exposure of approximately $10^{10}$ ergs $g^{-1}$(C) both wet and dry. Although the O-rings had hardened, they were considered satisfactory for this application. Silicone rubber O-rings (Armet Green and Grey, and Linear White) and a white Teflon O-ring were considered satisfactory when dry, but they hardened considerably when wet.

## Electrical Insulation

Electrical-insulation materials designated as DC-7-170 (silicone), Geon 2046 and Geon 8800 (polyvinyl chloride), Estane 5740X1 (polyurethane), Kynar (polyvinylidine fluoride), Kel-F-81 (polytrifluorochloroethylene), Duroid (a Fiberglas-reinforced Teflon)

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TABLE B-3. ULTIMATE PROPERTIES OF PRECISION RUBBER PRODUCTS CORP. O-RING COMPOUNDS 2277 SPECIAL AND 19007(5)

| Compound | Gamma Exposure, ergs/g$^{-1}$(C) | Neutron (n/cm$^2$, E>2.9 Mev) | Irradiation Time and Temperature, hr/°F | Medium | Tensile Strength, psi(a) | | Ultimate Elongation, %(a) | |
|---|---|---|---|---|---|---|---|---|
| | | | | | No. 222 O-Rings | Tensile Specimens | No. 222 O-Rings | Tensile Specimens |
| 2277 Special | Controls | Controls | 5/75 | Air | 2638/140/5 | 2795/233/2 | 372/23/5 | 523/--/1 |
| | $1.2 \times 10^9$ | $2.3 \times 10^{14}$ | 5/75 | Air | 2339/158/5 | 2674/146/3 | 287/14/5 | 362/11/3 |
| Neoprene containing 5 pph Antiox 4010 | Controls | Controls | 5/375 | Air | 1499/234/3 | 1379/102/3 | 160/21/3 | 144/6/2 |
| | $9.1 \times 10^8$ | $1.1 \times 10^{14}$ | 5/375 | Air | 788/60/5 | 1075/75/3 | 65/5/5 | 69/3/3 |
| | Controls | Controls | 5/75 | Oronite 8515 | 2584/82/3 | 2572/174/3 | 338/11/3 | 430/54/3 |
| | $1.2 \times 10^9$ | $2.3 \times 10^{14}$ | 5/75 | Oronite 8515 | 1771/216/5 | 2389/110/3 | 227/23/5 | 323/14/3 |
| | Controls | Controls | 5/375 | Oronite 8515 | 1631/87/3 | 1860/146/3 | 213/8/3 | 284/8/3 |
| | $9.1 \times 10^8$ | $1.1 \times 10^{14}$ | 5/375 | Oronite 8515 | 1247/454/5 | 2145/203/3 | 142/28/5 | 212/17/3 |
| 19007 Viton-B | Controls | Controls | 5/75 | Air | 1984/89/3 | 2021/135/3 | 254/23/3 | 305/1.2/3 |
| | $1.2 \times 10^9$ | $2.3 \times 10^{14}$ | 5/75 | Air | 2041/181/3 | 2149/312/3 | 141/5/3 | 149/12/3 |
| | Controls | Controls | 5/375 | Air | 2024/57/3 | 2061/159/3 | 254/8/3 | 282/20/3 |
| | $1.1 \times 10^9$ | $2.9 \times 10^{14}$ | 5/375 | Air | 1131/72/3 | 1285/55/3 | 146/5/3 | 191/7/3 |
| | Controls | Controls | 5/75 | 4P3E fluid(b) | 1973/44/3 | 1951/116/3 | 256/4/3 | 293/35/3 |
| | $1.2 \times 10^9$ | $2.3 \times 10^{14}$ | 5/75 | 4P3E fluid | 1937/180/5 | 2319/154/3 | 138/7/5 | 171/9/3 |
| | Controls | Controls | 5/375 | 4P3E fluid | 1863/141/3 | 2005/181/3 | 259/10/3 | 307/34/3 |
| | $1.1 \times 10^9$ | $2.9 \times 10^{14}$ | 5/375 | 4P3E fluid | 1818/59/4 | 1844/176/3 | 164/6/4 | 187/14/3 |

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(a) Data are given as $\bar{x}$/S.D./n where $\bar{x}$ = average value, S D. = standard deviation of an individual observation estimated from the range, and n = number of specimens used in calculating $\bar{x}$ and S.D.

(b) 4P3E-Fluid-mixed isomers of phenoxyphenyl ether.

### TABLE B-4. SUMMARY OF EFFECT OF IRRADIATION AT ROOM TEMPERATURE ON PRPC O-RING COMPOUND 4387 (SBR RUBBER)(7)

| Gamma Exposure, ergs/g$^{-1}$(C) | Specific Gravity at 25 C | | Compression Set, %(a) | Hardness, Shore A | | Tensile Strength, psi(b) | Ultimate Elongation, % |
|---|---|---|---|---|---|---|---|
| | Before | After | | Before | After | | |
| 0 | | | 9.04 | | | 1890.6 | 449.3 |
| $4.9 \times 10^8$ | 1.164 | 1.172 | 14.42 | 64.6 | 64.4 | 2014.1 | 453.3 |
| $1.8 \times 10^9$ | 1.166 | 1.173 | 27.88 | 65.0 | 65.0 | 2003.2 | 427.3 |
| $3.6 \times 10^9$ | 1.165 | 1.157 | 43.26 | 65.0 | 67.4 | 1811.2 | 339.4 |
| $1.2 \times 10^{10}$ | 1.162 | 1.160 | 74.95 | 65.0 | 75.6 | 1779.7 | 209.5 |
| $3.5 \times 10^{10}$ | 1.163 | 1.184 | 89.77 | 64.8 | 86.4 | 1749.2 | 65.5 |
| $1.6 \times 10^{11}$ | 1.160 | 1.211 | -- | -- | -- | 3601.8 | 18.3 |

(a) Total time in compression for all specimens, 310 hours.

(b) Tensile values are average for 15 samples. All others are average for 5.

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TABLE B-5. SUMMARY OF EFFECTS OF IRRADIATION AT ROOM TEMPERATURE ON PRPC O-RING COMPOUND 1387 (NITRILE RUBBER)(7)

| Gamma Exposure, ergs/g$^{-1}$(C) | Specific Gravity at 25 C | | Specific Gravity at 25 C | | Compression Set, %(c) | Hardness, Shore A | | Tensile Strength, psi(d) | Ultimate Elongation, % |
|---|---|---|---|---|---|---|---|---|---|
| | Before | After(a) | Before | After(b) | | Before | After | | |
| 0 | | | | | 5.63 | 70.2 | 70.4 | 2704.8 | 342.3 |
| $4.9 \times 10^{8}$ | 1.250 | 1.252 | 1.251 | 1.243 | 16.96 | 70.2 | 71.4 | 2512.3 | 300.7 |
| $1.8 \times 10^{9}$ | 1.249 | 1.253 | 1.251 | 1.249 | 36.91 | 70.4 | 71.0 | 2621.4 | 290.7 |
| $3.6 \times 10^{9}$ | 1.249 | 1.254 | 1.251 | 1.245 | 55.19 | 70.2 | 73.2 | 2809.1 | 266.7 |
| $1.2 \times 10^{10}$ | 1.250 | 1.258 | 1.252 | 1.251 | 82.96 | 69.6 | 82.6 | 3206.8 | 164.0 |
| $3.5 \times 10^{10}$ | 1.250 | 1.267 | 1.253 | 1.257 | 94.44 | 70.6 | 92.4 | 4128.3 | 64.0 |
| $1.6 \times 10^{11}$ | 1.244 | 1.293 | 1.254 | 1.280 | -- | -- | -- | 6018.7 | (e) |

(a) After irradiation in air.
(b) After irradiation in ASTM No. 3 oil. Total oil soak time, 171 hours.
(c) Total time in compression for all specimens, 310 hours.
(d) Tensile values are average for 15 samples. All others are average for five.
(e) Broke immediately. Elongation could not be measured.

and Mylar (polyester) were irradiated and their physical properties tested at General Dynamics.(1, 11) Only mechanical properties are presented here; the electrical properties of insulation materials are discussed in the section entitled Electronic Components and Equipment.

Silicone DC 7-170 increased in tensile strength 144 per cent while elongation decreased by 83.5 per cent when subjected to $8.8 \times 10^8$ ergs $g^{-1}$(C) in vacuum. Average weight loss was about 0.2 per cent. Mechanical properties were more severely affected by the vacuum-irradiation environment than by irradiation in air.

Polyvinyl chloride materials and polyvinylidene chloride are affected to approximately the same extent in vacuum and in air. Polyurethane and Duroid are less affected by irradiation of $10^9$ ergs $g^{-1}$(C) in vacuum than in air. Mylar increased in tensile strength, but decreased in elongation when irradiated in vacuum. No significant weight loss was noted. Mylar C was also irradiated at liquid nitrogen and liquid hydrogen temperatures.(2) The effect of the cryogenic temperatures was to increase tensile strength and decrease elongation. At the liquid nitrogen temperature, gamma irradiation decreased the tensile strength, but not below the original value at room temperature. This material appears satisfactory for applications in a vacuum-gamma radiation environment to $10^{10}$ ergs $g^{-1}$(C). However, it is susceptible to ultraviolet-radiation damage.

Koehler and Pefhany(6) reported polyethylene, Zytel Nylon 33, and polyvinyl chloride as satisfactory to $10^{10}$ ergs $g^{-1}$(C) in air when dry.

## Dielectric Materials

Kerlin and Smith(1, 11) investigated the physical properties of several dielectric materials when irradiated in air and in a vacuum ($10^{-6}$ to $10^{-7}$ torr). These included Marlex 6002 (high-density polyethylene), Teflon TFE, Tedlar (polyvinyl fluoride), and H-film (polyimide). The polyimide film showed the highest tensile strength and the greatest stability to radiation both in air and in a vacuum. After $3 \times 10^{10}$ ergs $g^{-1}$(C), tensile strength dropped only from 19,470 psi to 17,903 psi when irradiated in air and 18,877 psi when irradiated in vacuum. Elongation decreased from 128 per cent to 83 per cent when irradiated in air and 103 per cent when irradiated in vacuum. Tedlar also showed excellent stability to radiation both in air and in vacuum when irradiated to $10^9$ ergs $g^{-1}$(C). Marlex had a greater elongation than these before irradiation, but after $10^9$ ergs $g^{-1}$(C), this property decreased to a greater extent.

When irradiated at cryogenic temperatures (liquid hydrogen and liquid nitrogen)(2), tensile strength of the polyimide film increased and elongation decreased due to the temperature, but the effect of radiation up to $10^{10}$ ergs $g^{-1}$(C) was very slight.

## Thermal Insulation

Two polyurethane foamed materials manufactured by Chemical Plastics Research Co. were irradiated in vacuum at General Dynamics and tested for compression strength at 25 per cent deflection in air and in vacuum.(1) After $10^9$ ergs $g^{-1}$(C), compression strength of CPR-20 did not change when tested in air (100 psi to 99 psi). When tested in vacuum to $5 \times 10^8$ ergs $g^{-1}$(C), compression strength (compression deflection) at 25 per cent deflection increased to 124.5 psi. With the second material, CPR-1021-2, compression strength at 25 per cent deflection again did not change significantly when tested

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in air after being irradiated in vacuum to 5 x $10^8$ ergs $g^{-1}$(C) gamma. Values were 33 psi and 29.8 psi before and after irradiation. When tested in vacuum, after the same radiation exposure, compression strength increased to 49.4 psi.

Stafoam AA 402, also a polyurethane thermal-insulation material, was irradiated at cryogenic temperatures.(2) There appeared to be an approximate threshold point for compressive resistance of this material at an exposure of about 5 x $10^9$ ergs $g^{-1}$(C).

Styrofoam 22, a polystyrene thermal insulation, showed a radiation threshold of 2-5 x $10^9$ ergs $g^{-1}$(C) at cryogenic temperatures.

## Radiation Effects on Elastomeric Materials

Additional data on polyurethane rubber and foam, arylene modified silicones, nitrile rubber, and Neoprene rubbers were received at REIC during the report period.

### Polyurethane Rubber

Seilon UR-80T, a polyurethane elastomer produced by Seiberling Rubber Co., was irradiated at two temperatures, 75 F and 250 F; the gamma exposure was 9.4 x $10^{10}$ ergs $g^{-1}$(C) at the lower temperature and 8 x $10^{10}$ ergs $g^{-1}$(C) at the higher temperature.(7) At the lower temperature, compression set increased from 74 per cent to 95 per cent, tensile strength decreased from 4648.4 psi to 1967.8 psi, and elongation decreased from 623.8 per cent to 42.1 per cent. Shore A hardness changed from 96.5 to 92.5. At 250 F, after 8 x $10^{10}$ ergs $g^{-1}$(C), tensile strength was 1751.2 psi and elongation was 50.5 per cent. Hardness decreased from 95.8 to 84.8, and compression set increased to 106.7 per cent.

A flexible polyurethane foam, a blown polyether urethane produced by General Foam Co., was also irradiated at 75 F and 250 F. At the lower temperature, compression set at 50 per cent deflection increased from 8 per cent to 20 per cent at $10^9$ ergs $g^{-1}$(C), to 95 per cent at 8.3 x $10^9$ ergs $g^{-1}$(C), and to 100 per cent at 2.8 x $10^{10}$ ergs $g^{-1}$(C). At the highest exposure, 9.4 x $10^{10}$ ergs $g^{-1}$(C), the material adhered to the plates. At 250 F, compression set of the unirradiated material was 103 per cent. At 2.4 x $10^{10}$ ergs $g^{-1}$(C), the value at 50 per cent deflection was still 103 per cent. At 8 x $10^{10}$ ergs $g^{-1}$(C), difficulty in shrinkage and sticking to the plates again was encountered.

### Silicone Rubber

Ossefort(14) studied the thermal and radiation stability of arylene modified siloxanes prepared by Union Carbide Corporation. Although it was expected that these structures would improve thermal stability, this was not the case. However, it appeared that improved radiation resistance of the silicone rubbers resulted. The structures of the arylene modified siloxanes and silcarbanes are shown in Figures B-1 and B-2. The radiation resistance of these materials as compared to the silicone rubbers is shown in Figure B-3. It will be noted that they were not tested above 8 x $10^7$ ergs $g^{-1}$(C), but from the slope of the curves, the improvement in radiation resistance would be expected to extend to higher exposures.

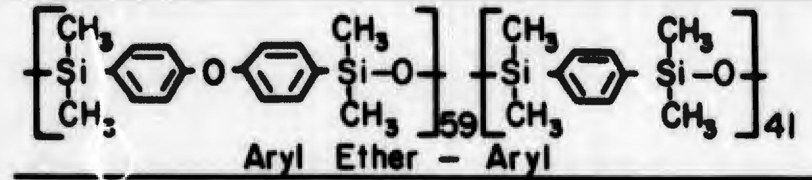
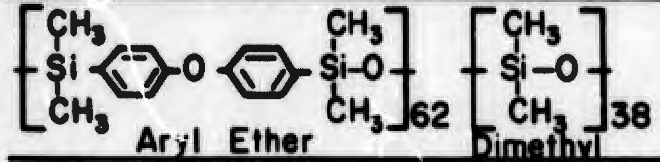
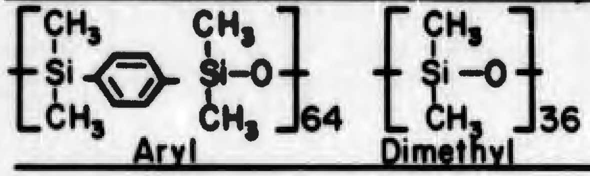
| Unit Structure* | Analyzed For | Calculated | Found |
|---|---|---|---|
|  Aryl Ether - Aryl | Carbon<br>Hydrogen<br>Silicon | 61.4<br>7.1<br>22.0 | 59.8<br>7.0<br>21.9 |
|  Aryl Ether; Dimethyl | Carbon<br>Hydrogen<br>Silicon | 52.0<br>7.1<br>26.0 | 51.4<br>6.7<br>25.4 |
|  Aryl; Dimethyl | Carbon<br>Hydrogen<br>Silicon | 48.6<br>7.8<br>31.0 | 45.1<br>7.3<br>30.8 |

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FIGURE B-1. ANALYTICAL DATA FOR ARYLENE MODIFIED POLYSILOXANES(14)

*As furnished by Union Carbide Corp. (All gums contained 0.3 wt % MeViSi-O-, which was neglected in the calculated values.)

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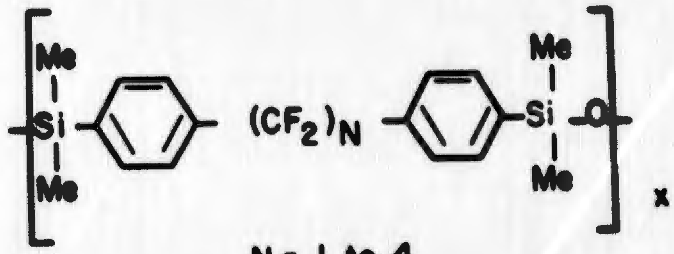
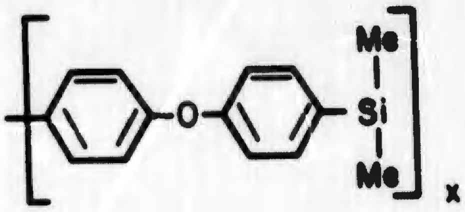


N = 1 to 4

a. Fluorinated Arylene Modified Polysiloxane



b. Arylene Modified Silcarbanes

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FIGURE B-2. SILOXANE AND SILCARBANE POLYMERS(14)

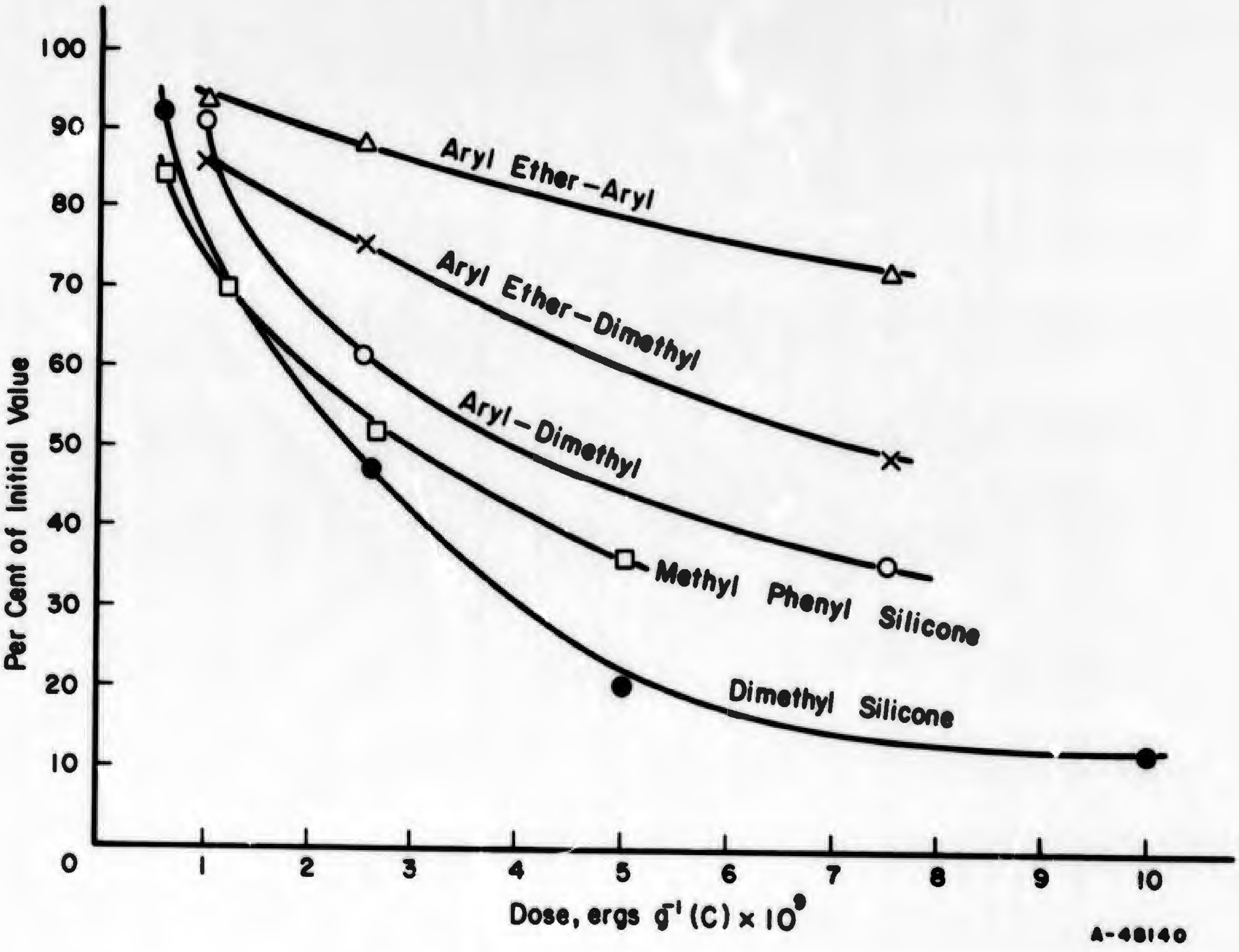

FIGURE B-3. EFFECT OF GAMMA RADIATION ON THE ULTIMATE ELONGATION OF VARIOUS SILOXANES(14)

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Two other phases of the program are in progress, but results of the studies are not yet available. These include the preparation of arylene modified siloxanes connected by perfluoromethylene groups at Stanford Research Institute and the preparation of arylene modified silcarbanes at Yarsley Research Laboratories in London.

## Nitrile Rubber

McGarvey(15) evaluated approximately 200 potential antirads to determine the best one for low acrylonitrile content NBR (78/18 butadiene/acrylonitrile). The activity of the antirad was judged by the per cent of initial strain and Shore A hardness values after a gamma exposure of 50 megarads [$5 \times 10^9$ ergs $g^{-1}$(C)]. Table B-6 shows the results of the three best antirads.

TABLE B-6. EVALUATION OF THE BEST ANTIRADS IN NBR(15)

| | Per Cent of the Original Property After Exposure to $5 \times 10^9$ Ergs $G^{-1}$(C) | | | |
|---|---|---|---|---|
| Additive (5 pph Polymer) | Tensile | Elongation | Hardness, Shore A | Strain, NBS |
| None (control) | 92 | 33 | 116 | 34 |
| 2,2-diphenyl-1-picrylhydrazyl | 109 | 61 | 104 | 58 |
| 1,1-diphenyl-2-picrylhydrazine | 98 | 63 | 108 | 55 |
| N,N'-diphenyl paraphenylenediamine | 82 | 51 | 110 | 55 |

The antirad activity of 1,1-diphenyl-2-picrylhydrazine ($DPPH_2$) present at a concentration of 5 pph polymer was also investigated in SBR, and butyl and natural rubber. A significant antirad activity was exhibited in only the SBR vulcanizate.

Bonanni(16) irradiated Buna N rubber to a radiation exposure of $7.9 \times 10^7$ ergs $g^{-1}$(C) in air, in a closed atmosphere, and in vacuum ($5 \times 10^{-5}$ torr). The per cent change in weight was negligible in each case. Tensile strength decreased more when irradiated in a vacuum than when irradiated in air. Values in a closed atmosphere were in general intermediate. Elongation followed somewhat the same pattern. Thus, this rubber appeared to be less satisfactory when irradiated in a vacuum than when exposed to radiation in air.

## Neoprene Rubber

Bonanni(16) also irradiated Neoprene rubber in air, in a closed atmosphere, and in vacuum ($5 \times 10^{-5}$ torr). At the maximum exposure, $7.9 \times 10^7$ ergs $g^{-1}$(C), there was no appreciable change in tensile strength. Elongation decreased to a greater degree when irradiated in a closed atmosphere (limited amount of air) than when irradiated either in vacuum or in air. In general, there was not much difference in elongation when irradiated in air or in a vacuum.

## Radiation Effects on Plastic Materials

### Acrylonitrile/Butadiene/Styrene Terpolymers (ABS)

Lewis(7) determined the effect of irradiation on two types of ABS polymers at 75 F and at 250 F. These materials were Kralastic MV and Kralastic SRA plastics. At 75 F, both materials increased in tensile strength when irradiated up to 2.8 x $10^{10}$ ergs $g^{-1}$(C). At 9.4 x $10^{10}$ ergs $g^{-1}$(C), tensile strength decreased by approximately 30 per cent of the original. The MV material increased in tensile strength from 3730 psi to a maximum of 5070 psi and then decreasing to 2140 psi. The SRA material increased from 4730 psi to 6330 psi and then decreased to 2820 psi. In both cases, hardness increased with increasing radiation exposure. At elevated temperatures this change takes place more rapidly, and at 250 F Kralastic MV nonirradiated had a tensile strength of 3670 psi, while the material after being irradiated to 8 x $10^{10}$ ergs $g^{-1}$(C) at 250 F decreased in tensile strength to 1230 psi. Under the same conditions, the SRA material decreased from 4350 psi to 875 psi. Hardness of both materials increased with increasing radiation at the elevated temperatures in approximately the same fashion as it did when irradiated at room temperature.

### Phosphonitrillic Chloride Polymers

General Dynamics(5) developed a resin which is a derivative of phosphonitrillic chloride and which is designated as AP-Resin-XHU. The resin contains a number of unreacted polar groups (hydroxyphenyl) which can be reacted with selected curing agents and monomeric or polymeric materials containing reactive constituents. Blends of this resin with phenolics, polyesters, epoxies, polyamides, and many elastomers can be prepared. The cured resin or resin-polymer blends are reported to have flame resistance, high heat stability, high structural strength, and excellent environmental resistance.

Several of the phosphonitrillic chloride polymeric blends were irradiated both at room temperature and at elevated temperatures. A blend of the AP-Resin-XHU and acrylonitrile was prepared, irradiated in air, and immersed in 5P4E polyphenyl ether (Monsanto OS-124) at temperatures ranging from 75 to 340 F.

In general, exposure of the blend to elevated temperatures and radiation resulted in an increase in tensile strength, elastic modulus (compression), and hardness. However, elongation decreased from 138 per cent to 94 per cent when the blend was heated to 110 F for 33 hours with no irradiation. When irradiated to 2.5 x $10^{10}$ ergs $g^{-1}$(C) in air at this same temperature, elongation decreased to 26 per cent. Immersed in the polyphenyl ether for 33 hours at 110 F with no irradiation, elongation was 50 per cent; after irradiation to 2.1 x $10^{10}$ ergs $g^{-1}$(C) in the oil at 110 F (33 hours), elongation was 30 per cent. A glass cloth laminate of this resin showed excellent radiation stability at 455 F.

### Polyamide (Nylon)

Koehler and Pefhany(6) reported that Nylon Zytel 33, when irradiated to 2 x $10^{10}$ ergs $g^{-1}$(C) in a dry atmosphere, was satisfactory and could be used in a gaging system for reactor pressure tubes designed to measure surface defects during periods of reactor shutdown. A nylon ring used in this equipment was also satisfactory, although its color changed to a brown.

## Polyimide

Stephenson(17) irradiated polyimide fibers, HT-1 (Du Pont), polybenzimidazole, and thiazole polymers with ultraviolet radiation. No volatile products were detected and no differences in degradation were noted between irradiations in air, nitrogen, or vacuum ($10^{-6}$ torr). Tensile strength decreased from $1.1 \times 10^5$ psi to $1 \times 10^5$ psi, while elongation decreased from 30 per cent to about 20 per cent in these environments. Irradiation of polybenzimidazole fibers with 253.7 mμ of light from a G30T8 lamp produced greater deterioration of tensile properties in oxygen than in nitrogen or in vacuum. Irradiation in nitrogen produced effects in elongation that were intermediate between those in oxygen and in vacuum. Fibers of the thiazole polymer appeared not to be affected differently in nitrogen, oxygen, or vacuum. No loss of tensile strength due to X-irradiation was apparent, but some decrease in elongation was noted.

Du Pont "H" film, considered for use as a hydrogen barrier, was irradiated (nuclear) while immersed in liquid nitrogen.(4) At this temperature, tensile and tear strengths of a 2-mil sheet were approximately equivalent to those of 2-mil Mylar. These properties were not significantly affected by exposure to $2 \times 10^9$ ergs $g^{-1}$(C). Radiation did not affect hydrogen permeability.

## Polycarbonate

Merlon polycarbonate plastic, when irradiated at 75 F to $3.5 \times 10^{10}$ ergs $g^{-1}$(C) in air, showed no appreciable change in hardness.(7) Its ultimate strength decreased from 8590 psi to 2070 psi. At $10^{10}$ ergs $g^{-1}$(C) elongation decreased from 104 per cent to 54 per cent. At $2 \times 10^{11}$ ergs $g^{-1}$(C) the material was too brittle to determine these properties.

## Poly-n-Vinyl Carbazole

Grinlan F plastic (poly-n-vinyl carbazole)(7) showed extremely good radiation resistance at room temperature. When irradiated to $2 \times 10^{11}$ ergs $g^{-1}$(C), there was no appreciable change in hardness, specific gravity, or tensile strength. Values for the last property before and after irradiation were 2900 psi and 2590 psi, respectively.

## Polystyrene

Lewis(7) irradiated polystyrene film at 75 F to $9.4 \times 10^{10}$ ergs $g^{-1}$(C). Tensile strength changed from 1120 psi to 512 psi. Ultimate elongation changed from 6.5 per cent to 3.2 per cent.

## Polyethylene and Polypropylene

Kerlin and Smith(1) found that Marlex 6002 (a high-density polyethylene), when irradiated in air to $10^9$ ergs $g^{-1}$(C), decreased in elongation from 907 per cent to 14 per cent. However, in vacuum, the decrease was only to 675 per cent. Tensile strength increased both in air and in vacuum; the increase was slightly higher in vacuum.

Lewis(7) reported data on a propylene-ethylene polyallomer produced by Eastman Chemical Products Inc. Polyallomers are defined as crystalline thermoplastic polymers produced from two or more different monomers. These are not copolymers in the usual sense nor are they blends, but are more like block polymers. Irradiated to $9.4 \times 10^{10}$ ergs $g^{-1}$(C) at 75 F, tensile strength decreased from 4380 psi to 1100 psi. Above $2.8 \times 10^{10}$ ergs $g^{-1}$(C), hardness decreased and the material became very tacky. Elongation decreased considerably between $3 \times 10^8$ and $10^9$ ergs $g^{-1}$(C). At 250 F, when irradiated to $2.4 \times 10^{10}$ ergs $g^{-1}$(C), the specimens were stuck to the foil wrapper and tore easily. At 200 F, tensile strength decreased from 4390 psi to 1300 psi at $2.9 \times 10^9$ ergs $g^{-1}$(C), while elongation decreased from 688 per cent to 34 per cent at $9.7 \times 10^8$ ergs $g^{-1}$(C).

## Polyurethane (Rigid Foams)

The effect of nuclear-radiation exposure at cryogenic temperatures was examined on four polyurethane foams by Yasui(4). The materials were Magnolia Foam, Marfoam, CPR 20-3 Foam, and Douglas Insulation. There was no statistically significant difference between the controls and irradiated specimens of Magnolia Foam or Marfoam. CPR-20-3 increased about 39 per cent in shear strength in the anisotropic direction. Yasui accounts for this by the fact that the individual cells within the foam were elongated in this direction and were mutually parallel. Radiation did not affect compressive properties.

## Programs in Progress*

Current programs on radiation effects in polymers are summarized below.

Aerojet General Corporation is studying the effects of the space environment on the compatibility of sealant materials and propellants.

Douglas Aircraft Company is devoting part of its program to a study of elastomers with high thermal-radiation reflectance properties as a protection against the environment of a nuclear explosion.

General Dynamics/Fort Worth is investigating the behavior of various polymers in complex environments.

Ion Physics Corporation is studying the development of polymer coatings which have greater strength and durability following electron irradiation.

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*Detailed information on these programs is given in REIC Memorandum 13-C, "Compilation of Nuclear, Pulsed, and Space Radiation Effects Projects" (U). Confidential.

The Johns Hopkins University is determining the behavior of common dielectrics at high frequencies in a high-vacuum environment.

Lockheed Aircraft Corporation is studying the optical-property behavior of thermal-control coatings by irradiating them with hydrogen plasma to simulate an interplanetary environment.

Lockheed Aircraft Corporation is also evaluating the effects of trapped-particle radiation on the thermal radiative properties of coatings and the synergistic effects of electrons, ultraviolet irradiation, and vacuum on the optical properties of thermal-control materials.

NASA, Langley Research Center has an internal program to study the effects of electron irradiation in air and in vacuum on a foil composed of aluminum and Mylar film.

NASA, Langley Research Center, in another internal program, is investigating the effects of electron irradiation on the tensile properties of polyurethane, polysulfide, and PBAA propellants. Gaseous products are also being studied.

The U. S. National Bureau of Standards is conducting research on the degradation mechanisms of polymers, the kinetics of these reactions, and the chemical structure and radiation stability of polymers exposed to radiant energy.

The U. S. Navy, Naval Air Engineering Center is studying the viscosity and UV transmission of nylon 66 after exposure to nuclear, ultraviolet, and thermal radiation.

The U. S. Navy, Naval Air Engineering Center is investigating the thermal radiation resistance of nonmetallic materials.

NRA, Inc., is studying the effects of radiation on polymers.

The Pennsylvania State University is determining the effects of radiation on the physical properties of various polymers.

The Pennsylvania State University is devoting part of its program to a study of the effects of radiation on the dynamic properties of polymers.

Radiation Applications is studying the upgrading of polyethylene and ethylene-propylene copolymers using radiation cross-linking and the improvement of low-temperature flexibility.

Sandia Corporation is determining the effect of neutron and gamma irradiation on the photoconductivity of organic dielectrics.

The University of Georgia is investigating the effects of neutron irradiation on the molecular structure of plastics and elastomers.

## Conclusions and Recommendations

Investigations continue to be directed toward the behavior of components in a space environment. The radiation levels are in general lower than those encountered with nuclear-propelled vehicles, and many of the elastomers and plastics are proving to be satisfactory. There have been some new materials developed which appear promising for nuclear and space applications. These include the polyimide polymers, the phosphonitrillic chloride polymers, and styrene-butadiene, neoprene, and nitrile elastomers containing antirads.

At the present time, on the basis of available information, it is not possible to generalize regarding the effect of radiation in air as compared with the effect in vacuum on polymeric materials. With some, radiation causes greater degradation in vacuum than in air; with others, the reverse is true.

The REIC is continually receiving requests for information on the behavior of commercial materials at high-radiation exposures. In this respect, it is believed that many of the Government contractors have tested various products and components, but the results have not been published. It would be an advantage if these data could be collected and processed into the REIC files. It could save duplication of efforts.

Work should be increased in fundamental studies directed toward determining damage mechanisms leading to the development of materials having improved radiation resistance.

Some confusion has arisen over the use of the term ambient temperature. It is recommended that the actual temperature range be given in future reports rather than the indefinite reference, ambient.

The REIC is preparing an addendum to REIC Report No. 21, dated September 1, 1961, on "The Effect of Nuclear Radiation on Elastomeric and Plastic Components and Materials". The addendum will contain more information on space environmental effects. This publication should be available about September 1.

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# SECTION C

## LUBRICANTS, FLOTATION AND HYDRAULIC FLUIDS

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# LUBRICANTS, FLOTATION AND HYDRAULIC FLUIDS

## Current State of the Art

Contract efforts in the area of lubricants and hydraulic fluids continued at a low rate of effort. The efforts were concentrated on materials for potential use in space. The environments of interest included vacuum and cryogenic to extremely high temperatures, besides the radiation environment.

Research on radiation effects on fuel materials during the report period was nil. A Soviet study describes the radiation-induced thermal cracking of gasoline at 450 to 600 C under various pressures during exposure to gamma radiation at a rate of $1.5 \times 10^5 \pm 10\%$ ergs $g^{-1}$(C) $hr^{-1}$.(1)* A handbook by Bolt and Carroll reviews the radiation-effects information on fuel materials which has been gathered over the last few years.(2)

## Flotation Fluids

Sperry Gyroscope Company(3) has determined G-values for hexafluorobenzene as a part of a search for radiation-resistant gyroscope flotation fluids. This fluid has proved more resistant to gassing at gamma exposures of $10^{10}$ ergs $g^{-1}$(C) than typical aromatic fluids. $G_{polymer}$ equaled 2.01 due to cross-linking and $G_{gas}$ equaled 0.001 for hexafluorobenzene. The liberated fluorine apparently back-reacts with the fluids forming saturated alicyclics.

In an English study,(4) Fluorolube 230, a flotation and damping fluid, was irradiated in silica glass containers sealed under a vacuum of $10^{-3}$ mm Hg. Samples were exposed to gamma radiation of $5 \times 10^9$ ergs $g^{-1}$(C). The viscosity decreased less than 10% at the lowest exposure. The samples did not discolor due to the irradiation. The amount of gas evolved was not measured; however, the amount was appreciable. The gas was acidic and probably consisted of hydrogen fluoride which reacted with the silica container to form water and silicon tetrafluoride gas. Further study is expected to be done on this fluid to determine the exact amount of gas evolved and its nature, since gas-bubble formation is a critical property for gyroscope uses.

## Lubricant and Hydraulic Fluids

Studies on mix bis(mix-phenoxyphenoxy) benzene (Mixed-5P4E) have continued at the Nuclear Aerospace Research Facility at General Dynamics/Fort Worth.(5,6) Three samples of oxidation-inhibited Mixed-5P4E were compared to the uninhibited oil. The inhibitors used were bis(p-phenoxyphenyl) diphenyltin, tetraphenyltin, and cobalt bis(N-phenyl-5-nitrosalicylimine). Samples were irradiated to a gamma exposure of $2.8 \times 10^{10}$ ergs $g^{-1}$(C) and an integrated fast-neutron flux of $6.8 \times 10^{15}$ n $cm^{-2}$ (E>2.9 Mev), at temperatures of 550 or 600 F. Data from irradiation runs at both temperatures indicate that Mixed-5P4E approaches its useful upper limit as a high-speed bearing lubricant in this temperature range at a gamma exposure of $2 \times 10^{10}$ ergs $g^{-1}$(C),

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*References are at end of Section C.

plus associated neutrons. When test temperatures are increased to 700 F, the maximum permissible exposure is reduced by approximately one order of magnitude. The primary cause of bearing failure was a heavy cokelike deposit on the bearing races and separators. These deposits were apparently caused by the coking out of thermally unstable products formed by radiolytic and oxidative processes. The cobalt bis(N-phenyl-5-nitrosalicylimine) was found to be unsatisfactory for use as a Mixed-5P4E antioxidant in this environment, based primarily on oxygen-absorption data. Both tetraphenyltin and bis(p-phenoxyphenyl) diphenyltin were effective as oxidation inhibitors and will be examined further in future tests. Inhibitor-concentration studies are to be continued; however, a tetraphenyltin concentration of 0.5% appears to have an "antirad" effect during irradiation. A concentration of 1.5% appears to be excessive, and the solubility characteristics of tetraphenyltin at this concentration were undesirable.

Two patents have been issued which describe radiation-resistant lubricants. The first, issued to British Petroleum Company, Ltd.(7), describes a polyphenyl ether of the general formula $PhO(C_6H_4O)_nPh_3$ (n=1-4), treated with an alkyl halide or an olefin. The product had a viscosity of 5.2 centistokes at 140 F. The 140 F viscosity increased 93% after an exposure to radiation of 2 x $10^{11}$ ergs $g^{-1}$(C). The second patent issued to Standard Oil Company of Indiana(8) describes a machine lubricant which showed a decrease in viscosity when irradiated to a gamma exposure of 8.7 x $10^9$ ergs $g^{-1}$(C) at ambient temperature. The lubricant which is not subject to polymerization when exposed to gamma irradiation is prepared using 25% or more of a hydrogenated polybutene oil plus the base stock lubricant.

Evaporation data were given at the 1963 Annual American Chemical Society Meeting(9) on nine promising space lubricant fluids in tests conducted at 140 to 160 F in a vacuum of $10^{-5}$ torr. Samples were not exposed to radiation in this study. The fluids tested were a modified diester, paraffinic petroleum, polyalkylene glycol, petroleum bright stocks (viscosity: 235 centistokes at 100 F and viscosity: 374 centistokes at 100 F), phenyl methyl polysiloxane, chlorophenyl methyl polysiloxane, straight-chain dimethyl polysiloxane, and branched chain dimethyl polysiloxane.

## Solid-Film Lubricants and Greases

The wear-life properties of three solid-film lubricants, irradiated to a gamma exposure of 1.7 x $10^{11}$ ergs $g^{-1}$(C) and integrated neutron fluxes of 3.7 x $10^{16}$ n $cm^{-2}$ (E>2.9 Mev) and 3.1 x $10^{15}$ n $cm^{-2}$ (E>0.48 ev), were studied by General Dynamics/ Fort Worth.(5) Samples were tested at temperatures of 850-1000 F for Lubricant A; 80 F, 850-900 F, and 1150 F for Lubricant B; and 80-170 F, 600 F, and 850-900 F for Lubricant C. Wear testing was conducted using the Hohman A-6 tester. A load of 110 lb per rub shoe was applied and a sliding speed of 355 rpm (128 ft/min) was maintained. Lubricant A ($PbS + MoS_2 + B_2O_3$) showed no significant effects on wear life as a result of the radiation exposure. There was a significant radiation-induced decrease in wear life of Lubricant B (Dynalube, a silver-matrix electrodeposited film formulation) at the 900 F test temperature. At the higher temperature, however, the lubricant performed exceptionally well. The lubricating properties of Lubricant C (Almosol SFD-560, PbO + $MoS_2$ + Modified Silicone Resin + Proprietory Lube Pigment) were improved by irradiation at the low temperature and unaffected at the 600 F test temperature. At 900 F its lubricating properties were poor as expected since it is resin bonded. Wear life of Lubricant C was significantly reduced at 900 F. In an earlier General Dynamics study(6), a solid-film lubricant, $MoS_2$ + graphite + sodium silicate, was exposed to the same reactor exposures as above and tested for wear life over a wide range of temperatures. The film had good lubricating properties under all test conditions, and the reactor irradiation had no valid effect on wear life. The wear life at 600 F was approximately 20% better than at 1200 F.

The effects of gamma radiation on the wear life of Electrofilm Lube-Lok's 77-S, 4396, 66-C, and 1000 has been determined.(10) The wear life of 77-S and 1000 was unaffected by gamma exposures from $8.7 \times 10^8$ ergs $g^{-1}$(C) to $8.7 \times 10^{10}$ ergs $g^{-1}$(C) at 400 and 550 F. After an exposure of $8.7 \times 10^9$ ergs $g^{-1}$(C), the wear life of 77-S actually increased by 30%. Wear life of 77-S was also unaffected by exposure to neutron radiation of $3 \times 10^{14}$ nvt to $3.2 \times 10^{16}$ nvt. Wear life of 66-C was increased and wear life of 4396 was unaffected after an exposure of $4.4 \times 10^9$ ergs $g^{-1}$(C).

A lubricating grease for nuclear-reactor parts is described by a patent assigned to Esso Research and Engineering Company.(11) The grease, made from 67.7% paraffinic bright stock, 8.2% of Penzoil resin, 23.2% of acetylene black and 0.9% of phenyl alpha naphthylamine, had a micropenetration of 108 which changed to 109 after exposure to radiation of $10^{10}$ ergs $g^{-1}$(C).

Other studies have been conducted on solid-film lubricants for use in the space environment; however, testing in a radiation environment has not been included. Outgassing characteristics of 11 plastic and carbon compositions, ten powders, and six composites have been tabulated in a vacuum of $10^{-6}$ mm Hg and at test temperatures from 160 F to the point of thermal degradation for the plastic material, and from 760 to 1160 F for the powders and composites.(12) Of the plastic and carbon compositions, polytetrafluoroethylene, polytetrafluoroethylene with glass-cloth filler, polychlorotrifluoroethylene, and carbon-graphite with PTFE impregnant gave the lowest amount of gas evolution up to 750 F. Antimony trisulfide, silver iodide, and tungsten diselenide had the least amount of gas evolution of the dry powders before bake-out. After bake-out for 24 hours, molybdenum disulfide, graphitic carbon, antimony trisulfide, gallium telluride, tungsten diselenide, and molybdenum diselenide all exhibited low gas-evolution rates up to 1160 F.

Bearing and gear materials for use in the vacuum environment of space of $10^{-9}$ torr and below are being studied.(13) Metal or plastic materials impregnated with solid films such as $MoS_2$, $WS_2$, $CaF_2$, and BN have been used. Ball retainers made of Teflon and glass fiber with molybdenum disulfide have been favorable. The use of platings such as gold and silver as solid dry lubricants have encountered difficulties in obtaining adherent coatings particularly on stainless steel races.

In another study(14) for potential use in the space environment, metal matrix composite materials of Ag-PTFE-$WSe_2$ were successfully used in a vacuum at pressures from $1 \times 10^{-6}$ to $8 \times 10^{-9}$ torr and temperatures from -160 F to 300 F to lubricate 20-mm-bore ball bearings under moderate loads and speeds, and to lubricate 35-mm-bore ball bearings and 6-inch gears under heavy loads and low-speeds for extended periods of time. Bearing starts were made at temperatures as low as -320 F for the 20-mm size and -188 F for the 35-mm size.

In a review of space-lubrication problems(15), the properties of Teflon and polyimides are discussed as solid-film lubricants for bearing systems. Even though these films are more stable in the vacuum, ultraviolet, and ionizing radiations in the space environment than conventional oils and greases, vibration will pose a serious problem to bearings lubricated with solid films since the continuous films are prone to flake or break away from the bearing surface.

Metallic-film lubrication of ball bearings have been tested for use in the vacuum and radiation environments of space. Bearings were tested in small 10,000-rpm motors in a multiport, oil-free vacuum system. Retainers of "S" Inconel and silver plated Circle "C" were outstanding and were capable of about a 1000-hour life when used with gold-plated balls and races.(16)

Six molybdenum disulfide based lubricant films provided very effective lubrication when exposed to a vacuum of $10^{-7}$ to $10^{-9}$ torr for 60 to 270 days.(17) There was no indication of failure during the testing. Compositions of the lubricants were silicate-bonded $MoS_2$, epoxy-bonded $MoS_2$, resin-bonded $MoS_2$, $TiO_2$-bonded $MoS_2$, rubbed-on $MoS_2$, and an $MoS_2$-containing vapor-deposited film. PTFE-based compositions and metal-film lubricants were less effective than the molybdenum disulfide lubricants under the test conditions.

## Programs in Progress*

The following organizations are engaged in active radiation-effects studies on lubricants and hydraulic fluids:

General Dynamics/Fort Worth is continuing its study of combined environments on lubricants for ball bearings in servo electric motors.

Douglas Aircraft Company is studying friction, wear, lubrication, and cold-weld tendencies of materials exposed to the vacuum, heat, and ultraviolet radiation environments of space.

General Dynamics/Fort Worth is determining the effects of reactor radiation on the wear life of five promising high-temperature solid-film lubricants for use under heavy sliding loads.

Lockheed Missiles and Space Company is continuing its internal evaluation of lubricants using R-3 size bearings operating at 8000 rpm in vacuum with a gamma exposure rate of $3.6 \times 10^5$ ergs $g^{-1}$(C).

Sperry Gyroscope Company is conducting an internal program to develop radiation-resistant gyroscope damping fluids and lubricants.

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*Detailed information on these programs is given in REIC Memorandum 13 C, "Compilation of Nuclear, Pulsed, and Space Radiation Effects Projects" (U), Confidential.

## Conclusions and Recommendations

Research on lubricants remained at a low rate of effort during the past year. The polyphenyl ethers, especially Mixed 5P4E, are considered the best high-temperature, radiation-resistant fluid lubricants. For flotation and hydraulic uses in high-radiation environment, hexafluorobenzene is good; however, gassing is a definite problem and the high cost prohibits its use in some systems. Systems requiring the use of a conventional grease during radiation exposure are still desirous of more radiation-resistant greases. Cal Research NRRG No. 335 is still promising. Solid-film lubricants, especially the molybdenum disulfide based lubricants are proving favorable not only under radiation exposure, but also in the extreme environments of space. Solid-film lubricants are not usable in many systems; thus there is continuing need for a breakthrough in the area of fluid lubricants for the combined environments of nuclear radiation, extreme temperatures, and vacuum.

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# SECTION D

# CERAMICS

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# CERAMICS

## Current State of the Art

Interest in ceramics for nuclear applications continues at a slightly lower level than last year, as indicated by fewer contracts in this area. Ceramic fuel-materials interest, not covered in this report, remains high.

### Ceramic Oxides

The magnetic properties of hematite irradiated to a fast-neutron exposure of $5 \times 10^{17}$ n cm$^{-2}$ at 250 C were studied by Ogilvie(1)*. Although only small changes occurred in the magnetic properties after exposure to $6 \times 10^{16}$ n cm$^{-2}$, pronounced differences occurred at $5 \times 10^{17}$ n cm$^{-2}$. Table D-1 summarizes the results.

TABLE D-1. SPONTANEOUS MOMENT, REMANENCE, AND COERCIVE FORCE(1)

| Direction in Crystal | Temperature, K | Flux, n cm$^{-2}$ | Spontaneous Moment, emu/g | | Remanence, emu/g | Coercive Force, oe |
|---|---|---|---|---|---|---|
| [00.1] | 77 | 0 | 0.086(a) | 0.091(b) | 0.071 | 439 |
| | | $6 \times 10^{16}$ | 0.083 | 0.083 | 0.070 | 526 |
| | | $5 \times 10^{17}$ | 1.01 | 0.94 | 0.465 | 702 |
| | 273 | 0 | | 0.10 | 0.10 | |
| | | $6 \times 10^{16}$ | | 0.10 | 0.10 | |
| | | $5 \times 10^{17}$ | 0.98 | | 0.40 | 526 |
| [11.0] | 350 | 0 | | 0.42 | | |
| | | $5 \times 10^{17}$ | 1.32 | | 0.63 | 386 |

(a) Obtained from hysteresis curves.
(b) Obtained from magnetization curves.

In a General Electric investigation(2), it was determined that a single crystal of $UO_2$ irradiated to $1.4 \times 10^{20}$ fissions/cm$^3$ did not change in appearance. The hardness of the specimen increased significantly, and the melting point increased from 2790 C to 2840 C.

Hentz(4) reported that following irradiation to an exposure of $5.3 \times 10^{21}$ ev at 36 C, silica-alumina turned a very dark color and no gas was evolved. After the introduction of isopropylbenzene, decolorization occurred.

An investigation of the effect of $5 \times 10^{14}$ fissions/cm$^3$ on $UO_2$ was conducted by the Oak Ridge National Laboratory(3). Although no structural damage was evident at this level, a volume decrease did occur when a $UO_2$ crystal was irradiated in air in a polyethylene capsule and caused microcracking. A tentative explanation of these observations is that organic products released from the polyethylene react with the $UO_2$ to form UC.

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*References are at end of Section D.

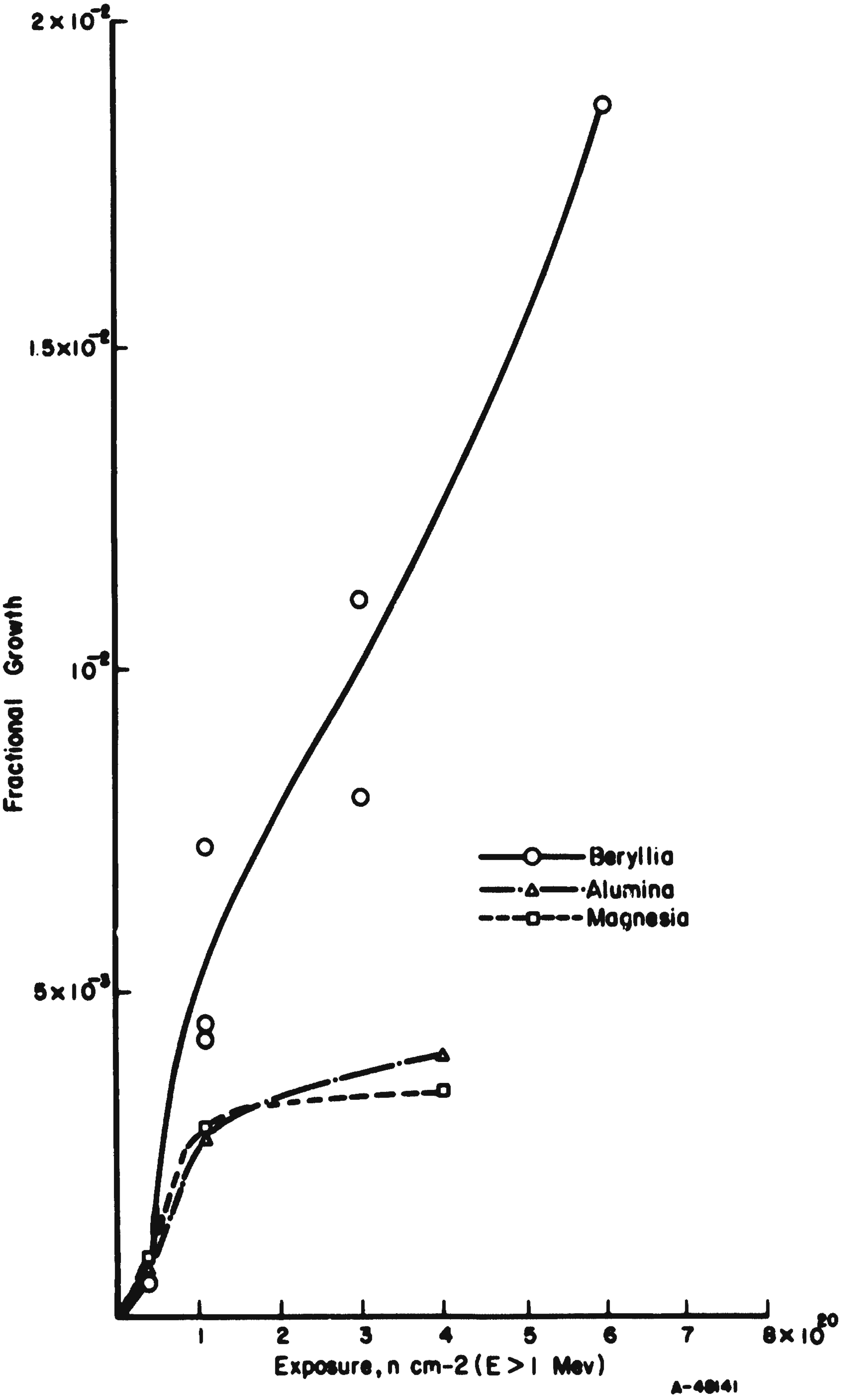

FIGURE D-1. GROWTH IN BERYLLIA, MAGNESIA, AND ALUMINA ON IRRADIATION AT ABOUT 150 C, AS A FUNCTION OF EXPOSURE(6)

Samples of alumina and forsterite were irradiated to a pulsed neutron exposure of $2.0 \times 10^{20}$ n $cm^{-2}$, while others received a total gamma exposure of $5.1 \times 10^{7}$ ergs $g^{-1}$(C). Linden and Lascaro(5) found that there was no significant change in the dielectric constant, dissipation factor, and volume surface resistivity in either group.

Desport and Smith(6) reported an investigation of the dimensional changes of alumina irradiated to a fast-neutron exposure of $4 \times 10^{20}$ n $cm^{-2}$ at <150 C. Figure D-1 shows the results.

## Magnesium Oxide

Magnesia specimens were irradiated at $1.1 \times 10^{20}$ n $cm^{-2}$, E > 1 Mev, at 150 C to determine the growth and annealing.(6) The specimens were annealed at temperatures up to 1800 C. The damage as indicated by growth was only about 80 per cent annealed out despite the high annealing temperature. The growth is shown as a function of exposure in Figure D-1.

A study of the change in catalytic activity of ultraviolet- and neutron-irradiated MgO was undertaken by Lunsford and Leland(7). The results of this study are summarized in Table D-2.

TABLE D-2. SUMMARY OF CATALYTIC CHANGES OF MgO(7)

| Thermal Activation 2 Hr at Indicated Temperature, C | Type of Irradiation | Change in Activation Energy | Change in Pre-Exponential Factor |
|---|---|---|---|
| 291 | Neutron | Increases | Large increase |
| 291 | Ultraviolet | No change | Large increase |
| 372 | Neutron | No change | Small increase or decrease |
| 520 | Neutron | Increases | Little change |

In a report by Hoigne and Ballantine(8), the activity of magnesium oxide catalysts increased after an exposure of $2 \times 10^{10}$ ergs $g^{-1}$(C) at -78 and 25 C in vacuum and in hydrogen atmospheres. The results, which were not affected by the environment, indicated that the physical absorption characteristics did not change.

Charman and Dell(9) of the Atomic Energy Research Establishment (AERE) investigated the surface reactivity of nickel oxide and magnesium oxide after fast-neutron irradiation to an exposure of $7.8 \times 10^{19}$ n $cm^{-2}$ at 25-40 C. The chemisorption of O and H was greatly enhanced following irradiation. The calculated number of displacements in the surface region of the oxide lattice leads to values of the same order of magnitude as the observed number of gas atoms chemisorbed.

In another experiment on magnesium oxide at the AERE(10), crystals were irradiated to an exposure of $3 \times 10^{19}$ fast n $cm^{-2}$. Damage in the form of dislocation loops occurred which was partially responsible for the macroscopic growth. At an exposure of $4 \times 10^{20}$, the damage was extremely resistant to annealing.

Groves and Kelly$^{(11)}$ investigated the microstructural damage in MgO after irradiation to a neutron exposure of $4 \times 10^{19}$ n cm$^{-2}$ at <200 C. The length of the slip bands from a microhardness indent became considerably compressed, although the microhardness only increased 10%. Upon annealing the crystals coarsened and then returned to normal. After irradiation at 650 C to an exposure of $5.4 \times 10^{19}$ n cm$^{-2}$, dislocation loops occurred but were removed by annealing.

Sambell and Bradley of AERE$^{(12)}$ irradiated magnesium oxide single crystals in an exposure range of $2.4 \times 10^{19}$ to $1.7 \times 10^{20}$ n cm$^{-2}$ at 150 C. No plastic flow was evident in the fractured specimens, even in the vicinity of the fracture itself. It was concluded that fracture resulted from pits produced by polishing or by impurities within the crystal.

## Beryllium Oxide

M. Salesse$^{(13)}$ studied the effect of neutron irradiation to an exposure of $2.2 \times 10^{20}$ n cm$^{-2}$ at less than 100 C and subsequent annealing on BeO. Bubbles of helium occurred in the grain joints and measurements of the specific heat of BeO confirmed that the gas did evolve as a result of the irradiation and annealing.

General Electric Company, Nuclear Materials and Propulsion Operation$^{(14)}$, continued its study of radiation effects in AOX, UOX, and Minox AAA-grade BeO. The results of their program established that the best stability is obtained with $\leqslant$5-micron grain-size material. The $\leqslant$5-micron grain-size materials exhibit both smaller expansion and greater strength retention than materials with large grain sizes in exposures up to $\sim 1.8 \times 10^{21}$ n cm$^{-2}$ ($E \geqslant 1$ Mev).

Expansion is the principal effect of irradiation in BeO. At 100 C irradiations, the expansion in specimens of 3- to 20-micron grain sizes occurs at a rate of 0.2 to 0.3 per cent $\Delta V/V$ per $10^{20}$ n cm$^{-2}$ ($E \geqslant 1$ Mev) up to about $1.7 \times 10^{20}$ n cm$^{-2}$, and then increases to a rate of approximately 1 per cent $\Delta V/V$ per $10^{20}$ n cm$^{-2}$. The expansion consists of volume components from lattice expansion and from cracks which form at grain boundaries as a result of greater expansion in the crystallographic c-axis than in the a-axis. The expansion due to grain-boundary cracks increases with increasing exposure and is 2 to 3 times as large as that from lattice expansion at exposures from approximately 4 to $10 \times 10^{20}$ n cm$^{-2}$.

The components of volume expansion in BeO irradiated at elevated temperatures are given in Table D-3.

Grain-boundary cracking, according to the General Electric Company investigation, is influenced by the grain size and density of the material and by the irradiation temperature. Cracking occurs at volume expansion in the range of approximately 0.4 to 1.2 per cent. In 1000 C irradiations, it occurs at the highest exposure, approximately 1.5 to $2.5 \times 10^{20}$ n cm$^{-2}$, in specimens with grain sizes of 20 microns or less, and it appears to occur at lower exposures in a given grain size the lower the specimen density. In elevated-temperature irradiations, it occurs at higher exposures the higher the temperature. Strength, elastic constants, and the thermal expansion of BeO are essentially unchanged if grain-boundary cracks are avoided. The strength changes, once cracks have formed, are less severe in specimens of small (<5 microns) grain size.

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TABLE D-3. COMPONENTS OF VOLUME EXPANSION IN BeO IRRADIATED AT ELEVATED TEMPERATURES(14)

| Sample | | Temperature, C | Flux, $10^{14}$ nv (> 1 Mev) | Neutron Exposure, $10^{20}$ n cm$^{-2}$ (> 1 Mev) | Lattice Expansion, % | | | Macroscopic Volume Increase, % | Volume Increase Due to Porosity[b], % | Surface-Connected Porosity, % of total volume |
|---|---|---|---|---|---|---|---|---|---|---|
| Composition[a] | Grain Size, microns | | | | $\Delta a/a$ | $\Delta c/c$ | $\Delta V/V$ | | | |
| AOX | 12 | 1000 | 2.0 | 2.4 | ~0 | 0.04 | 0.04 | 0.63 | 0.2 | <0.1 |
| AOX | 20 | 1030 | 1.5 | 3.5 | ~0 | 0.04 | 0.04 | 0.73 | 0.3 | <0.1 |
| AOX | 44 | 1000 | 1.7 | 4.1 | ~0 | 0.06 | 0.06 | 0.98 | 0.2 | <0.1 |
| AOX | 17 | 800 | 1.9 | 8.6 | 0.04 | 0.25 | 0.33 | 2.0 | 0.8 | 2.7[c] |
| AOX | 17 | 600 | 1.8 | 8.3 | 0.07 | 0.81 | 0.95 | 2.9 | 1.0 | 3.6[c] |
| UOX-0.5 MgO (wt %) | 20 | 970 | 1.6 | 3.8 | | | | 0.60 | | |
| UOX-0.5 MgO (wt %) | 12 | 940 | 2.4 | 10.8 | | | | 0.92 | | |

(a) The density of the samples was 2.9 g/cm$^3$.

(b) Values are the difference between macroscopic and true volume increases.

(c) These values indicate appreciable grain-boundary cracking.

Figure D-1 shows the growth of beryllia irradiated from 1.1 to 5.5 x $10^{20}$ n $cm^{-2}$.(6)

Beo was irradiated by the Austrailian Atomic Energy Commission Research Establishment(15) at temperatures between 60-100 C and 500-700 C to exposures of 3.5-6.5 x $10^{20}$. All specimens turned gray and microcracking occurred. The dimensions and volume of the samples were increased, while the thermal conductivity decreased.

In later studies by Hickman and Walker(16) of the Austrailian Atomic Energy Commission, BeO samples were subjected to a slightly higher neutron exposure range of 1 x $10^{20}$ to 1 x $10^{21}$ n $cm^{-2}$ at 75-100 C. The coarse-grained, hot-pressed material exhibited extensive intergranular cracking at 2.5 x $10^{20}$ n $cm^{-2}$ and powdered at 5 x $10^{20}$ n $cm^{-2}$. The cold-pressed, sintered, coarse-grained sample showed both intergranular and transgranular microcracking at an exposure of 5 x $10^{20}$, although no microscopic evidence appeared in any of the cold-pressed samples at $10^{21}$ n $cm^{-2}$. The fine-grained, cold-pressed, sintered material did not show microcracking at $10^{21}$ n $cm^{-2}$. The lattice parameter changes in three of the samples are summarized in Table D-4.

TABLE D-4. LATTICE PARAMETER CHANGES IN BERYLLIUM OXIDE IRRADIATION AT 5 x $10^{20}$ n $cm^{-2}$(16)

| Powder Source | Fabrication Method | Density, % of theoretical | Grain Size, (μ m) | Condition | c Parameter Change, % | a Parameter Change, % |
|---|---|---|---|---|---|---|
| Pechiney | Hot pressed at 1750 C for 1/2 hr at 1 tsi | 96-98 | 10-20 | Powdered | 1.6 ± 0.2 | 0.105 ± 0.005 |
| Brush UOX, preground | Cold pressed at 20 tsi Sintered at 1500-1550 C | 95-96 | 2-3 | Solid | 0.5 ± 0.1 | 0.14 ± 0.01 |
| Brush UOX, preground | Cold pressed at 20 tsi Sintered at 1500-1550 C | 95-96 | 2-3 | Crushed | 1.4 ± 0.1 | 0.10 ± 0.01 |

A study was conducted at the Oak Ridge National Laboratory(17) on beryllium oxide irradiated to a neutron-exposure range of $10^{20}$-2.6 x $10^{20}$ fast n $cm^{-2}$, at temperatures from 100 - 1100 C. Those specimens irradiated at the high exposures and low temperatures showed the greatest damage, and fractures were observed in samples irradiated to high exposures and 800 to 950 C. The thermal conductivity of BeO decreased. Annealing of the damage was also observed.

An investigation by the Oak Ridge National Laboratory(18) confirmed the fact that high exposures of fast neutrons severely damages beryllium oxide. At an exposure of 1 x $10^{21}$ n $cm^{-2}$, serious circumferential fracturing ensued. In another group of specimens irradiated to 3.65 x $10^{21}$ fast n $cm^{-2}$ at 583-1100 C, 35 of the 48 samples remained intact, but increased in volume from 0.9 to 4.4%.

In a third Oak Ridge investigation(19), beryllium oxide was irradiated to an exposure of 1.8 x $10^{17}$ n $cm^{-2}$ at <100 K. Figure D-2 indicates the increase in the thermal resistivity of the oxide. It was concluded that the defects produced in BeO are mobile at temperatures below room temperature.

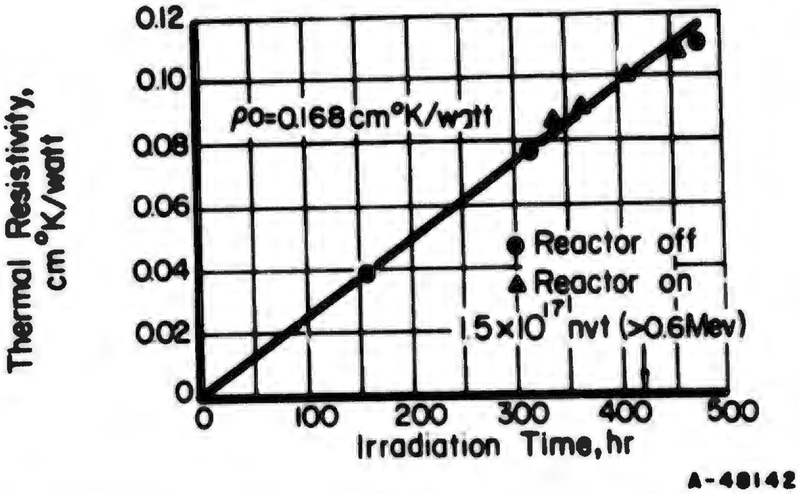

FIGURE D-2. INCREASE IN THERMAL RESISTIVITY VERSUS IRRADIATION TIME

## Glasses and Silicates

Considerable research has been reported by Russian scientists on irradiation of quartz and glass. In studying the dielectric properties of irradiated quartz, Nesterov, et al.(20), found that the electrical conductivity changed from $1.5 \times 10^{-13}$ to $9 \times 10^{-15}$ $(\text{ohm-cm})^{-1}$ after a total exposure of $10^6$-$10^7$ ergs $g^{-1}(C)$ at -60 and 20 C. The dielectric loss also increased. After the termination of irradiation, however, the properties returned to their original values. The extent of the increase of tan $\delta$ during irradiation was found to depend on the structure of the material. At equal radiation exposures, tan $\delta$ of monocrystalline quartz increases much more than that for fused or fine crystalline quartz.

Starodubtsev and Vakhidov(21) exposed crystalline quartz to gamma radiation at liquid nitrogen or room temperature, and next to ultraviolet irradiation for 10 minutes. The samples were then cooled to liquid nitrogen temperature and heated to 100 C at 2.5 ± 0.3°/min. Thermally stimulated emission was observed which was associated with electron transitions between different local states.

Another group of Russians(22,23) studied the effect of neutron irradiation on the structural variations of α-quartz. The Raman spectra of the material before and after irradiation were compared. A model was proposed that accounts for the structural variation due to neutron radiation.

The dielectric constant of quartz crystals changed -1.5% and -0.7% normal to and along the optic axis, respectively, after irradiation to a neutron exposure of $2 \times 10^{19}$ n $cm^{-2}$. The refractive index changed -0.3% and the extraordinary refractory index changed -0.05%. Zubov and Grishina(24) concluded that the dielectric-constant change may be an indicator of changes in defects with exposure.

Soviet investigators(25) also studied the radiation resistance of specially formulated foam glasses. The results of irradiation with $10^{18}$ n $cm^{-2}$ fast neutrons are summarized in Table D-5. It appears on the basis of this research that glass with $TiO_2$, $ZrO_2$, and $Cr_2O_3$ additions can be produced which can be used as heat-resistant insulation in equipment operated in a gamma-neutron environment.

TABLE D-5. RADIATION RESISTANCE OF FOAM GLASSES(25)

| Batch Composition for Foam Glass | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Original Glass | | Addition | | Gassing Agent | | Bulk Density, g/cm$^{-3}$ | | Compression Strength, g/cm$^{-3}$ | |
| Type | % | Type | % | Type | % | Unirradiated | Irradiated | Unirradiated | Irradiated |
| Window | 95 | $Al_2O_3$ | 5 | SiC | 1 | 0.24 | 0.21 | 10.4 | 0.8 |
| Window | 90 | $TiO_2$ | 10 | C | 0.5 | -- | 0.33 | -- | 17.7 |
| Window | 90 | $TiO_2$ | 10 | SiC | 1 | 0.47 | 0.36 | 36.7 | 31.7 |
| Window | 90 | $TiO_2$ | 10 | $MnO_2$ | 4 | 0.61 | 0.58 | 55.0 | 38.0 |
| Window | 90 | $ZrO_2$ | 10 | C | 0.5 | 0.35 | 0.37 | 24.3 | 23.2 |
| Window | 90 | $ZrO_2$ | 10 | SiC | 1 | 0.43 | 0.44 | 51.8 | 55.2 |
| Window | 90 | $ZrO_2$ | 10 | $MnO_2$ | 4 | 0.41 | 0.40 | 15.3 | 22.8 |
| Barytes | 80 | $Cr_2O_3$ | 20 | C | 0.5 | 0.23 | 0.33 | 22.8 | 29.9 |
| Barytes | 80 | $Cr_2O_3$ | 20 | SiC | 1 | 0.52 | 0.50 | 50.5 | 48.2 |
| Barytes | 70 | $Cr_2O_3$ | 30 | SiC | 0.5 | 0.61 | 0.57 | 52.4 | 74.4 |
| Barytes | 70 | $ZrO_2$ | 30 | C | 0.5 | 0.51 | 0.89 | 36.5 | 79.3 |

A patent was issued to the compagnie de Saint-Gobain(26) for a glass (98% $SiO_2$, 1% $GeO_2$, and 1% $Na_2O$) which exhibits strong thermoluminescence after exposure to gamma or X-ray irradiation at 300 C. The formulation had a sensitivity of 50 MR for X-rays of 150 kv and was not saturated at $1.0 \times 10^5$ r.

Optical-grade Amersil fused silica was irradiated to an integrated neutron flux of $5 \times 10^{19}$ n cm$^{-2}$ by the Naval Ordnance Laboratory(27). A decrease of 85 per cent in ultrasonic loss, an increase of less than 2 per cent in the shear modulus, and a decrease of approximately 20 per cent in compressibility occurred.

Fused silica, aluminosilicate glass, medium-grade quartz, and chemically hardened glass were exposed to 0.5 to 160 Mev protons and 2 Mev electrons by Gigas, Ewbank, and Gruber.(28) The transmittance and absorbency properties were measured in order to evaluate their usefulness as spacecraft window materials. The changes in these properties of the aluminosilicate glass, Corning 1723, are revealed in Table D-6, while Figure D-3 indicates the transmittance properties of glass following proton irradiation.

TABLE D-6. POSTPROTON IRRADIATION EXAMINATION OF TYPE 1723 GLASS(a) (0.4 Mev)(28)

| Time, PDT | Sample Number | Absorbence | | | Transmittance | | |
|---|---|---|---|---|---|---|---|
| | | Unirradiated | Irradiated | Change | Unirradiated | Irradiated | Change |
| 1650 | I | 0.030 | 0.038 | -.008 | 93.4 | 91.5 | 1.9 |
| 1600 | II | 0.025 | 0.032 | -.007 | 94.4 | 92.8 | 1.6 |
| 1525 | III | 0.025 | 0.045 | -.020 | 94.4 | 90.0 | 4.4 |
| 1045 | IV | 0.002 | 0.040 | -.018 | 95.1 | 91.2 | 3.9 |
| 1615 | V | 0.035 | 0.036 | -.001 | 92.3 | 92.1 | 0.2 |
| 1545 | VI | 0.035 | 0.035 | 0 | 92.3 | 92.3 | 0 |

(a) Evaluated at a wavelength of 0.55 $\mu$.

It was concluded that GE 101 quartz was unsuitable as a spacecraft window material since irradiation causes severe discoloration, varying absorbency characteristics across the specimen, and a transmittance of 10 per cent at 0.55 microns. The Corning 1723 was also deemed unsuitable because of surface pitting and cracking. The chemically hardened glass, Corning Chemcor, and the fused silica, Corning 7940, appeared to be more radiation resistant. A composite of 0.5 inch quartz and two Corning 1723 vacuum-separated panes was recommended for a spacecraft window.

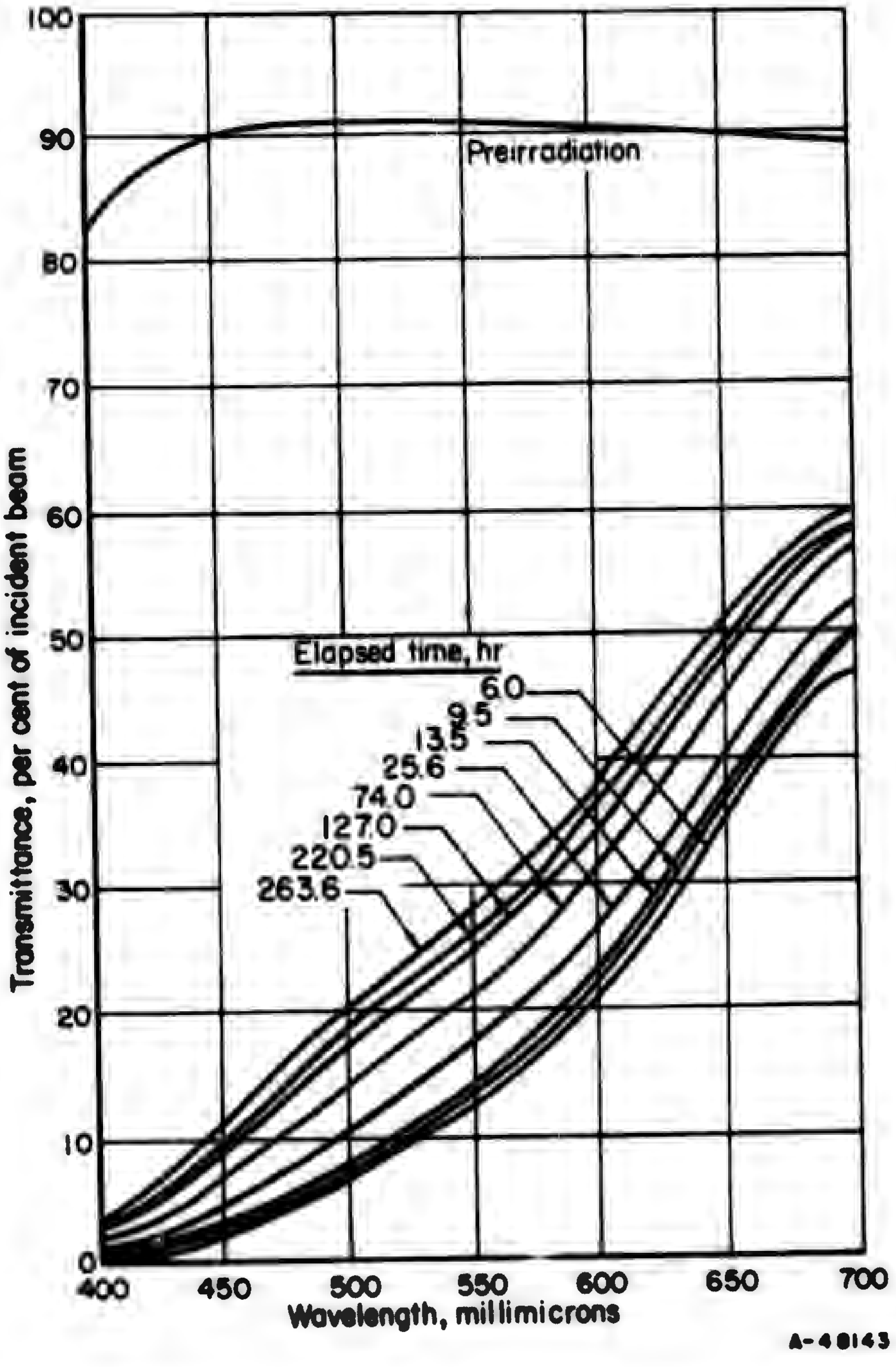

FIGURE D-3. TRANSMITTANCE OF PROTON-IRRADIATED GLASS AS A FUNCTION OF WAVELENGTH(28)

Corning Type 1723; thickness 0.25 in.; total proton exposure = 1 x $10^{13}$ n $cm^{-2}$

## Graphite and Diamond

Hanford Atomic Products Operation(29) has made several studies on dimensional changes in graphite. The dimensional changes and coefficient of thermal expansion of pyrolytic graphite after irradiation to an exposure of 6 x $10^{20}$ n $cm^{-2}$ at 650 C were investigated. It was found that changes in the coefficient of thermal expansion were slight and contraction occurred in the parallel direction. In the transverse direction, however, expansion occurred and the coefficient of thermal expansion changed from 23 x $10^{-6}$ in/in/C to 20 x $10^{-6}$ in/in/C, between 25-425 C.

Baker of the Hanford Operation(30) calculated the thermal conductivity of graphite before and after irradiation at 650 C to an exposure of 1.2 x $10^{21}$ n $cm^{-2}$. In all three samples, the thermal conductivity decreased; however, the material with the highest crystallinity had the highest thermal conductivity and temperature coefficient, while the sample that was the least crystalline had the lowest thermal conductivity and temperature coefficient.

In another report from Hanford(31) graphite samples were irradiated to an exposure of 6500 Mwd/AT at 625 C while under stresses of 150 and 300 psi. Table D-7 gives the results.

TABLE D-7. SAMPLE CONTRACTION RATES(31)

| Graphite | Orientation | Unstressed Samples, %/Mwd/AT x $10^5$ | Stressed Samples, %/Mwd/AT x $10^5$ | P. L. (%) + | Stressed to Unstressed Ratio |
|---|---|---|---|---|---|
| TSX | Transverse | 0.97 ± 0.13 | 1.58 ± 0.25 | 95 | 1.6 |
| ACOT-LS | Transverse | 0.4 ± 0.08 | 1.23 ± 0.21 | 99 | 2.5 |
| NC-8 | Transverse | 0.89 ± 0.34 | 2.52 ± 0.43 | 99 | 2.8 |
|  | Parallel | 0.90 ± 0.12 | 2.92 ± 1.31 | 95 | 3.2 |
| CSF | Transverse | 1.07 ± 0.13 | 2.11 ± 0.33 | 99 | 2.0 |
|  | Parallel | 1.43 ± 0.18 | 1.90 ± 0.27 | 85 | 1.3 |
| TSGBF | Transverse | 0.98 ± 0.08 | 1.37 ± 0.25 | 90 | 1.4 |
|  | Parallel | 1.40 ± 0.10 | 1.59 ± 0.11 | 75 | 1.1 |

Nightingale and Woodruff(32) measured the dimensional changes in graphite bars irradiated to an exposure of 1.8 x $10^{21}$ n $cm^{-2}$ at 500-600 C. It was concluded that the contraction in the large samples was twice that of the smaller bars. Table D-8 summarizes the results.

TABLE D-8. DIMENSIONAL CHANGE OF LARGE AND SMALL GRAPHITE SAMPLES(32)(a)

| Graphite Type | Sample Size, inches | Length Change, per cent Transverse | Length Change, per cent Parallel |
|---|---|---|---|
| CSO – graphitized at 2700 C | 4 x 4 x 24 | -0.45 ± 0.06 | -0.69 |
| CSF – graphitized at 2700 C, then gas purified | 4 x 4 x 24 | -0.53 ± 0.06 | -0.76 |
|  | 0.43 diam x 4 | -0.29 | -0.37 |
| CSGBF – gas baked, then graphitized | 4 x 4 x 24 | -0.56 ± 0.06 | -0.79 |
|  | 0.43 diam x 4 | -0.27 | -0.48 |

(a) Exposure: 18,650 Mwd/at = 1.8 x $10^{21}$ nvt(E> 0.18 Mev).

A study was conducted in Italy(33) on the electrical resistivity, Young's modulus, specific heat, thermal conductivity, lattice parameters, and dimensional changes of graphite following irradiation.

Goggin(34) studied the Young's modulus of graphite that was irradiated at liquid nitrogen temperature by 4 Mev electrons. The Young's modulus remained constant following irradiation, but, as Figure D-4 indicates, there was a change during the postirradiation annealing.

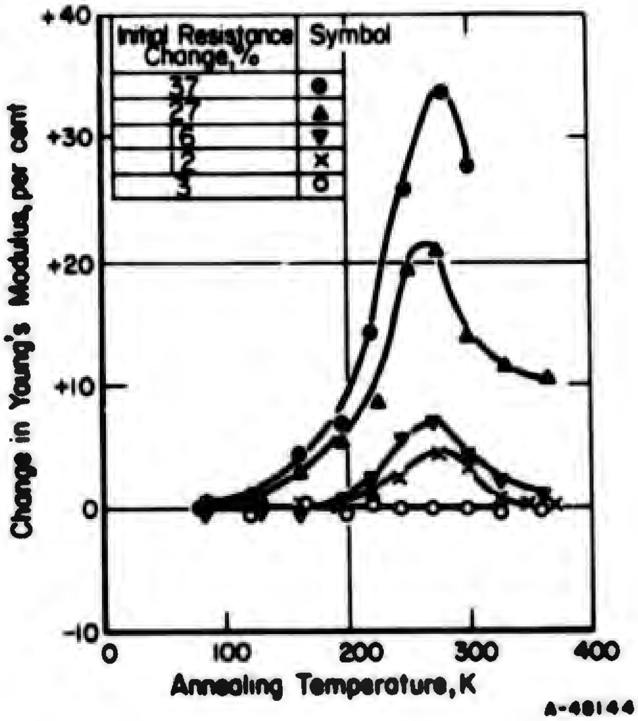

FIGURE D-4. CHANGE IN YOUNG'S MODULUS OF GRAPHITE AS A FUNCTION OF ANNEALING TEMPERATURE

The dimensional change, Hall coefficient, electrical and thermal conductivity, C-axis values, and stored energy of six samples of graphite irradiated to a neutron exposure of 1.7 x $10^{19}$ n $cm^{-2}$ at 30-40 C and then periodically annealed were measured by Aronson of Brookhaven National Laboratory(35). The resultant changes are summarized in Table D-9.

TABLE D-9. PROPERTY CHANGES IN IRRADIATED AND PERIODICALLY ANNEALED GRAPHITE(35)

| Group | Number of Anneals | Average Total Growth, % | C-Axis Values (A) | Hall Coefficient (emu) |  | Electrical Resistivity, ohm-cm x $10^{-3}$ |  | Thermal Conductivity at 350 C, Btu/hr-°F-ft | Total Stored Energy, cal/gm |
|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  | -194 C | 27 C | -194 C | 27 C |  |  |
| AGOT | Unirradiated | None | 6.735 | -1.71 | -0.67 | 2.17 | 1.27 | 60.9 | None |
| A | 17 | 0.010 ± 0.005 | 6.755 | 3.31 | 1.21 | 3.93 | 3.19 | 30.4 | 7 |
| B | 9 | 0.031 ± 0.009 | 6.761 | 2.74 | 1.31 | 3.90 | 3.41 | -- | -- |
| C | 6 | 0.028 ± 0.012 | 6.772 | 2.56 | 1.35 | 3.47 | 3.16 | 18.4 | -- |
| D | 3 | 0.074 ± 0.002 | 6.792 | 1.97 | 1.23 | 4.06 | 3.69 | -- | -- |
| E | 2 | 0.072 ± 0.013 | 6.790 | 2.05 | 1.20 | 4.03 | 3.76 | 15.6 | -- |
| F | 1 | 0.117 ± 0.003 | 6.835 | 1.23 | 0.91 | 4.14 | 3.92 | 13.4 | 79 |
| F' | 0 | 0.163 ± 0.007 | 6.900 | 0.52 | 0.45 | 4.47 | 4.16 | -- | 160 |

Clark and Thompson(36, 37) of the University of Reading observed the changes in the optical density, absorption spectra, and coloration of diamonds after neutron, gamma, and electron irradiation. The effect of subsequent annealing on these properties was also investigated.

Samples of pile-grade graphite were irradiated to an exposure of ~15 MWD/AT at 30 C and then annealed at 350 C.(38) Seventeen cycles of these reirradiations and anneals were performed. Oxidation rates were obtained at 360, 400, 440, and 495 C. Significant changes were not observed in the rates at 400 C. Reirradiation and annealing removed the defects which caused the enhancement of oxidation following the first irradiation, which was probably caused by specific interstitial clusters. The results are illustrated in Figure D-5.

Diamonds irradiated with fast neutrons have been studied by electron paramagnetic resonance (EPR) by Denning and Poindexter.(39) The concentration of free spins and the line width of the signal have been studied as a function of total irradiation, before and after heat treatment. The signal intensity is proportional to total integrated radiation to $10^{18}$ n/cm$^2$. Over a thousand free spins were created for each primary neutron collision with a carbon atom. The EPR intensity decreases rapidly after heat treatment. The line widths were found to be independent of radiation up to $10^{17}$ n/cm$^2$, and they also decrease markedly after heat treatment. The EPR phenomena in irradiated diamond are quite different from those in graphite. It is suggested that highly irradiated diamond is a mosaic of microcrystals separated by a cement of damaged, amorphous, diamondlike glass.

## Programs in Progress*

Current programs on radiation effects in ceramics are summarized below.

The U. S. Air Force, Research and Technology Division, is devoting part of its internal program to the study of the effects of electron irradiation on the transmission properties of infrared windows.

Brookhaven National Laboratory is studying the electrical properties and the fundamentals of irradiation damage and recovery in graphite.

The General Electric Company, Hanford Atomic Products Operation, is investigating radiation effects on nuclear graphite.

The General Electric Company, Hanford Atomic Products Operation, is also studying the effects of radiation on the dimensional stability of boronated graphite.

The General Electric Company, Research Laboratories, is conducting research on the effects of radiation on mica.

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*Programs are described in more detail in REIC Memorandum 13-C, "Compilation of Nuclear, Pulsed, and Space Radiation Effects Projects" (U). Confidential.

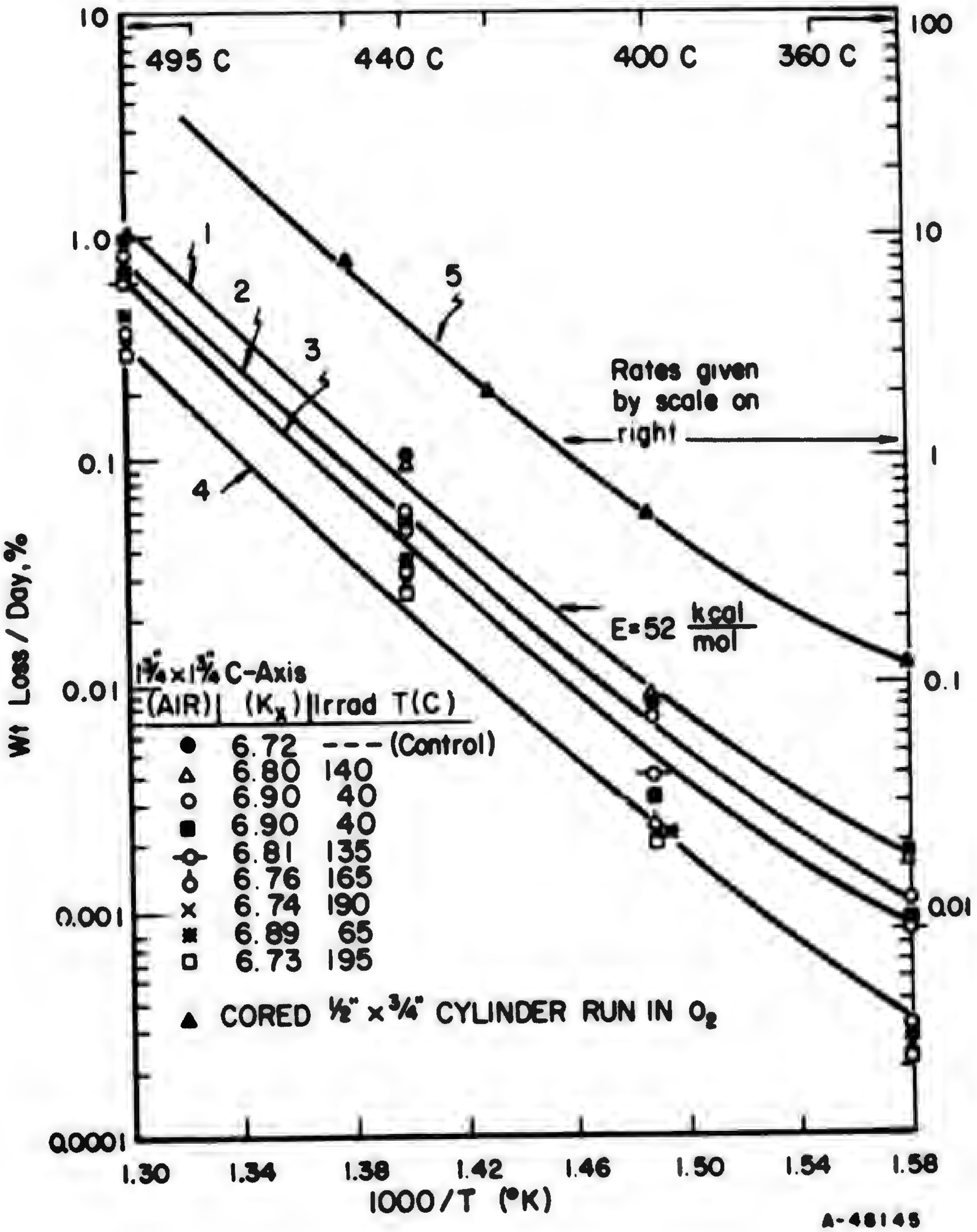

FIGURE D-5. GRAPHITE OXIDATION RATES AFTER 17 IRRADIATIONS AND 350 C ANNEALS(38)

The General Electric Company, Special Purpose Nuclear Systems Operation, is investigating the electrical properties of aluminum oxide in a radiation environment.

North American Aviation, Atomics International, is studying the behavior of pure and boron-doped beryllium oxide following fast-neutron and helium irradiations.

Sandia Corporation is determining the effect of radiation on the physical, electrical, and thermal properties of $Ca_xBa_{1-x}TiO_3$, $BaTiO_3$, and $PbTiO_3$. Isochronal annealing experiments are also being conducted.

The University of Chicago, Argonne National Laboratory, is conducting research on the effects of irradiation at low temperatures on hydrogen bonded glasses.

The University of Connecticut is investigating the growth and stability of radiation defects and property changes in various metal oxides.

The University of North Dakota is conducting research on the effect of neutron and gamma irradiation on silica gels.

The University of Pittsburgh is investigating the paramagnetic resonance and thermoluminescence of germanium-doped quartz.

## Conclusions and Recommendations

Data on ceramic fuel materials and ablative applications provide some property data which may be used in structural and device applications. While no data were generated on the Air Force or General Electric Company, Special Purpose Nuclear Systems Operation studies during the period covered by this report, the data and information generated during the coming year should be of interest to systems designers.

It is evident from the information collected on ceramics over the past year that data on combined environmental effects is still needed. Combined environmental data and information on proton-electron and neutron-gamma radiation, X-rays, ultraviolet radiation, temperature, vacuum, vibration, and shock are necessary to assist the design engineer in his quest for materials for advanced designs.

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## SECTION E

# SPACE RADIATION ENVIRONMENT

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# SPACE RADIATION ENVIRONMENT

## Current State of the Art

In the past year no significant changes have been made in the picture of space radiation environment. The most important items were the report of Explorer 15 data by McIlwain(1)*, the preliminary reports on the Interplanetary Monitoring Probe (IMP) (Explorer 18), and data reported on Injun 3. Another noteworthy event was establishment by the National Aeronautics and Space Administration of a solar flare warning center.(2) Also, a base line for all solar phenomena is expected to be established as a result of the extensive measurements program of the International Quiet Sun Year (1964-65).

## Trapped Radiation and the Van Allen Belts

A report by McIlwain(1) discusses the results of the Explorer 15 measurements made in the period November, 1962, through January, 1963. Mappings of the Explorer 15 findings for high-energy protons, high-energy electrons, and low-energy electrons are shown as Figures E-1, E-2, and E-3. Some of the more important findings reported in this article are:

(1) There is an unexpected secondary peak in the distribution of high-energy protons.

(2) The spatial distributions of high- and low-energy electrons are quite different.

(3) Electrons in the region above L = 1.7 earth radii in the B,L coordinate system are strongly perturbed by the magnetic disturbances.

(4) A new outer zone of both high- and low-energy electrons was formed by the magnetic storm which began December 18, 1962.

Another observation set forth by McIlwain was that the necessary communication of such large masses of data as space radiation measurements is best done by computer program comparisons and computation on radiation damage along various trajectories based upon measurements programs.

Another interesting set of observations was supplied by the Injun 3 geophysical research satellite. Injun 3 was designed to provide detection apparatus to investigate dynamics and structure of radiation zones at high latitudes and, more specifically, to examine such phenomena as auroral glow, very-low-frequency (VLF) radio emissions, and ionospheric disturbances.(3) The instrument complement of Injun 3 included 18 various particle detectors, 3 photometers and a very-low-frequency receiver. Four intense events were observed for data in regard to the energy spectra and flux

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*References are at end of Section E.

distribution of 10 kev electrons in the outer zone of the Van Allen belts. Three of these events were observed in January, 1963, and one event in March, 1963. It was found that the energy spectrum of electrons of energies in the range between 10 kev and 1 Mev cannot be fit by either a power law or the exponential form of the usual spectrum model. Predictions from either model vary widely from observations made within a single pass, and these variations probably are neither purely spatial nor temporal. That observation is that the differential number and energy distribution function of electrons does not always continue to rise toward lower energies in the energy range between 10 kev and 40 kev. It can be suggested as a result of these experiments that a maximum in the differential electron number-energy spectrum may sometimes exist in this energy range of 10 to 40 kev. This spectrum peak also seemed to be associated with auroral phenomenon.

Several reports from the Soviet Union on trapped-radiation measurements were presented at the COSPAR International Meeting held in Warsaw in June, 1963. One of these papers(4) discussed measurements of the charged-particle radiation in the outermost Van Allen regions as measured by the Soviet Mars-1 probe. The most important results were plasma measurements of the outermost radiation belt which indicated that large numbers of low-energy (80 to 200 ev) electrons are present in the outer regions of the earth's magnetosphere. Other measurements reported upon were those made by the Soviet satellites Cosmos 3 and Cosmos 5.(5) Cosmos 3 was launched on April 24, 1962, and had a useful life through June 1962. Cosmos 5 was launched on May 28, 1962. Both of these were operated at inclinations of 49 degrees and fairly low altitudes. No significant changes in the information on the environment resulted from data relayed from these satellites. A third Soviet paper concerned the radiation-belt structure at an altitude of 320 kilometers as examined by the Vostok 2 spacecraft.(6)

IMP data are still being gathered and reduced. Preliminary results were reported at a NASA Symposium in March, 1962. Data discussed to date have not indicated any surprising features of the interplanetary plasma. First data publication will soon appear in a paper by Dr. Ness to be published in the Journal of Geophysical Research.

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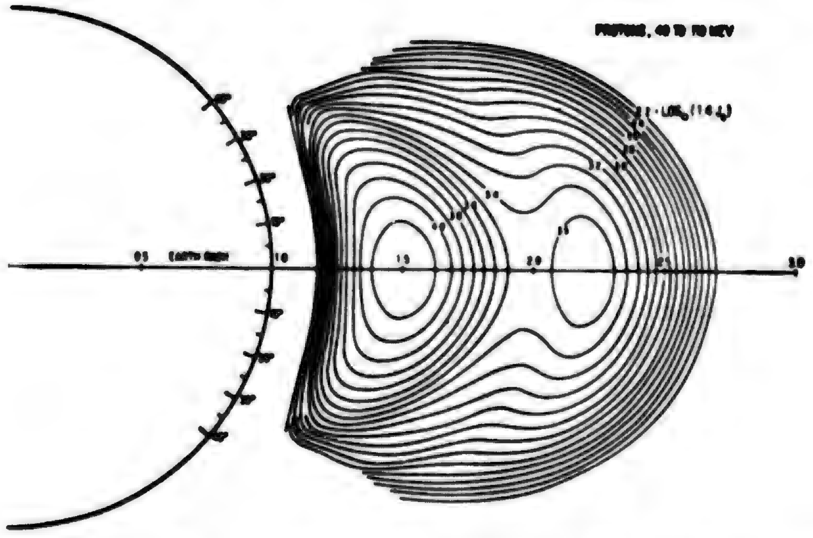

FIGURE E-1. CONTOURS OF CONSTANT INTENSITY OF HIGH ENERGY PROTONS PLOTTED IN MAGNETIC-DIPOLE COORDINATES.(1) THE HORIZONTAL LINE REPRESENTS THE MAGNETIC EQUATOR.

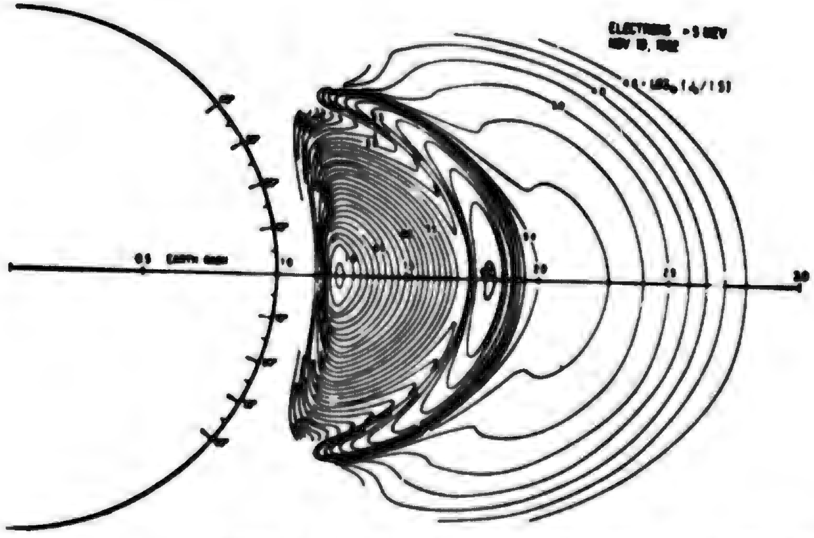

FIGURE E-2. SPATIAL DISTRIBUTION OF HIGH-ENERGY ELECTRONS ON 10 NOVEMBER 1962. THE INTENSITIES REPRESENTED BY ADJACENT CONTOURS DIFFER BY A FACTOR OF 1.259.(1)

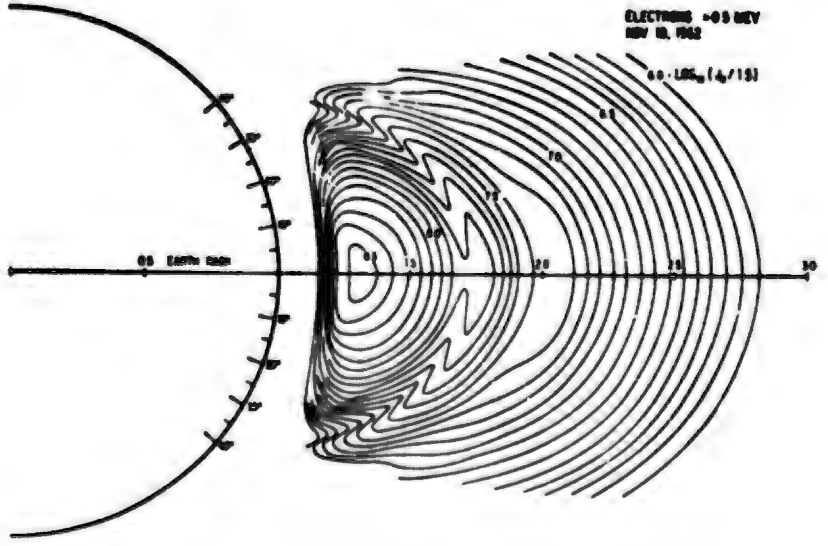

FIGURE E-3. SPATIAL DISTRIBUTION OF LOW-ENERGY ELECTRONS ON 10 NOVEMBER 1962.(1)

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# SECTION F

## DOSIMETRY

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# DOSIMETRY

## Current State of the Art

Current dosimetry procedures as they are applied to radiation-effects experiments using reactor sources (both pulsed and steady state) and pulsed electromagnetic radiation sources (X- and gamma rays) are discussed. In particular, the state of the art in reactor dosimetry is considered in some detail. Although reactors have represented an important source in radiation-effects experiments for many years, the complexity of the radiation environment has presented many difficulties with regard to accurate dosimetry. Uncertainties in the specification of the radiation environment amounting to a factor of two or three have not been uncommon. In light of the unattractiveness of system overdesign to accommodate such uncertainties, considerable effort on the part of researchers is being devoted to means of improving reactor dosimetry techniques. As an example of such work, Committee E-10 of the ASTM on Radioisotopes and Radiation Effects includes a subcommittee whose efforts are devoted entirely to problems on neutron dosimetry, with the major emphasis placed on the standardization and better use of radioactivant techniques for measuring the reactor neutron environment.(1)*

Pulsed electromagnetic radiation sources have been included in the discussion on dosimetry because of the importance of these sources in transient-radiation-effects studies pertaining in particular to simulation of the electromagnetic pulse from bombburst environments.

## Reactor Dosimetry

### Neutron

Atom displacements in solids may affect the various properties of many types of materials. In reactor irradiations this type of radiation effect is caused primarily by the interaction of fast neutrons with the material. The number of displacements per unit flux is a function of the neutron energy distribution, since the energy transferred to the material by neutron collisions depends on the incident neutron energy. The number of displacements produced by an incident neutron increases with increasing neutron energy, with the resulting radiation effect becoming significant at neutron energies of several kev and increasing in importance up to energies of a few Mev where energy loss through ionization tends to saturate the effect. Since the neutron energy spectrum in various reactors, and even within a given reactor, may show significant variations, the study of displacement-induced radiation effects requires some knowledge of the neutron energy distribution. For this reason considerable attention has been focused on this area of reactor neutron dosimetry in order to improve existing techniques and develop new and better methods.

Because of their ability to discriminate against the gamma background, use in high-flux environments, and small size, radioactivants provide a valuable tool for determining reactor neutron spectra. In order to determine the neutron spectrum from

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*References are at end of Section F.

the experimentally measured activities of radioactivants, the activation integral, given by the product of the cross section and neutron flux integrated over energy, needs to be solved for the neutron flux. Several methods, all of which involve certain initial assumptions concerning the shape of the neutron spectrum or cross section, can be used in this analysis.

Recently, Ringle(2) investigated various methods of calculating the neutron spectrum from about 2 Mev to 30 Mev using (n,p), $(n,\alpha)$ and (n,2n) threshold reactions. Included in his investigation were the flux integral method, step function approximation, polygonal method, cross section expansion method, Legendre polynominal expansion method, and Fourier expansion method. Through the application of these methods to various types of trial spectra, he concludes that only three, the polygonal, cross-section, and Legendre methods, appear to offer good results.

Another method of solving the activation integral that is not included in the work by Ringle involves an approximation of the neutron spectrum by exponential functions.(3) In this technique the neutron energy spectrum is divided into energy bands in such a way that for each band a threshold detector is available and in every energy band the spectrum is assumed to be an exponential function given by

$$(E) = Ae^{-kE} \quad .$$

Dierckx bases his selection of an exponential function on an investigation of the fast-neutron spectra of different reactor types which shows that, in defined energy bands with a width of a few Mev, the fast-flux spectrum above 0.5 Mev depends approximately exponentially on the energy. Dierckx has applied his technique to the measurement of fast-neutron spectra at the Reactor BR-1, apparently with good success.

In addition to the methods discussed by Ringle and the procedure suggested by Dierckx, other methods such as that proposed by Lanning and Brown(4) have been used to solve the activation integral and find the unknown spectrum. All of these methods require an accurate knowledge of the reaction cross sections as a function of energy over the energy interval of interest. In general, the application of these methods for obtaining an accurate representation of the differential neutron spectrum from 10 kev up to several Mev has been at best only partially successful. The principal reason for this inadequacy is the current lack of a sufficient number of reactions with well-known cross sections over the desired energy interval. Obviously, the initial assumptions concerning the shape of the neutron spectrum will affect the accuracy of the results as well as the energy interval over which the technique is applicable. Use of new cross section data as they become available will help to improve these methods and extend their usefulness over a wider energy interval. People at IIT have collected cross section data for 32 foil detector reactions that were in the literature and made available through the help of the Sigma Center at Brookhaven National Laboratory and the members of ASTM Subcommittee E-10, Task Group on Dosimetry.(5) After the cross section data were reviewed, evaluated, and tabulated, a best-fit curve was drawn through the plotted points and the results were transferred to IBM punch cards. Although this information was collected as part of an IIT procedure to monitor neutron spectra with radioactivants (this procedure was referenced in an earlier REIC report), its use in other radioactivant methods should be helpful. An additional reaction not included in the compilation by IIT is the $Rh^{103}(n, n')Rh^{103m}$ reaction. Day and his coworkers have investigated the use of Rhodium.(6) They found that the energy sensitivity of this monitor is comparable to the $U^{238}$ (n,f) reaction.

Because of deficiencies in the use of radioactivants to obtain an accurate representation of the neutron spectrum from 10 kev up to several Mev, calculation of the neutron spectrum by multiregion, multigroup techniques has been used by many researchers. Moteff and Beeler(7), and Dahl and Yoshikawa(8) used this approach. In order to indicate the degree of reliance of such calculations, the measured activities of radioactivants can be compared with calculated activities using the theoretical spectrum. It should be noted that many reactor experiments present complex geometries not amenable to the determination of a theoretical neutron spectrum.

Although there are several types of four-pi neutron spectrometers that are able to reject gamma rays using such techniques as pulse height and pulse shape discrimination, the gamma discrimination breaks down in most types when the gamma count rate becomes very high, thus limiting their practical use in reactor measurements.

The experimenter is referred to in an article by Wallace(9) who has evaluated several types of four-pi fast-neutron spectrometers with respect to absolute sensitivity, energy resolution, and discrimination against gamma rays.

$He^3$ spectrometers have excellent gamma discrimination because of the low Z of the He and the 770-kw Q-value of the $He^3(n,p)T$ reaction. In addition when the counter is surrounded by an anticoincidence counter ring to reject escaped protons and ions produced in the counter walls, the gamma counts, which are largely due to wall effects, are automatically eliminated except at the ends. This spectrometer, like the other types discussed by Wallace, cannot be used in intense gamma fields. An additional limitation in the use of $He^3$ spectrometers for measuring neutron distributions with a large percentage of neutrons having energies greater than one or two Mev arises from the elastic scattering of the higher energy neutrons present, masking the $He^3(n,p)T$ peaks due to lower energy neutron groups. Sayers and Coppola(10) have demonstrated that pulse rise-time discrimination can be used as a means to distinguish between pulses from the $He^3(n,p)T$ and $He^3(n,n)He^3$ reactions. Use of this method will increase the useful application of $He^3$ spectrometers for measuring neutron spectra.

Through the use of proper shielding and collimation, people at ORNL(11) have developed a fast-neutron spectrometer capable of measuring neutron spectra in the intense gamma-ray fields characteristic of nuclear reactors. The sensitive element of the spectrometer consists in a layer of $Li^6$ supported between two silicon surface-barrier diodes. Detection of the alpha particle and triton from the $Li^6(n,\alpha)T$ reaction was used to determine the incident neutron energy. In order to determine the background due to neutron interactions in the silicon of the diodes, the $Li^6F$ layer was replaced by a $Li^7F$ layer. The length of the spectrometer including the collimator is about 32 inches and the diameter is approximately 10 inches. A shielded-diode spectrometer of this design was used to measure fast-neutron spectra from about 1 Mev to 12 Mev at the Bulk Shielding Reactor (BSR-1) and the Tower Shielding Reactor-II (TSR-II).

An international symposium on neutron detection, dosimetry, and standardization sponsored by the IAEA was held at Harwell England, in December, 1962. The proceedings of the symposium which are available in two volumes provide an excellent review of the current state of the art in neutron dosimetry.(12) Reference to these volumes has already been made.

## Gamma

Two types of dosimeters insensitive to neutrons and capable of measuring the high gamma doses normally associated with reactor radiation effects experiments are oxalic acid and nitrous oxide. The use of oxalic acid for this purpose was discussed in the previous REIC report on dosimetry.

The nitrous oxide dosimeter measures gamma exposures over the range $10^7$ to $5 \times 10^{11}$ ergs $g^{-1}$(C). The use of this dosimeter is based on the measurement of the pressure of the $N_2 + O_2$ gas formed by the radiation induced decomposition of the nitrous oxide gas. Because of its strength, low-neutron sensitivity and short half-life of induced radioactivity quartz is normally chosen as the dosimeter container for reactor irradiations. Flory(13) has demonstrated the practical application of this dosimeter to measure gamma exposure in reactor experiments. Purification of nitrous oxide gas, filling the dosimeter containers, and the vacuum system used for readout of the irradiated dosimeters are described by Flory.

## Dosimetry Pertaining to Pulsed-Radiation Sources

In addition to the restrictions placed on dosimetry devices used to monitor the neutron and gamma environment of steady-state reactors, devices used to monitor the environment of pulsed facilities have to be capable of functioning in a high-intensity, short-duration, radiation burst. Radioactivants are essentially independent of the radiation intensity and can be used to measure the neutron exposure and energy spectrum. Although use of the $Li^6F$ sandwich neutron spectrometer to measure the neutron spectrum of pulsed reactors has not been reported, it appears that this type of spectrometer could be used.

Chlorinated hydrocarbon chemical dosimeters (Sigoloff dosimeters) are commonly used to measure the gamma exposure per burst. Van Lint(14) has designed a thin calorimeter that can be used for pulsed-reactor gamma dosimetry. By selecting a material with small neutron-capture cross section and high atomic weight, the calorimeter can be made insensitive to neutrons.

The use of semirad devices, solid-state detectors, and plastic scintillators to measure the neutron and gamma intensity as a function of time (pulse shape) has been reported. All of these devices are commercially available.

The standard technique used to record the output of these sensors is to display the signal pulse on a fast oscilloscope and record the pulse shape photographically. Since this type of readout system is awkward and somewhat unreliable, the Nuclear Aerospace Research Facility is developing an electronic readout device which is intended to supplant presently used photo-oscillographic readout of pulsed-radiation sources.(15)

In general, the dosimetry techniques used to monitor the gamma environment of pulsed reactors can be extended to pulsed electromagnetic radiation sources such as flash X-rays and bremsstrahlung from pulsed electron accelerators. Furthermore, dosimetry devices excluded from use in reactors because of the neutron background can be used to measure pulsed gamma environments. The experimenter is referred to the previous dosimetry report for additional information.

The LiF thermoluminescent dosimeter has been investigated independently by several people(16,17) and found to possess attractive characteristics which make it an ideal device for measuring X- and gamma exposure from pulsed facilities. Typical characteristics of the dosimeter are: (1) linear response to exposure from about 0.1 R up to exposure greater than $10^4$ R; (2) a relative energy response of 1.0 from 1.3 Mev to 100 kev and a gradual increase to 1.4 (unshielded) or 1.1 (shielded) from 100 kev to 40 kev; (3) long-term exposure storage properties. Tochilin and Goldstein(18) have measured the exposure-rate response of five dosimeter systems including the thermoluminescent type over a range from $10^4$ to $10^8$ R per second. They found no exposure rate dependence for the systems that were tested.

## Programs in Progress

Current programs on radiation dosimetry are summarized below.

Battelle Memorial Institute is investigating silicon fast-neutron diode dosimeters.

Battelle Memorial Institute is also studying the optimization of performance in the silicon junction fast-neutron dosimeters.

General Dynamics/Fort Worth is investigating neutron measurements in combined environments.

Oak Ridge National Laboratory is investigating threshold detectors and counters for neutron dosimetry, counters for gamma-ray dosimetry, and spectrometers for neutron and gamma-ray dosimetry.

The Philco Corporation is studying a system to detect, differentiate, and resolve the flux of each of the various types of charged particles in space.

Solid State Radiations is devoting a portion of its program to the development of a high-angular-resolution proton spectrometer for space applications.

Texas Nuclear Corporation is studying dose-equivalent space detectors.

The U. S. Army Ballistic Research Laboratories is conducting internal research on the development of a neutron spectrometer for use in both pulsed and steady-state environments.

The U. S. Army Electronics Research and Development Laboratories is investigating gamma and neutron dose-rate meters.

The U. S. Army Electronics Research and Development Laboratories is also devoting a portion of its program to studying a spectroscope for the measurement of fast-neutron spectra as a function of time.

The U. S. Navy Naval Research Laboratory is studying the thermoluminescent dosimeters $CaF_2$:Mn and LiF.

Westinghouse Electric Corporation is conducting research in the development of a miniature neutron detector from SiC, a large band gap semiconductor.

## Conclusions and Recommendations

Although in the last few years considerable effort has been directed toward improving dosimetry techniques and procedures for measuring the reactor radiation environment as it pertains to radiation-effects experiments, the current dosimetry methods in general have limited accuracy and resolution. Radioactivants provide a basic and in many cases the only practical method for monitoring the reactor neutron environment. Additional and better cross section data will undoubtedly improve the resolution and accuracy of this method. It is strongly recommended that current gaps in available cross section data be filled, particularly those types of reactions that can be conveniently utilized to measure the reactor fast-neutron flux environment.

The recommended procedures for reporting information are:

(1) All gamma exposures should be reported in terms of the field through its interaction with a reference material, the unit agreed upon being ergs per gram absorbed in carbon (ergs $g^{-1}$ (C)).

(2) Neutron fields should be described by the product of the neutron density and neutron velocity given by nv = flux in units of n $cm^{-2}$ $sec^{-1}$. The time integral of flux is given by nvt in units of n $cm^{-2}$. The term "dose" to define this quantity should not be used.

(3) The terms "absorbed dose" and "absorbed dose rate" will be used in those cases in which the energy absorbed in the material under study is reported (either by direct measurement or calculation), the unit agreed upon being rads, (the rad corresponds to an energy absorption of 100 ergs $g^{-1}$ by the particular material under study). Otherwise, the terms "exposure" and "exposure rate" will be used.

It should be noted that the terms "exposure dose" and "exposure dose rate" recommended in previous years have been changed to "exposure" and "exposure rate", respectively. This change in nomenclature which is also recommended by the

International Commission on Radiological Units* makes a future change from "absorbed dose" to "dose" possible.

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## SECTION G

## STRUCTURAL METALS AND ALLOYS

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# STRUCTURAL METALS AND ALLOYS

## Current State of the Art

The nuclear-reactor industry uses a large spectrum of materials in many different reactor concepts. Among those reactor concepts in use and under consideration are pressurized water, boiling water, nuclear superheaters, homogeneous, gas-cooled, and sodium-cooled reactors. Many components of these reactors such as fuel cladding, pressure tubing, and structural members are subjected to neutron environment. Hence, radiation-induced property changes in structural metals and alloys continue to be of importance. The effect of some of the higher temperature operations of these reactors on these materials pose additional problems related to corrosion, materials compatibility, and materials strength. It is necessary not only to know the effect of a neutron environment on the short-time properties of these materials but, more important, to know the effect of neutrons at elevated temperatures on the long-time creep properties and the fatigue properties. This section discusses mechanisms of radiation-induced property changes, engineering property changes in ferritic steels, austenitic steels, and some elevated-temperature materials. An attempt will be made to generalize the progress of experimental investigations in these various areas. Trends rather than data are to be reported, and some recommendations with regard to continuance of experimental investigations will be made.

## Mechanisms of Radiation-Induced Property Changes

The changes in engineering properties of materials during irradiation are related to the neutron damage; hence, the mechanisms of neutron damage are considered. It has been generally accepted that changes in properties are related to the formation of point defects within the structural materials. Mass neutron damage has been associated with a displacement mechanism which creates a large number of point defects in a material. Displacements occur during the interaction of neutrons with atoms knocking from their lattice sites,creating a vacancy-interstitial pair. The displacement mechanism creates very large numbers of these vacancy-interstitial pairs. Damask(1)* has reviewed the effect of irradiation in nonfissionable materials and alloys. He considered the generation of point defects and the interaction with existing dislocations, and their influence upon such things as ordering, disordering, enhancement of diffusion, formation and resolution of precipitates, and nucleation of phase changes. Balluffi(2) described specifically the interaction of point defects in face-centered-cubic metals. He also discussed the effect and mobility of these defects during annealing at elevated temperatures. Fleischer(3) and Cottrell(4) discussed the hardening processes noted during neutron irradiation in copper and other materials in terms of formation of the vacancies and interstitial pairs. The changes in properties such as increases in hardness and yield strength, and loss of ductility may in some cases be caused by interstitials or interstitial discs. Interstitial discs are defined as a localized straining caused by the expansion of the lattice to accommodate the interstitial atoms. These effects have been likened to commonly understood solution hardening. Cottrell further related the concept of property changes to clustered lattice defects which can be

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*References are at end of Section G.

produced by the knock-on atoms from the fast-neutron event. Increased hardness in steel may then be more analogous to a type of precipitation hardening. In an attempt to relate the dependence of neutron damage with property changes, Hanstock(5) has expressed empirically the relationships among neutron flux, exposure, and energy spectrum, and the resulting property changes. He has also included the effects of irradiation temperature. For the low-carbon mild steel the brittle transition temperature is dependent on the square root of irradiation damage. Hanstock found that the irradiation-temperature dependency can be related to an annealing process which has an activation energy of about 1.84 ev between 140 and 325 C.

In studies related to mechanisms of changes in properties in other materials, Peacock(6) has studied molybdenum at 160 C and niobium at 120 C. He found that the recovery of properties in these two metals may be explained in terms of lattice vacancies diffusing either to interstitial impurities or to defect clusters (that may have been created by irradiation) where they are annihilated. The maximum recovery was noted after an exposure of about $3 \times 10^{19}$ n $cm^{-2}$. At the lower irradiation levels it appears that the recovery mechanism may be related to trapped interstitials by impurities while the annihilation at clusters is probably important as neutron exposure is increased. Makin(7) has used an electron microscope to study and measure cluster density in irradiated copper. Specimens were irradiated up to $2.5 \times 10^{18}$ neutrons per square centimeter. He found two distinct sizes of clusters and also two distinct stages of recovery. He attributed the smaller clusters to vacancies and the larger ones to the interstitials.

A number of investigators, including Minier-Cassayre(8), have studied the interaction during postirradiation annealing between point defects formed during irradiation and existing impurities. The first of these studies included iron irradiated with neutrons at 78 K and containing from 0.087 to 0.074 atomic per cent nickel. These studies were made by comparing the resistivity of the materials before and after irradiation. Spitsyn(9) also studied annealing and explained annealing in semiconductors on the basis of impurity atoms capturing vacancies from the Frenkel pairs formed by irradiation. Pauleve(10) and Schindler(11) have used magnetic-properties studies to show the effects of irradiation on magnetic iron-nickel and Permalloy. Pauleve studied order and disorder transition for an iron-nickel (50-50) alloy. It was found that a new critical temperature of 320 C was apparent after irradiation. Schindler made measurements of changes in the magnetic susceptibility and lattice parameters of evaporated Permalloy films irradiated with 2.2 mev helium particles and reactor neutrons. He found only minor changes in these properties resulting from the helium particle irradiation after a total of approximately $10^{17}$ helium particles per square centimeter. Regardless of the magnetic state of the sample prior to irradiation, the magnetic susceptibility increased when irradiations with either neutrons or helium particles took place in a zero-applied field. Irradiation effects to a number of materials in fundamental research studies are summarized in References 12-15. Most of these studies involved neutron irradiation resulting in defect formation and the recovery from the annealing of defects at elevated temperatures.

## Ferritic Steels

Carbon and low-alloy steels for pressure-vessel applications have been utilized in many of the pressure vessels of power reactors. The increase in brittle-ductile

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transition temperatures resulting from irradiation continues to be a problem. Many studies have been continued in this area to determine the extent of increase of the transition temperature, the effect of subsequent annealing, and in some instances, the effect of further irradiation after the annealing has been completed. Other areas of extensive study have included surveillance-type programs to ascertain the conditions of these irradiated vessels throughout the reactor life. Impact samples and tensile samples are prepared from the same heats of materials used in fabricating the pressure vessels. These specimens are inserted in the vicinity of the reactor core and receive neutron exposure at a somewhat faster rate than does the pressure vessel itself. At specific time intervals throughout the life of the pressure vessel, these samples are discharged and tests are made to determine the extent of change in the materials as a function of neutron irradiation. A summary of known surveillance programs is given for existing reactors in Table 1.(16) One may expect increases in a ductile-brittle transition temperature of as much as 450 F for neutron exposures of 5 x 10(17,19).

Major emphasis is on experimental programs to determine changes in the brittle-ductile transition temperature. General Atomics(19) is investigating the effect of biaxial stresses on A-302B pressure-vessel steel after irradiation. Oak Ridge(20) workers have studied the impact properties of pressure-vessel steel A212B after irradiation to 2.5 x $10^{18}$ n cm$^{-2}$. Their studies are for comparison of the brittle-ductile transition properties of standard V-notch Charpy impact bars and subsize impact specimens. The Naval Research Laboratory(18) investigations of A212B are concerned with the irradiation of these pressure-vessel steels at elevated temperatures close to the temperature of operation of the pressure vessel. Their studies continue to emphasize the effect of the annealing properties of these irradiated steels and the reirradiation characteristics after their recovery anneal. The investigations are directed toward possible techniques which may utilize high reactor operating temperatures to anneal the pressure vessel periodically. In addition to the tensile properties, some impact properties are being determined. McLaughlin(21) reviewed the effect of irradiation on a number of pressure-vessel steels and has summarized the effect on the ductile-to-brittle transition temperatures.

Impact samples from some of the early surveillance programs are just starting to be evaluated. Impact samples from the Dresden Reactor and from the Yankee Reactor will be tested, and the effects of neutron irradiation from the transition temperature of the pressure-vessel materials will be evaluated.

## Stainless Steels

The austenitic stainless steels have found much application in reactor components. Two properties have made these materials attractive: (1) they do not exhibit a brittle-ductile transition temperature, and (2) their elevated-temperature corrosion and strength properties are desirable for some of the reactors utilizing higher temperatures of application. The areas of increased interest in the irradiation effects of the austenitic stainless steels have been in the areas of elevated temperature. Preliminary studies on reactor components have indicated that the total elongation is considerably reduced by combination of irradiation with elevated temperature and stress. Many studies have been directed toward investigation of the contribution of elevated temperatures. These studies have been carried out at General Electric-Hanford, at

TABLE G-1. SUMMARY OF PRESSURE-VESSEL SURVEILLANCE PLANS FOR 21 U. S. REACTORS(16)

| Reactor | Pressure-Vessel Material | Belt Line Flux for Life, n cm$^{-2}$ (>1 Mev) | Surveillance Planned or Under Way | Surveillance Material | Surveillance Specimens |  | Flux Accumulated to Date (>1 Mev) |
|---|---|---|---|---|---|---|---|
|  |  |  |  |  | Charpy | Tensile |  |
|  |  |  |  | Reactors Now in Operation |  |  |  |
| Vallecitos BWR | A212-B | $5.4 \times 10^{18}$ | No | -- | -- | -- | $3.2 \times 10^{17}$ |
| ASTR[(a)] | SA240-S | $6.4 \times 10^{18}$ | No | -- | -- | -- | -- |
| Dresden | A302-B | $9.2 \times 10^{18}$ | Yes | Representative | 428 | 24 | $5.8 \times 10^{16}$ |
| SM1 | A212-B | $1.0 \times 10^{19}$ | Yes | Representative | 24 | 4 | $1.47 \times 10^{18}$ |
| Yankee | A302-B | $2.04 \times 10^{19}$ | Yes | Original and representative | 168 | 192 | $6.4 \times 10^{17}$ |
| PWR | A302-B | $6.0 \times 10^{19}$ | No | -- | -- | -- | -- |
| RER(a) | Not operating | $7.49 \times 10^{19}$ | No | -- | -- | -- | -- |
| SM1A | A350-LF-1(Mod.) | $8.05 \times 10^{19}$ | Yes | Original (plus A201 and A212-B) | 84 | 14 | Startup: 3/1962 |
| PM-2A | A350-LF-3 | $2.13 \times 10^{20}$ | No | -- | 36 | 3 | $5.4 \times 10^{18}$ |
|  |  |  |  | Reactors Not Yet in Operation |  |  |  |
| Vallecitos ESR | A212-B | $5.6 \times 10^{17}$ | No | -- | -- | -- | Not applicable |
| Humbolt Bay | A302-B | $1.0 \times 10^{18}$ | Yes | Original | Not indicated |  | Not applicable |
| Pathfinder | A212-B | $1.14 \times 10^{18}$ | Yes | Not indicated | 135 | 90 | Not applicable |
| Hallam | 304 Stainless | $2.0 \times 10^{18}$ | No | -- | -- | -- | Not applicable |
| Piqua N. P. | A212-B | $3.4 \times 10^{18}$ | Yes | Representative | 144 | 24 | Not applicable |
| Consolidated Edison | A212-B | $7.6 \times 10^{18}$ | No | -- | -- | -- | Not applicable |
| Bonus N. P. | A212-B | $9.13 \times 10^{18}$ | Yes | Original | 120 | -- | Not applicable |
| Saxton | A212-B | $9.6 \times 10^{18}$ | Yes | Original | 156 | 24 | Not applicable |
| Elk River | A302-B | $1.9 \times 10^{19}$ | Yes | Representative | 240 | 240 | Not applicable |
| Big Rock Point | A302-B | $1.24 \times 10^{19}$ | Yes | Original | Not indicated |  | Not applicable |
| Carolinas | Zircaloy-4 | $1.4 \times 10^{21}$ | Yes | Not indicated | Not indicated |  | Not applicable |
| Enrico Fermi | A240-45<br>304 Stainless | $3.6 \times 10^{21}$ | No | -- | -- | -- | Not applicable |

(a) Test reactors.

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Battelle Memorial Institute, and at Harwell in England. Studies of the irradiation effects in austenitic 20-25 alloy have indicated that the elevated-temperature irradiation enhances the precipitation phenomenon in which the precipitates nucleate and grow in the vicinity of grain boundaries, thus limiting the ductility of the material. It is believed that the loss of ductility is related to the thermal neutron event in which boron-10, present in most materials as impurity atoms, transmutes to lithium and helium. In the latter case, helium diffuses under irradiation at elevated temperatures and in some instances during annealing following irradiation to grain boundaries or second-phase particles, thus reducing the ductility of the material. Although the ductility has been reduced considerably as measured by total elongation, the fracture is often of a ductile type. The main evidence of loss of ductility apparently is related to a localization of the deformation mechanism. Thus, once the stress has been overcome by the flow stress, the deformation takes place in a highly localized area, thus reducing the uniform elongation and total elongation. This mechanism of irradiation hardening probably would not be catastrophic in the event of continued irradiation, i.e., lead to a brittle-type fracture, but could under some specialized conditions produce failure by shear. Many studies, both here and in the United Kingdom, have been related to this recently detected phenomenon, and electron microscope studies have also been made on thin films in an attempt to identify the formation of a second phase from the interaction radiation defects with existing dislocations. Conclusive evidence has been found in many of these investigations to indicate gas bubbles are located at the grain boundaries.

These studies suggest that irradiation temperatures of approximately 50 C do not produce the maximum radiation-induced property changes, as previously believed. Therefore, the most effective investigation on stainless steels and, for that matter, other materials, is the investigation in which the irradiation conditions are as near as possible to the conditions anticipated for the components in service.

## Elevated-Temperature Materials

Materials for application in reactors operating in the elevated-temperature range of 1500-2000 F include such materials as Hastelloy X, the niobium-base alloys, molybdenum, molybdenum-base alloys, tungsten, tungsten-base alloys, and tantalum. A number of investigations studying the short-time properties after irradiation of these materials are being carried out at General Electric-Hanford.(22) General Electric-Cincinnati(23) continues their investigation of tungsten and tungsten-rhenium alloys, comparing the effects of irradiation on the stress-to-rupture properties of these materials. Test temperatures are as high as 4500 F. Irradiation temperatures are somewhat lower, and usually fall in the range of 1500-2000 F.

## Current Programs

An effort has been made by the Division of Reactor Development of the AEC to coordinate programs and correlate data being generated on AEC-DRD supported programs on radiation effects on structural materials. Cooperation is maintained between nine sites and working committees composed of members from the various sites to standardize, where possible, radiation experimentation and reporting techniques in an attempt to provide more meaningful data. Work being conducted under this group's coordination includes the following. Workers at Argonne National Laboratory are studying dosimetry techniques to compare radiation-induced property changes with the neutron spectrum. The use of the manganese-54 activity in the iron-base materials can provide a measure of neutron exposure. These dosimetry measurements and damage indexes are related to the changes in properties of impact specimens from the carbon steels.

Battelle Memorial Institute is continuing its irradiation surveillance program on 347 stainless steel, a program whose objective is to determine the changes in tensile properties, fatigue properties, and impact properties after exposures as great as $3 \times 10^{22}$ n cm$^{-2}$. The current status of this program is that irradiations have been achieved as high as $1.5 \times 10^{22}$ n cm$^{-2}$, and property changes have been determined for exposures as great as $1.1 \times 10^{22}$ n cm$^{-2}$. Specimens having an accumulation of approximately 1.5 x .022 n cm$^{-2}$ will be tested in the near future. Extensive use of nickel and iron dosimetry is made in this particular program.

Brookhaven National Laboratory is conducting a study of the effects of radiation on the plastic and fracture properties of reactor structural materials, with the work centered primarily on the carbon, low-alloy, and stainless steels. Preliminary studies have been made on high-purity iron. The effects of exposures of approximately $1 \times 10^{20}$ n cm$^{-2}$ at temperatures below 100 C, and 100, 200, 300, and 350 C, are being determined. The effects of alloying elements and impurities on the irradiated iron-polycrystallines have been determined. Electron-transmission studies and microscopy studies to determine dislocation distributions are also under way. Some limited studies on the determination of the effects of radiation on the properties of high-purity tantalum and columbium are being made.

General Atomic is studying the effects of radiation on the A-302B pressure-vessel steel. They expect to irradiate various materials to several levels of exposure and test these materials under biaxial stress conditions. Four types of specimens are being investigated over the temperature range of -50 to +350 F. These specimens include tensile, tube, notched-tensile, and impact specimens.

General Electric, Hanford Laboratories, is conducting studies on a number of materials. These rather extensive studies include the oxidation tests of nickel-base alloys in $CO_2$ under reactor conditions, in-reactor creep of Zircaloy-2 in the 250 C and 30,000-psi stress range, irradiation in a hot-water-loop facility of a number of tensile and bend specimens of Zircaloy-2, AM350, 304, 348, and 410 stainless steels. The total radiation exposure for these materials will be in range of $5 \times 10^{19}$ to $4 \times 10^{21}$ n cm$^{-2}$. Fast-flux dosimetry is obtained from iron, titanium, and nickel monitors that are encapsulated along with the samples. The control samples for these radiation tests are subjected to a similar thermal history as the irradiated materials to provide a standard for comparison of radiation effects. Fast-neutron-spectra calculations are made for a number of reactor lattice positions. Environmental studies designed to provide data for understanding the chemical processes controlling the rate and the extent of corrosion and/or embrittlement reactions in reactor structural materials are under way. Several fundamental studies are being made to study the effect of impurity content on materials during irradiation; materials such as high-purity molybdenum, iron, and nickel are anticipated for this program.

The Naval Research Laboratory's objectives are primarily to detect and observe the notched ductility properties of several reactor steels under irradiation and to study recovery of properties by postirradiation heat treatment. The effects of cyclic irradiation-annealing treatment are also under way. The Mechanics Division of the Naval Research Laboratory is currently making studies to determine the fracture toughness of materials in terms of the notched parameters. A measure of the yield strength of the same materials will be made. Materials used in this particular program include A212B and 350LF (PM-2) steels. Both of these steels are pressure-vessel steels and are currently being used as construction materials in nuclear reactors.

The General Electric Nuclear Materials Propulsion Operation is continuing radiation-effects investigations in the area of elevated temperatures. The effects of radiation on the rupture strength of tungsten, tungsten-rhenium, and molybdenum irradiated and tested at temperatures as high as 150 C are being investigated. Additional investigations will be carried out on these materials. The total exposures for most of these materials fall in the range of $1 \times 10^{20}$ to $1 \times 10^{21}$ n $cm^{-2}$. The results of the work indicate that short-time properties are not adversely affected by irradiations at elevated temperatures followed by subsequent testing at the same temperature. However, the long-time properties or creep-rupture properties are somewhat decreased as a result of irradiation. A reduction in ductility is noted. Studies are under way to make calculations and observations related to any metallurgical changes occurring in aged materials during irradiations at elevated temperatures.

Oak Ridge National Laboratory studies are primarily in the fundamental area to study effects of radiation defects in metals. These studies are based upon increases in resistivity and hardnesses observed during irradiation and related to room-temperature deformation characteristics. The short-time creep properties of several materials are being studied as parts of other programs. For example, the stress-to-rupture tube-burst tests on Zircaloy-2, 304 stainless steel, and a columbium-1 zirconium alloy have been made.

Phillips Petroleum Company has initiated a program to study the in-reactor fatigue properties of certain structural materials. The work to date has primarily consisted of the designing and initiation of construction of equipment to conduct these tests. In addition to these induced property changes in reactor materials, a comprehensive study of dosimetry is being made. The effects of fast-neutron exposures on reaction rates of a number of dosimeters have been studied. The evaluation of wire- or foil-type dosimeters for fast-neutron irradiation are under investigation, and a number of experimental points have been determined.

Programs not a part of the AEC-DRD coordinated effort include work at Lockheed to determine the effects of radiation at cryogenic temperatures on the properties of a wide variety of structural alloys, surveillance programs for the Dresden, Yankee, Virginian and Carolina, Rock Point, and Elk River power reactors, and a large number of fundamental programs. Efforts are being made to conduct tests on a full-sized reactor pressure vessel of reactor PM-2A which has seen service. This will probably be a joint effort of Navy Reactors Branch, Army Reactors, and the Division of Reactor Development of the USAFC.

## Recommendations

Future studies in both areas of basic and applied research are recommended. It is believed that, although these suggestions are not all-inclusive, they represent definite needs for a better understanding of radiation-induced property changes and determination of design parameters for reactor designers.

In the area of the fundamental studies on radiation effects, it is recommended that further investigations be directed toward:

(1) Learning more of the role of focusing collisions

(2) Studying and determining factors governing the clustering of interstitial atoms

(3) Showing that ordered lattices disorder much more during deuteron bombardment than can be explained by the simple theory of displacements alone

(4) Relating radiation effects on the basis of the point-defect production and interaction with existing dislocations.

In the area of applied research or determination of radiation-induced property changes in materials, the following additional studies should be made:

(1) Determine and evaluate the effect of neutron-radiation rates

(2) Study the combined effect of temperature and neutrons

(3) Learn more about metallurgical processes such as aging, recovery, and recrystallization during irradiation

(4) Study the property changes of more materials during irradiation after a postirradiation annealing

(5) Learn whether there is some level of neutron exposure after which changes in properties are very small

(6) Determine the effectiveness of surveillance-type programs

(7) Establish empirical relationships by which the effects of long-time irradiation may be extrapolated

(8) Study the role of boron in stainless steels and other metals for high-temperature application.

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# APPENDIX I

## OPERATION OF THE RADIATION EFFECTS INFORMATION CENTER DURING 1963-1964

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# APPENDIX I

## OPERATION OF THE RADIATION EFFECTS INFORMATION CENTER DURING 1963-1964

The operation of the REIC during the 12-month period from June, 1963, through May, 1964, is summarized by listing (1) visitors, (2) travel, (3) technical inquiries, (4) publications, (5) dissemination of information, and (6) operation of the Technical-Data File. In addition to this summary, future plans for the period June 1, 1964, through May 31, 1965, are briefly outlined.

### Visitors

The following breakdown shows the number of visitors for the contract years 1963-1964 and 1962-1963.

| | 1963-1964 | 1962-1963 |
|---|---|---|
| Total Visitors | 40 | 44 |
| Industrial | 21 | 33 |
| Air Force | 9 | 2 |
| Army | 4 | -- |
| Navy | 1 | -- |
| AEC | 0 | 7 |
| Other Government Agencies | 5 | 2 |

### Travel

During the 1963-1964 contract year, personnel from REIC made 13 visits to 16 different organizations to keep abreast of current developments in radiation effects.

### Technical Inquiries

The number of requests for information received by REIC during the contract years 1963-1964 and 1962-1963 are listed below.

| | 1963-1964 | 1962-1963 |
|---|---|---|
| Technical Requests | 383 | 243 |
| Nontechnical Requests | 761 | 585 |
| | 1144 | 828 |

## Publications

In addition to monthly unclassified accessions lists and regular classified accessions lists, the REIC has published the reports and memoranda listed in Appendix II.

## Dissemination of Information

The REIC has continued to disseminate reports, memoranda, and accessions lists on both a classified and an unclassified basis. As of May 31, 1964, the unclassified distribution required 703 copies and the classified distribution required 194 copies.

Following is a breakdown of the distribution lists as of May 31, 1964.

| Organization | Unclassified List | | Classified List | |
|---|---|---|---|---|
| | Addresses | Copies | Addresses | Copies |
| Air Force | 10 | 10 | 31 | 54 |
| Army | 15 | 16 | 17 | 19 |
| Navy | 11 | 11 | 9 | 13 |
| Other Government Agencies | 36 | 41 | 15 | 33 |
| Industrial (Includes AEC) | 587 | 625 | 53 | 104 |
| | 659 | 703 | 125 | 223 |

## Operation of the Technical-Data File

The following breakdown describes the status of the REIC Technical-Data File as of May 31, 1964, after 7 years of operation. Figures for the previous year's operation are included for comparison.

| | Total as of May 31, 1963 | Total as of May 31, 1964 |
|---|---|---|
| Documents Accessioned | 19,111 | 24,206 |
| Documents Extracted | 9,951 | 12,517 |
| Rejected Items | 2,973 | 3,301 |

The rejected items may be categorized as follows:

| | Total as of May 31, 1963 | Total as of May 31, 1964 |
|---|---|---|
| No Pertinent Information | 2,018 | 2,102 |
| No New Information | 779 | 982 |
| Duplicates | 158 | 177 |
| No Radiation Effects | 18 | 40 |
| Total Items Rejected | 2,973 | 3,301 |

## Future Plans

Reports and memoranda in the following areas are being considered for publication during the coming year:

(1) Structural metals and alloys

(2) Propellants and explosives

(3) Electronics in a space environment

(4) Transient radiation effects

(5) Dosimetry and reporting practices

(6) Radiation effects on transducers

(7) Biannual listing of radiation-effects projects

(8) The annual state-of-the-art report.

# APPENDIX II

## LIST OF AVAILABLE REIC PUBLICATIONS

## LIST OF AVAILABLE REIC PUBLICATIONS

Radiation Effects Information Center
Battelle Memorial Institute
Columbus, Ohio 43201

Requests for unclassified information or unclassified REIC published reports and memoranda should be sent:

Radiation Effects Information Center
Battelle Memorial Institute
505 King Avenue
Columbus, Ohio 43201
Attention E. N. Wyler

Requests for classified REIC published reports and memoranda should be sent, via your contracting officer for endorsement, to the following address:

Air Force Materials Laboratory
MAAM (John H. Charlesworth)
Wright-Patterson AFB, Ohio

### Unclassified Reports

| Report Number | Title |
|---|---|
| 1 | The Effect of Nuclear Radiation of Semiconductor Materials (December 20, 1957). AD 147399 |
| 1 (First Addendum) | The Effect of Nuclear Radiation on Semiconductor Materials (March 31, 1959). AD 210758 |
| *6 | A Survey of Current Research and Developments in the Field of Dosimetry (May 31, 1958). AD 157173 |
| 6 (First Addendum) | A Survey of Current Research and Developments in the Field of Dosimetry (March 31, 1959). AD 210766 |
| 10 | The Effect of Nuclear Radiation on Semiconductor Devices (April 30, 1960). AD 240433 (Supersedes Memos Nos. 4, 5, 6) |
| 10 (First Addendum) | The Effect of Nuclear Radiation on Semiconductor Devices (July 15, 1961). AD 262081 |
| 16 | Survey of Irradiation Facilities (February 28, 1961). AD 256953 |
| 17 | The Effect of Nuclear Radiation on Structural Adhesives (March 1, 1961). AD 256954 (Supersedes Reports Nos. 7 and 11) |
| 18 | The Effect of Nuclear Radiation on Electronic Components (June 1, 1961). AD 260303 (Supersedes Reports Nos. 2, 8, 12, 14, and 15 and Memos Nos. 2, 7, 12, 14, and 20) |
| 19 | The Effect of Nuclear Radiation on Lubricants and Hydraulic Fluids (May 31, 1961). AD 261278 (Supersedes Report No. 4) |
| 20 | The Effect of Nuclear Radiation on Structural Metals (September 15, 1961). AD 265839 (Supersedes Report No. 5) |
| 21 | The Effect of Nuclear Radiation on Elastomeric and Plastic Components and Materials (September 1, 1961) AD 267890 (Supersedes Reports Nos. 3, 9, and 13 and Memos Nos. 1, 3, 8, 13, and 17) |
| 23 | Proton and Electron Damage to Solar Cells (April 1, 1962) |
| 24 | Radiation Effects State of the Art 1961-1962 (June 30, 1962). NASA N62-16454 |
| 25 | The Effect of Nuclear Radiation on Transducers (April 19, 1963) |

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*Out of Print in REIC: Available only from DDC, Defense Documentation Center, Cameron Station, Alexandria, Virginia (Name changed from ASTIA, Armed Services Technical Information Agency).

## Unclassified Reports

| Report Number | Title |
|---|---|
| 26 | Transient Radiation Effects on Electronic Components and Semiconductor Devices (April 19, 1963) |
| 27 | The Effect of Nuclear Radiation on Ceramic Reactor-Fuel Materials (June 30, 1963) |
| 28 | Radiation-Effects State of the Art 1962-1963 (June 30, 1963) |
| 29 | Effect of Nuclear Radiation on Metallic Fuel Materials (September 30, 1963) |
| 30 | Effect of Nuclear Radiation at Cryogenic Temperature on Semiconductor Materials (September 30, 1963) |
| 31 | Part I. A Survey of Irradiation Facilities (September 30, 1963)<br>Part II. A Survey of Particle Accelerators (September 30, 1963) |
| 32 | Space Radiation Damage to Electronic Components and Materials (September 30, 1963) |
| 33 | Survey of Current Research and Developments in the Field of Dosimetry (September 30, 1963) |

## Classified Reports

| Report Number | Title |
|---|---|
| 1-C | The Effect of Nuclear Radiation on Hydraulic, Pneumatic, and Mechanical Systems for Subsonic, Transonic, and Low-Supersonic Speed Aircraft (Title Unclassified) (Secret, Restricted Data) (May 31, 1958) |
| 1-C (First Addendum) | The Effect of Nuclear Radiation on Hydraulic, Pneumatic, and Mechanical Systems for Subsonic, Transonic, and Low-Supersonic Speed Aircraft (Title Unclassified) (Secret, Restricted Data) (March 31, 1959) |
| 1-C (Second Addendum) | The Effect of Nuclear Radiation on Hydraulic, Pneumatic, and Mechanical Systems (Title Unclassified) (Secret, Restricted Data) (September 15, 1960) |
| 2-C | The Effect of Nuclear Radiation on Ceramic Materials (Title Unclassified) (Secret) (June 30, 1959), AD 157173 |
| 2-C (First Addendum) | The Effect of Nuclear Radiation on Ceramic Materials (Title Unclassified) (Secret, Restricted Data) (June 15, 1961) |
| 4-C | The Effect of Nuclear Radiation on Electrical and Electronic Systems (Title Unclassified) (Secret, Restricted Data) (March 15, 1960) |
| 6-C | The Effects of Nuclear Weapon Bursts and Simulated Bursts on Electronic Components (Title Unclassified) (Secret, Restricted Data) (May 31, 1961) |

## Unclassified Memoranda

| Memorandum Number | Title |
|---|---|
| *9 | The Effect of Nuclear Radiation on Glass (November 30, 1958) AD 207701 |
| 10 | Format for Reporting Radiation Effects Data (May 15, 1959) AD 218251 |
| *11 | The Effect of Nuclear Radiation on Hydrocarbon Fuels (November 30, 1958) AD 207702 |

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*Out of Print in REIC: Available only from DDC, Defense Documentation Center, Cameron Station, Alexandria, Virginia (Name changed from ASTIA, Armed Services Technical Information Agency).

## Unclassified Memoranda

| Memorandum Number | Title |
|---|---|
| *15 | The Effect of Nuclear Radiation on Hoses and Couplings (March 31, 1959) AD 225504 |
| *16 | The Effect of Nuclear Radiation on Refrigerants (June 30, 1959) AD 219510 |
| *18 | The Effect of Nuclear Radiation on the Performance of a Hydraulic Flight Control System (June 15, 1959) AD 219512 |
| 21 | Space Radiation and Its Effects on Materials (June 30, 1961) |
| 23 | Radiation Dosimetry: An Annotated Bibliography (September 15, 1961) |
| 24 | A Selected Bibliography of Pulsed Radiation Effects (June 1, 1963) |
| 25 | The Beneficial Uses of Radiation Effects |

## Classified Memoranda

| Memorandum Number | Title |
|---|---|
| 1-C | The Effects of Nuclear Radiation on Fluorolubes and Other Gyroscope Fluids (Title Unclassified) (Secret) (September 5, 1958). AD 302126 |
| 2-C | The Effect of Nuclear Radiation on Explosives and Solid Propellants (Title Unclassified) (Secret, Restricted Data) (June 15, 1959) |
| 3-C | Dose-Rate Effects on Materials, Components, and Systems (Title Unclassified) (Secret, Restricted Data) (July 31, 1959) |
| 13-C | Compilation of Nuclear, Pulsed, and Space Radiation Effects Projects (Title Unclassified) (Confidential) (April 15, 1964) |

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(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

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13. ABSTRACT

Developments in the field of radiation effects on electronic components including semiconductors, polymeric materials, lubricants, flotation fluids, hydraulic fluids, structural metals and alloys, ceramics, space radiation environment, and dosimetry are reviewed. Programs currently being conducted in radiation effects are briefly given for each section of the report.

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| 14. KEY WORDS | LINK A | | LINK B | | LINK C | |
|---|---|---|---|---|---|---|
| | ROLE | WT | ROLE | WT | ROLE | WT |
| Radiation Effects | | | | | | |
| Electronics | | | | | | |
| Semiconductor Devices | | | | | | |
| Polymers | | | | | | |
| Lubricants | | | | | | |
| Flotation Fluids | | | | | | |
| Dry Film Lubricants | | | | | | |
| Adhesives | | | | | | |
| Coatings | | | | | | |
| Metals | | | | | | |
| Ceramics | | | | | | |
| Van Allen Radiation | | | | | | |
| Dosimetry | | | | | | |
| Bibliography | | | | | | |
| Gamma Rays | | | | | | |
| Neutron Radiation | | | | | | |
| Electron Radiation | | | | | | |
| Proton Radiation | | | | | | |
| X-ray Radiation | | | | | | |
| Space Radiation | | | | | | |
| Steel | | | | | | |
| Stainless Steel | | | | | | |
| Aluminum Oxide | | | | | | |
| Beryllium oxide | | | | | | |
| Graphite | | | | | | |
| Diamond | | | | | | |
| Glass | | | | | | |
| Electron Tubes | | | | | | |
| Transistors | | | | | | |
| Diodes | | | | | | |
| Solar Cells | | | | | | |
| Capacitors | | | | | | |
| Resistors | | | | | | |
| Pulsed Irradiation | | | | | | |
| Microcircuitry | | | | | | |
| Epoxy resins | | | | | | |
| Silicones | | | | | | |
| Transducers | | | | | | |
| Quartz | | | | | | |
| Laminated Plastics | | | | | | |
| Electromagnetic Radiation | | | | | | |
| Neutron Spectrum | | | | | | |
| Neutron spectrometer | | | | | | |
| Nitrous Oxide Dosimeter | | | | | | |
| Scintillators | | | | | | |
| Thermoluminescent Dosimeter | | | | | | |
| Radiological Units | | | | | | |
| Polyurethanes | | | | | | |
| Phenolic Resins | | | | | | |
| Polyester Resins | | | | | | |
| Fluoroethylene Polymers | | | | | | |
| Magnesium Oxide | | | | | | |

# ERRATA SHEET
for

RADIATION-EFFECTS STATE OF THE ART
1963-1964
REIC Report No. 34
June 30, 1964

Scale for electron exposure in Figure B on page iii should read

$10^{8}$ $10^{10}$ $10^{12}$ $10^{14}$ $10^{16}$ $10^{18}$ $10^{20}$

* Electron Exposure, e cm$^{-2}$($\sim$50% absorption)

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