

WADC TECHNICAL REPORT 53-373  
SUPPLEMENT 5  
ASTIA DOCUMENT NO. 206861

**A REVIEW OF THE AIR FORCE MATERIALS  
RESEARCH AND DEVELOPMENT PROGRAM**

*HELEN HINES MAXWELL  
MATERIALS LABORATORY*

*DECEMBER 1958*

**WRIGHT AIR DEVELOPMENT CENTER  
AIR RESEARCH AND DEVELOPMENT COMMAND  
UNITED STATES AIR FORCE  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO**

# Contrails

## FOREWORD

This report was prepared by Mrs. Helen Hines Maxwell of the Technical Data Section, Technical Services Branch, Materials Laboratory, Directorate of Laboratories, Wright Air Development Center, Wright-Patterson AFB, Ohio.

Technical Reports prepared by Materials Laboratory project engineers (those having no contract number) and Materials Laboratory contractors during the period 1 July 1957 - 30 June 1958 are abstracted herein.

Abstracts of Technical Notes of general public interest and Technical Reports not previously included are given in this supplement.

This report has been released to the Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. for sale to the general public. To order copies of this report, address inquiries to the Department of Commerce.

Military organizations and prime contractors of the Department of Defense engaged in research and development should direct inquiries to Armed Services Technical Information Agency, Arlington Hall Station, Arlington 12, Virginia.

The general public is invited to submit request for copies of technical reports and technical notes which are abstracted herein to the Department of Commerce. Only submit requests for reports that state OTS release. Military Organizations and prime contractors should submit their requests with the ASTIA document number to the Armed Services Technical Information Agency.

The author is greatly indebted to Mrs. Louise Maguire, Mrs Delcencia M. Yates and Major Morris N. Myrick for their assistance and counsel in the preparation of this report.

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## ABSTRACT

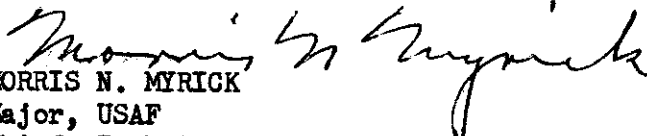
Three hundred and three (303) technical reports and technical notes written during the period 1 July 1957 - 30 June 1958 are abstracted herein. These reports cover the following areas of research: Adhesives, biochemistry, electronic materials, materials physics, metallurgy, packaging, petroleum products, plastics, protective treatments, rubber and textiles.

A contractor index, investigator index and a numerical index of all the technical reports and technical notes issued during the period March 1953 - 1958 are provided.

## PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

  
MORRIS N. MYRICK  
Major, USAF  
Chief, Technical Services Branch  
Materials Laboratory

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I. TECHNICAL REPORTS AND TECHNICAL NOTES 1 JULY 1957 - 30 JUNE 1958

## ADHESIVES, STRUCTURAL

WADC TR 53-126 Pt IV  
ASTIA Document No. AD 130884

July 1957

**SUBJECT:** ELEVATED TEMPERATURE RESISTANT MODIFIED EPOXIDE RESIN  
ADHESIVE FOR METALS

**INVESTIGATOR:** Marguerite Naps

**CONTRACT:** AF 33(616)-3347

**CONTRACTOR:** Shell Development Company

**ABSTRACT:** Metal to metal adhesives which are useful at temperatures up to 500°F, primarily on stainless steel, have been studied. The most promising formulations are based on a solid polyepoxide designated as Experimental Resin X-131. This material is used in organic solvents for the preparation of supported adhesive tapes with a glass fabric carrier. The adhesives are cured at elevated temperature (330°F) and usually at 100 psi. Bonds to type 301 steel are characterized by brittleness and mediocre reproducibility.

An adhesive (Formulation No. 1236) based on a combination of Experimental Resin X-131, EPON<sup>a</sup>) 1009 and Formvar 15/95 E (polyvinyl formal) appears to have the best thermal stability on aging at 500°F of any of the formulations investigated. The adhesive contains aluminum dust as the reinforcing filler and is cured with diaminodiphenylsulfone. Shear strength at 500°F on 301 steel is 1200 psi after 200 hours exposure at 500°F and about 800 psi after 400 hours exposure at 500°F. Strength retention on rapid heating is mediocre, however. The shear strength decreases from about 4000 psi to 530 psi as the test temperature is increased from room temperature to 500°F. Postcuring the bonds at 400°F improves the hot strength slightly to about 800 psi.

Exploratory investigations of adhesives based on ten new experimental resins were unsuccessful for the development of a formulation which is superior in heat resistance to Formulation No. 1236. Several promising paste-type and hot melt adhesives comparable in bond performance to X-131 resin adhesive tapes have been evaluated briefly. The new experimental resins are similar to Experimental Resin X-131 in brittleness and are characterized by a slow rate of cure with conventional curing agent systems.

Manuscript released by author September 1958 for publication as a WADC Technical Report.

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WADC TR 54-447 Pt II  
ASTIA Document No. AD 130879

July 1957

**SUBJECT:** WEATHERING OF ADHESIVE-BONDED LAP JOINTS OF CLAD ALUMINUM ALLOY  
**INVESTIGATOR:** H. W. Eickner  
**CONTRACT:** DO 33(616)56-9  
**CONTRACTOR:** Forest Products Laboratory  
**ABSTRACT:** Lap-joint panels of clad 2024-T3 aluminum bonded with nine metal-bonding adhesives were weathered at Panama Canal Zone; Fairbanks, Alaska; Miami, Fla.; State College, N. Mex; and Madison, Wis. Panels were exposed while stressed in bending and while unstressed. Test panels were also exposed to several laboratory conditions.

Three of the adhesives (2 epoxy resins and a phenolic-neoprene-nylon) were deteriorated seriously at the Canal Zone and Florida. Exposure at the other 3 sites was much milder, and only some stressed epoxy-bonded panels failed at these sites. Through 1 year, conditions at the Canal Zone caused greater bond deterioration than those in Florida. After 3 years, however, the deterioration was generally worse in Florida, and this exposure caused 2 other adhesives (nitrile-rubber type) to deteriorate. Stressing the bonded panels over bending frames generally accelerated the deterioration of those adhesive bonds that were subject to deterioration.

Laboratory exposure to salt-water spray or exposure to continuous or cyclic conditions of 120°F and 97 percent relative humidity promoted deterioration in the same types of bonds that deteriorated from weather at the Canal Zone or Florida sites.

In general, the adhesives that performed well in these exposure tests were types that have qualified under the applicable Military specification, while those that showed appreciable deterioration have not.

WADC TR 55-87 Pt II  
ASTIA Document No. AD 150989

February 1958

**SUBJECT:** TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING  
**INVESTIGATOR:** Samuel N. Muchnick  
**CONTRACT:** AF 33(616)-2347  
**CONTRACTOR:** The Franklin Institute  
**ABSTRACT:** A preliminary investigation of the effectiveness of mild acidic or basic solutions of wetting agents for treating metal surfaces prior to adhesive bonding shows that the strong acid solutions presently in use may possibly be replaced by these milder solutions. Joint strengths approximating the values obtained with strong acid solutions have been obtained with aluminum, stainless steel, and titanium. The pH, concentration of components, and ionic type of wetting agent are specific for each type of metal.

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Additional correlative tests were developed for evaluating surface treatments. The interfacial contact angle between oil and water on the treated surface shows a greater sensitivity than the contact angle in air, and makes it possible to differentiate between two treatments that give zero contact angles against water. Contact resistance and hydrogen overvoltage measurements can be definitely correlated with the contact angle measurements. The contact resistance measurement which also evaluates the effect of oxide thickness appears to be a useful instrument for industrial application.

A study of the role of the adsorbed film on adhesive behavior indicates that the more reactive metals, aluminum and magnesium, are more sensitive to differences in the chemical structure of the film. There is also an indication that these films may affect the adhesional characteristics of some type of bonding agent.

The temperature of the rinse water following treatment of aluminum is shown to be important in the adhesional characteristics of the metal. Reproducibility and specificity of attractive forces between adhesive and metal were investigated, and it was apparent that the material and procedural parameters must be studied along with specific affinities in an adhesive system.

WADC TR 55-87 Pt III  
ASTIA Document No. AD 131093

September 1957

SUBJECT: TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING  
PART III. Contact Angle and Contact Resistance  
Devices for Quality Control  
INVESTIGATOR: Richard H. Hollinger  
CONTRACT: AF 33(616)-2347  
CONTRACTOR: The Franklin Institute  
ABSTRACT: Two instruments have been developed for quality control use to determine whether surfaces of metals have been adequately prepared for adhesive bonding. The contact resistance apparatus measures the break-through voltage of a surface film at a given voltage. The contact angle apparatus measures the angle of contact between a drop of water and the specimen surface.

WADC TR 55-87 Pt IV  
ASTIA Document No. 150993

February 1958

SUBJECT: TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING  
INVESTIGATOR: Harry G. Bickford  
CONTRACT: AF 33(616)-2347  
CONTRACTOR: The Franklin Institute

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**ABSTRACT:** The scope includes the development and evaluation of mild surface treatments for clad aluminum alloy, stainless steel, and titanium alloy and investigations of the critical factors involved in the treatment of magnesium alloy. Methods of evaluating the degree to which a surface has been cleaned are discussed with regard to the ease of conducting the tests and with regard to their reliability. Effects of electrical potentials, strong alkali and acid exposure of treated surfaces, and time delays between treatment and priming of the surfaces on bond strength are subjects of discussion. Results of radioactive-tracer work using isotopic chromium are used to lay a basis for a theory of bonding.

WADC TR 55-87 Pt V  
ASTIA Document No. AD 150998

February 1958

**SUBJECT:** TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING  
**INVESTIGATOR:** Edmund Thelen James B. Drew  
Richard Hollinger Charles J. Varker  
Thomas I. Haigh, Jr. Wallace E. Frank  
**CONTRACT:** AF 33(616)-2347  
**CONTRACTOR:** The Franklin Institute

**ABSTRACT:** The scope includes analysis of strong and mild surface treatments for clad aluminum alloy, stainless steel, and titanium alloy and of the best treatment for magnesium alloy. Reaction of bonded surfaces to sand and vapor blast treatments is analyzed. The resistance of the treatments to salt spray are evaluated. Field-repair treatments are discussed and results of tests tabulated. Quality control instrumentation is outlined. Application of d-c potentials to bonded surfaces was determined to be deleterious. Radio-tracer studies using isotopic chromium provided valuable information regarding the role of chromium ions in metal surface preparation. A means of providing continuous indication of bond strength during the life of the plane is suggested.

WADC TR 55-87 Pt VI  
ASTIA Document No. AD 150999

February 1958

**SUBJECT:** TREATMENT OF METAL SURFACES FOR ADHESIVE BONDING  
PART VI. Summary of Methods  
**INVESTIGATOR:** Richard Lindsay, Jr.  
**CONTRACT:** AF 33(616)-2347  
**CONTRACTOR:** The Franklin Institute  
**ABSTRACT:** Methods for surface preparation of stainless steel, titanium, aluminum, and magnesium for adhesive bonding are contained in this summary. The treatments are those that were found to be most promising when used with certain adhesives.

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WADC TR 55-271 Pt III  
ASTIA Document No. AD 151107

April 1958

SUBJECT: RESEARCH ON ELEVATED TEMPERATURE RESISTANT INORGANIC  
POLYMER ADHESIVES  
INVESTIGATOR: Harold H. Levine  
CONTRACT: AF 33(616)-3824  
CONTRACTOR: Quantum, Incorporated  
ABSTRACT: Attempts to prepare heat stable materials for formulation  
with the ammeline-phosphorus pentoxide inorganic adhesive are described.  
The two approaches deemed as most promising were epoxysilicon and epoxy-g-  
triazine compounds.

The synthesis and preliminary evaluation of 2,4-bis(glycidyl)-  
6-methoxy-g-triazine is described. With certain formulations a tensile sheer  
value of about 2100 psi was obtained from room temperature up to and including  
400°F.

An unexpected degree of reactivity was found in 2,4-bis  
(glycidyl)-6-methoxy-g-triazine and materials such as diphenylsilanediol and  
commercial silicone polymers could be used as true curing agents.

The inorganic adhesive prepared from ammeline and phosphorus  
pentoxide cures the 2,4-bis(glycidyl)-6-methoxy-g-triazine. Further work is  
necessary on this particular system.

Synthesis of trimethylsilylpropylene oxide, as a model com-  
pound, was shown by infra-red analysis but could not be isolated.

WADC TR 55-491 Pt III  
ASTIA Document No. AD 142226

December 1957

SUBJECT: RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC  
STRUCTURAL ADHESIVES  
INVESTIGATOR: Richard M. Spriggs Henry G. Lefort  
Charles N. Williams Dwight G. Bennett  
CONTRACT: AF 33(616)-2556  
CONTRACTOR: University of Illinois  
ABSTRACT: The object of the investigation was to develop high temper-  
ature resistant structural adhesives for Type 301 and 302 stainless steel or  
other desired alloy metals, particularly Type 17-7 PH, from ceramic-oxide  
glassy-bond coatings, cermets with sintered metal bonds, air setting, temper-  
ature-resistant silicates, aluminates, oxychlorides, oxysulfides, and ceramic-  
oxide resin bonded coatings.

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Nine new ceramic glassy-bond adhesives developed to mature within the relatively low temperature range of 1300-1500°F, gave only nominal shear strengths of 530 psi at 600°F and 750 psi at 800°F.

Glassy-bond ceramic adhesive UI 117-50, when matured at 1750°F during the hardening treatment RH 950 for 17-7 PH stainless steel, gave average (two to four specimens) shear strengths ranging from 990 psi at room temperature to 980 psi at 1000°F. The curve was quite flat through four of five temperature points. It reached a high of 1750 psi at 900°F.

Glassy-bond ceramic adhesives UI 1067-3 and UI 1067-4, containing 10 and 20 per cent carbonyl iron, respectively, both developed more than 1500 psi at room temperature and at 1000°F, with higher values at 600 and 800°F. Their shear strength-temperature curves were quite flat with all values being within about 10 per cent of the average values which were slightly above 1700 psi.

The advantageous use of carbonyl iron (20%) and silicon powder (10%) is shown with ceramic adhesive UI 117-63 which gave an average shear value of 1455 psi at four temperature points (room to 1000°F). The highest value was 1505 psi at room temperature. The lowest was 1415 psi at 800°F. All of the values were within about three per cent of the average.

Adhesive bonded specimens subjected to the severe environment of 100 hours immersion in boiling water, in general, lost strength but one adhesive (UI 117-59) containing 10% carbonyl iron and used with a 28 mesh stainless steel carrier actually showed an increase in strength after immersion in boiling water.

Other specimens, both with and without additions of carbonyl iron, were subjected to 143 hours of heating at 1000°F plus 25 thermal shock cycles between room temperature and 1000°F. Their shear strengths were improved rather than harmed by this treatment.

The great potential usefulness of metal powders in ceramic adhesives is shown by UI 117-64 which contains 30 per cent carbonyl iron. This adhesive, although it fell off sharply on either side, developed an average of more than 5600 psi at 800°F.

In the field of air-setting or low temperature cure adhesives it appears that those of the aluminum phosphate type offer some promise.

Tests conducted with air-setting adhesives indicate that their moisture sensitivity can be reduced through the use of a dense adhesive structure, or by the addition of a sealant such as tetraphenyltin.

WADC TR 56-320 Pt II  
ASTIA Document No. AD 150967

February 1958

SUBJECT: RESEARCH ON STRUCTURAL ADHESIVE PROPERTIES OVER A WIDE  
TEMPERATURE RANGE

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INVESTIGATOR: H. R. Merriman  
H. L. Goplen  
CONTRACT: AF 33(616)-3902  
CONTRACTOR: The Martin Company

ABSTRACT Time-deformation creep curves were obtained for a number of adhesives using specified test temperatures and stress levels which resulted in time-to-rupture life in the 100-to 200-hour range. The stress levels used in this project were determined from information obtained from work reported in WADC TR 56-320.

The adhesives tested were AF-6, PA-101, Flastilock 608, Metlbond 4021, FM-47 Liquid, FM-47 Film, Redux E (Type R), Cycleweld 55-20, Shell 422, and HT-424. Two metal alloys were used, 2024-T3 clad and bare aluminum alloy and Type 301,  $\frac{1}{2}$  hard stainless steel.

The tests were conducted on  $\frac{1}{2}$ -inch lap coupons, at three different temperatures for each adhesive. Stress was applied by dead weight loading. The deformation of the glue line was measured by observing the displacement of three fine scribe lines across the vertical edges of the lap joint. A Gaertner 100-power microscope with a bifilar micrometer eyepiece and 10X objective lens was used to measure deformation of the glue line.

The results obtained in these tests indicated that the method of measuring creep deformation by observing scribe lines through a microscope, properly controlled, provides a reproducible test procedure. It was noted that each specific adhesive had an approximately constant total deformation and creep rate for the three tests temperatures and corresponding stress levels.

WADC TR 57-513  
ASTIA Document No. AD 142152

November 1957

SUBJECT: STRUCTURAL ADHESIVES AND SANDWICH CONSTRUCTIONS  
WADC - UNIVERSITY OF DAYTON JOINT SYMPOSIUM

INVESTIGATOR: Theodore J. Martin

ABSTRACT: This report is a compilation of papers presented at the Wright Air Development Center - University of Dayton joint symposium on Structural Adhesives and Sandwich Constructions, 12-13 June 1957, Dayton, Ohio. The papers, for the most part, reviewed work done on Materials Laboratory research and development contracts.

WADC TR 57-696  
ASTIA Document No. 151126

April 1958

SUBJECT: ROOM TEMPERATURE CURING STRUCTURAL ADHESIVES FOR METALS  
INVESTIGATOR: E. A. Blommers B. D. Halpern  
E. W. Lane

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**CONTRACT:** AF 33(616)-3654  
**CONTRACTOR:** The Borden Company  
**ABSTRACT:** Formulations containing novolac epoxy resins have been evaluated as room temperature, low pressure cured metal-to-metal adhesives. A number of combinations of novolac epoxy resins, Epon 562, Thiokol LP-3, fillers and mixtures of primary and tertiary amines have been found to give bonds with good strengths and salt spray resistance.

Attempts to use cyclohexene diepoxides in room temperature curing adhesive formulations did not prove successful. Copolymerization of methacrylateoethyl phosphates with methyl methacrylate in redox systems at room temperature yielded adhesives with mediocre strengths.

## BIOCHEMISTRY

WADC TN 57-232

August 1957

**SUBJECT:** EVALUATION OF FOUR AROMATIC FLUORINE COMPOUNDS AS FUNGICIDES FOR VEGETABLE AND CHROME TANNED LEATHERS  
**INVESTIGATOR:** Jerome J. Jansing  
William T. Roddy  
**CONTRACT:** AF 33(616)-2466  
**CONTRACTOR:** University of Cincinnati  
**ABSTRACT:** Fungus resistance evaluations were made on vegetable tanned and chrome tanned leather materials treated with basic formulations of selected aromatic fluorine chemicals.

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

WADC TR 58-32  
ASTIA Document No. AD 151034  
OTS Release

March 1958

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

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

## BRAZING ALLOYS

WADC TR 57-648  
ASTIA Document No. AD 151013  
OTS Release

March 1958

SUBJECT: DEVELOPMENT OF OXIDATION AND LIQUID SODIUM RESISTANT  
BRAZING ALLOYS

INVESTIGATOR: Domenic A. Canonico  
Harry Schwartzbart

CONTRACT: AF 33(600)33406

CONTRACTOR: Armour Research Foundation

ABSTRACT: The first year's work has been completed at Armour Research Foundation on a program which has as its objectives: (1) the development of filler metals for brazing type 310 stainless steel to Inconel, the brazements to be resistant to oxidation and attack by molten sodium at 1650°F, and (2) the development of a simple test for the ductility of a brazed joint.

Three metals -- iron, chromium, and nickel -- were selected, from oxidation and sodium resistance considerations, as the base metals from which to develop acceptable brazing alloys. From a study of all the available pertinent phase diagrams 91 alloys were devised, of which 16 satisfied the flow temperature and flowability requirements of the program. The flow temperature was between 1750°F and 1900°F. Minimum flowability was three inches of length along a six-inch T-specimen.

T-specimens brazed with these 16 alloys were subjected to an oxidizing atmosphere for 500 hours at 1650°F. Four alloys successfully withstood oxidation to a depth of less than 0.003 inch.

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A test for ductility of brazed joints, based on the premise that the load necessary to cause cracking in the fillet of a braze is a measure of the ductility of the brazing alloy, has been developed. The test has shown itself to be capable of differentiating two filler alloys, although the large amount of scatter obtained required that a statistical analysis of the data be applied. Further development should reduce the scatter.

## CERAMICS

WADC TR 55-500 Pt I  
ASTIA Document No. AD 130904

July 1957

SUBJECT: AN INVESTIGATION OF INFRARED TRANSMITTING MATERIALS  
INVESTIGATOR: N. J. Kreidl R. A. Weidel  
H. C. Hafner E. C. Letter  
J. R. Hensler

CONTRACT: AF 33(616)2769

CONTRACTOR: Bausch & Lomb Optical Co.

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

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

WADC TR 57-86

January 1957

SUBJECT: HOT PRESSING OF BERYLLIA ROCKET NOZZLES  
INVESTIGATOR: J. K. Evans  
G. D. Kriebel  
CONTRACT: AF 33(616)3699  
CONTRACTOR: The Beryllium Corporation

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

WADC TR 57-135  
ASTIA Document No. AD 131031  
OTS Release

August 1957

**SUBJECT:** A STUDY OF GRADED CERMET COMPONENTS FOR HIGH TEMPERATURE TURBINE APPLICATIONS

**INVESTIGATOR:** Henry W. Lawendel  
Claus G. Goetzel

**CONTRACT:** AF 33(616)-3670

**CONTRACTOR:** Sintercast Corporation of America

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

The graded layers were applied during the infiltration of the wedge by filling with an excess of infiltrant a gap formed between the skeleton and an investment mold used in the infiltration process. Thus a structure was obtained in which the graded surface layer constitutes a continuation and extension of the nickel alloy matrix of the cermet core containing dispersed titanium carbide inclusions. The test results indicate a substantial improvement to be gained in ballistic impact strength by providing metal-enriched areas at the edges and tip of the airfoil. An oxida-

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tion-resistant ductile alloy layer deposited as a cladding on the entire surface of the simulated turbine component resulted in an equally important improvement of the thermal shock resistance of the titanium carbide cermet bodies.

WADC TR 57-577 Pt I  
ASTIA Document No. AD 150957  
OTS Release

February 1958

**SUBJECT:** DEVELOPMENT AND EVALUATION OF INSULATING TYPE  
CERAMIC COATINGS  
PART I. Development and Small Scale Testing

**INVESTIGATOR:** S. Sklarew  
C. A. Hauck  
A. V. Levy

**CONTRACT:** AF 33(616)-2957

**CONTRACTOR:** Marquardt Aircraft Co.

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

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

WADC TR 57-665  
ASTIA Document No. AD 150965  
OTS Release

February 1958

**SUBJECT:** DEVELOPMENT AND EVALUATION SERVICES ON CERAMIC  
MATERIALS AND WALL COMPOSITES FOR HIGH-TEMPERATURE  
RADOME SHAPES

**INVESTIGATOR:** John J. Dorsey

**CONTRACT:** AF 33(616)-3397

**CONTRACTOR:** P. R. Mallory & Co., Inc.

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

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

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

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

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

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

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

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

WADC TR 58-12 Pt I  
ASTIA Document No. AD 151079  
OTS Release

March 1958

SUBJECT: HIGH-TEMPERATURE ELECTRICAL INSULATING INORGANIC COATINGS ON WIRE

INVESTIGATOR: Clifton G. Bergeron Robert J. Beals  
Arthur L. Friedberg William M. Faber  
Paul F. Schwarzlose

CONTRACT: AF 33(616)-3943

CONTRACTOR: University of Illinois

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

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

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

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

WADC TR 58-13 Pt I  
ASTIA Document No. AD 151062  
OTS Release

March 1958

**SUBJECT:** HIGH-TEMPERATURE INSULATION FOR WIRE  
**INVESTIGATOR:** J. D. Walton and J. N. Harris  
**CONTRACT:** AF 33(616)-3944  
**CONTRACTOR:** Georgia Institute of Technology  
**ABSTRACT:** A literature survey was undertaken to review possible methods for use in developing an electrical insulating coating for the high temperature protection of wire.

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

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

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

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

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

## CHEMISTRY

WADC TR 53-147 Sup 1  
ASTIA Document No. AD 131072

September 1957

**SUBJECT:** NON TOXIC SOLVENTS FOR CLEANING AIRCRAFT ENGINES  
AT EXTREMELY LOW TEMPERATURES

**INVESTIGATOR:** Robert E. Cretcher

**ABSTRACT:** Two reciprocating aircraft engine degreasing formulae consisting essentially of methylene chloride and aliphatic petroleum hydrocarbon, suitably inhibited, had been recommended on the basis of laboratory evaluations as being suitable for use under arctic conditions. Service tests were conducted by three operational activities of the Alaskan Air Command during the winter seasons of 1954-1955, and 1955-1956. The results of the operational usage of the two degreaser formulae and a control solvent indicate generally that either of the test formulae would be suitable for the purpose at temperatures above  $-20^{\circ}\text{F}$ . One of the test materials evaporates more slowly than the other but may be used as both cleaning and rinse solvent. While the second material evaporates more rapidly, a straight petroleum solvent is required to rinse the cleaning solvent from the treated surfaces. Test results are discussed and the proposed specification requirements for the recommended degreaser are presented.

WADC TR 56-495  
ASTIA Document No. AD 130888

July 1957

**SUBJECT:** DEVELOPMENT OF NON-TOXIC ORGANIC SOLVENTS OR SOLVENT SYSTEMS FOR GENERAL CLEANING APPLICATIONS

**INVESTIGATOR:** Kenneth K. Aoki  
Arthur J. LaRue  
Brock G. Peacock

**CONTRACT:** AF 33(616)-2775

**CONTRACTOR:** Wyandotte Chemicals Corporation

**ABSTRACT:** A search has been made for new solvents or solvent combinations which could satisfactorily replace carbon tetrachloride in general cleaning applications. Inhibited methylchloroform and combinations of methylchloroform with up to 15 volume percent of Freon 112 were formulated. Freon 112 is used to reduce the evaporation rate of methylchloroform which is slightly higher than that of carbon tetrachloride. Although the corrosion inhibition properties of the formulated mixtures cannot be considered satisfactory, the target specifications appear to have been met in respect to minimum flash point, decrease in flash point during evaporation and toxicity. The solvents are essentially equal to or better than carbon tetrachloride in respect to deleterious action on various electrical insulators, plastic laminates, and organic coatings. The recommended solvents can be economically produced in the required volume. Although the present limited production of Freon 112 and its consequent high cost may limit the propor-

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tion which can be used in the solvent combination, there is reason to anticipate materially reduced costs under volume production. Areas in which continuing development work is needed are the further refinement and proving of several possible inhibitor systems for optimum results, long term storage and stability tests under anticipated storage conditions, practical application tests, and final clearance in respect to toxicity by Air Force medical personnel.

WADC TR 57-187  
ASTIA Document No. AD 151073

March 1958

**SUBJECT:** LOW TEMPERATURE AIRCRAFT SURFACE CLEANERS

**INVESTIGATOR:** Elias Kimmel  
Bernard Berkeley  
Daniel Schoenholz

**CONTRACT:** AF 33(600)-32244

**CONTRACTOR:** Foster D. Snell, Inc.

**ABSTRACT:** Physical and chemical characteristics of different types of soil normally encountered on aircraft surfaces were studied. Laboratory soil removal tests were developed and used to evaluate all important classes of organic solvents. Seventeen different amine oleates were examined for their ability to form dilute water-in-naphtha emulsions. A combination of diethylamine oleate and triethanolamine oleate allowed the incorporation of 1.5 parts of water in naphtha and naphtha-chlorinated solvent blends. Emulsifier systems were compounded to render hydrocarbon and hydrocarbon-chlorinated solvent blends readily rinsable with diluted deicing fluids. Field tests at Loring A. F. B. and Mitchel A. F. B. established the efficacy of water-containing and self-emulsifiable cleaners. Two cleaners conforming to all target requirements are recommended for removal of oily, and traffic film, type soils from aircraft surfaces at temperatures of 0° to 32°F. One cleaner is used in conjunction with a naphtha rinse; the other is flushed with either naphtha or a glycol-alcohol-water fluid. Raw material costs, methods of preparation, sources of supplies and ingredient specifications are reported for both recommended cleaners.

WADC TR 57-193  
ASTIA Document No. AD 151022

March 1958

**SUBJECT:** DEVELOPMENT OF NON-CRESYLIC CARBON REMOVERS

**INVESTIGATOR:** Rubin Weiner  
J. Walter Carroll

**CONTRACT:** AF 33(600)31890

**CONTRACTOR:** Pennsalt Chemicals Corporation

**ABSTRACT:** The usual carbon remover formulations now in use contain chlorinated hydrocarbons, cresols, and wetting agents. As such they present a serious toxicity and waste disposal problem and are therefore unsuitable for field use where only limited facilities are available. Major Air Force overhaul depots are successfully removing engine varnish (polymerized

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oils) and loosening carbon deposits from reciprocating engine parts with this material. However in this laboratory, they have been found unsatisfactory for removing carbon deposits from either jet engine or reciprocating engine parts.

Two formulations, which this laboratory developed, appear to be much more effective in removing carbon deposits than the present specification compositions. Laboratory tests indicate that these two formulations are satisfactory with respect to corrosivity and toxicity, present no serious waste disposal problems, and are not expensive to manufacture.

WADC TR 57-606  
ASTIA Document No. AD 142158  
OTS Release

November 1957

SUBJECT: ORGANIC COMPOUNDS OF GALLIUM - A SUMMARY  
OF THE LITERATURE  
INVESTIGATOR: Andrew J. Frank  
Richard W. Sullivan  
Virgil W. Lichtenberg  
CONTRACT: AF 33(616)-2939  
CONTRACTOR: Denver Research Institute  
ABSTRACT: The literature concerning organic compounds containing gallium has been surveyed, and the properties of all organo-metallic and organic coordination compounds of gallium are summarized. An annotated bibliography of all references is provided.

## COATINGS

WADC TR 53-511 Pt II  
ASTIA Document No. AD 130780

June 1957

SUBJECT: DEVELOPMENT OF A HEAT RESISTANT COATING FOR  
PROTECTION AGAINST RAIN EROSION  
INVESTIGATOR: Frank A. Jeffries  
CONTRACT: AF 33(616)-2231  
CONTRACTOR: The Goodyear Tire and Rubber Company  
ABSTRACT: High speed flight through rain causes damage to exposed parts of aircraft. Neoprene coatings have been used to protect parts against this damage.

Skin friction due to the high speeds of aircraft and the practice of thermal de-icing, have made it necessary to investigate erosion resistant coatings which will withstand temperatures up to 500°F.

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All available elastomers and plastics giving promise of withstanding such temperatures have been examined. Of these, only acrylic ester rubbers, Teflon, and silicone rubbers meet the temperature requirement. Teflon is poor in erosion resistance.

The silicone and acrylic ester rubbers, although they do not have as high a degree of erosion resistance as Neoprene in the present stage of development, afford a fair degree of protection even after exposure to 500°F. Inconsistent adhesion has prevented the ultimate in erosion resistance from being realized.

Hypalon S-2 will not withstand 500°F, but does afford a fair degree of erosion resistance where temperatures remain below 400°. Here again, adhesion is a problem.

A Goodyear experimental rubber shows promise of being useful as a primer for high temperature coating systems.

WADC TR 56-80  
ASTIA Document No. AD 131056  
OTS Release

September 1957

SUBJECT: RESISTANCE OF PROTECTIVE COATINGS TO SYNTHETIC LUBRICATING AND HYDRAULIC FLUIDS  
INVESTIGATOR: Robert W. Damm  
George F. Salathe  
William A. McIntyre  
CONTRACT: AF33(616)-2919  
CONTRACTOR: The Sherwin-Williams Company  
ABSTRACT: This report consists principally of a detailed evaluation of the resistance of 56 coatings or coating systems to five synthetic lubricating and hydraulic fluids in conjunction with supplementary tests designed to expose these coatings to the maximum limits encountered by high speed aircraft under service conditions. Test work was conducted by The Sherwin-Williams Company in the Chicago Lacquer Laboratories' Aircraft Finishes Group. Results show the following candidate coatings to have the most promise in resisting maximum detrimental effects of synthetic fluids and accompanying environmental conditions:

## Skin Coatings

Candidate Coating No. C-1 - Andrew Brown,  
Brolite Synflex A-136 Black Enamel.

Candidate Coating No. C-6 - Sherwin-Williams,  
Experimental White System.

Candidate Coating No. C-55 - Sherwin-Williams,  
M49 W C4 System.

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## Engine Coatings

Candidate Coating No. C-31 - Pratt & Lambert,  
Heat Resistant Aluminum Enamel #7897.

WADC TR 56-80 Sup 1  
ASTIA Document No. AD 142030  
OTS Release

October 1957

**SUBJECT:** DEVELOPMENT OF PROTECTIVE COATINGS RESISTANT TO SYNTHETIC LUBRICATING AND HYDRAULIC FLUIDS

**INVESTIGATOR:** Robert W. Damm  
George F. Salathe  
William A. McIntyre

**CONTRACT:** AF 33(616)-2919

**CONTRACTOR:** The Sherwin-Williams Company

**ABSTRACT:** This report is a presentation of the work performed in developing both aircraft skin and engine coatings that are resistant to the detrimental effects of synthetic fluids and the accompanying environmental conditions encountered by modern high speed aircraft. As a result of this work the contractor evaluated a skin coating system and an engine coating and developed two engine coatings. The skin coating system is as follows: Primer, Sherwin-Williams Modified Wash Primer, P40 G C1; Top-coat, Sherwin-Williams White Modified Acrylic Enamel, M49 W C5. The three engine coatings are: Pratt & Lambert Aluminum Engine Coating #7897; Sherwin-Williams Aluminum Silicone Jet Engine Coating, M49 S C2; and Sherwin-Williams Aluminum Silicone Jet Engine Coating, M49 S C3. All these systems are capable of closely approaching complete resistance to the severe conditions involved.

WADC TR 56-250  
ASTIA Document No. AD 110410

October 1956

**SUBJECT:** SUMMARY OF DEVELOPMENT AND EVALUATION OF INSULATING TYPE REFRACTORY COATINGS

**INVESTIGATOR:** S. Sklarew  
C. A. Hauck  
A. V. Levy

**CONTRACT:** AF 33(616)-2957

**CONTRACTOR:** Marquardt Aircraft Company

**ABSTRACT:** Initial development of metal reinforced refractory coatings to provide thermal insulation to aircraft structural members operating in the temperature range 2000° to 3000°F is reported. Reinforced refractories 0.080 to 0.15-inch thick, providing thermal drops of 5° to 10°F per 0.001 inch of thickness have been successfully tested in small scale. These reinforced coatings averaged half the density of steel.

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WADC TR 56-338 Pt I  
ASTIA Document No. AD 142317  
OTS Release

January 1958

**SUBJECT:** BIBLIOGRAPHY OF ICE AND FROST CONTROL  
**INVESTIGATOR:** Thomas H. McConica, III  
**CONTRACT:** AF 33(616)-3156  
**CONTRACTOR:** Arctic Research Inc.  
**ABSTRACT:** This report reviews all unclassified publications relating to the control of ice and frost which could be obtained by this laboratory during the period July 1955 to June 1956. In preparing this bibliography an attempt was made to exclude publications not in the area of interest indicated by the title. Otherwise an unwieldy report would have resulted. Where abstracts or original reports could be reviewed a brief summary of the report is given. In addition, when known, the depository and call number of each referenced report is included. Depositories are coded as follows:

AMAU - Air University, Maxwell Air Force Base, Ala.  
ASTIA- Armed Services Technical Information Agency, Dayton, O.  
(Note: All "AD" and "ATI" numbers refer to ASTIA)  
DAL - Army Library, Washington, D. C.  
DLC - Library of Congress, Washington, D. C.  
DWB - Weather Bureau, Washington, D. C.

All publications have been classified according to subject matter and coded using the arrangement given on page v.

A review of the patent literature is included in Part II of this report.

WADC TR 56-338 Pt II  
ASTIA Document No. AD 142318  
OTS Release

January 1958

**SUBJECT:** BIBLIOGRAPHY OF ICE AND FROST CONTROL  
**INVESTIGATOR:** Thomas H. McConica, III  
**CONTRACT:** AF 33(616)-3156  
**CONTRACTOR:** Arctic Research Inc.  
**ABSTRACT:** This report constitutes an annotated bibliography of patents relating to the control of ice and frost on aircraft. All patents of interest in the U. S. Patent Office under classification list No. 244, subclass 134, up until July 1956, are included. Additional patents have been located through searching Chemical Abstracts, other bibliographies, etc. All patents have been classified according to subject matter and coded using the arrangement given on page v.

A review of unclassified publications is included in Part I of this report.

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WADC TR 57-158 Pt I  
ASTIA Document No. AD 142033  
OTS Release

October 1957

SUBJECT: DEVELOPMENT OF WHITE THERMALLY REFLECTIVE  
RAIN EROSION RESISTANT COATINGS

INVESTIGATOR: George K. Vogelsang

CONTRACT: AF 33(616)-3027

CONTRACTOR: Gates Engineering Company

ABSTRACT: Radomes and other coverings for housing radar antennae exposed to rain during high speed flight require maximum protection against rain erosion. This study is for the development of improved coatings for exterior plastic components, which will be rain erosion resistant, thermally reflective and have satisfactory weatherability.

White coatings formulated from polyacrylic rubber or Kel-F elastomer yield the best overall diffuse reflectances, while Neoprene has the best resistance to rain erosion.

A white Neoprene coating system has been developed which satisfies the major requirements of this program.

Studies are under way to improve the above coating system and to render it anti-static.

WADC TR 57-158 Pt II  
ASTIA Document No. AD 142185

December 1957

SUBJECT: DEVELOPMENT OF WHITE HEAT RESISTANT AND WHITE  
ANTI-STATIC RAIN EROSION RESISTANT COATINGS

INVESTIGATOR: George K. Vogelsang

CONTRACT: AF 33(616)-3027

CONTRACTOR: Gates Engineering Company

ABSTRACT: Numerous elastomeric coatings have been developed and tested for retention of thermal reflectance and resistance to rain erosion after 100 hours aging at 400°F. Although Neoprene affords the best resistance to rain erosion, it has limited heat resistance, while Kel-F elastomer is the exact opposite.

A method has been developed to utilize ionically conductive white coatings in conjunction with electron conductive coatings for the development of white anti-static rain erosion resistant coatings. Preliminary results have been very encouraging, but considerable additional work is required before white anti-static coating systems are developed to a point where they will consistently conform to the requirements set forth in Specification MIL-C-7439B.

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WADC TR 57-158 Pt III  
ASTIA Document No. AD 142286

January 1958

**SUBJECT:** DEVELOPMENT OF WHITE HEAT RESISTANT AND WHITE ANTI-STATIC RAIN EROSION RESISTANT COATINGS

**INVESTIGATOR:** George K. Vogelsang

**CONTRACT:** AF 33(616)-3027

**CONTRACTOR:** Gates Engineering Company

**ABSTRACT:** Improved white rain erosion resistant coatings were obtained by simultaneously using combinations of Neoprenes, titanium dioxides, the inclusion of moderate quantities of acid acceptors and stabilizers, and the use of extra quantities of antioxidants. White Neoprene rain erosion resistant coatings can be firmly bonded to polyester or epoxy glass fiber laminates using Primers Gaco KV-6006-N or KV-8510-E.

Improved electron conductive coatings for use in the "Series-Discharge" Anti-Static Coating System were prepared by blending white Neoprene non-conductive coating solutions with Neoprene conductive coatings, utilizing Vulcan XC-72, conductive carbon black. Several new white ionically conductive coatings were developed.

A number of "Series-Discharge" Anti-Static Coating Systems were found to possess and retain conductivities in the order of 0.5 and 15 megohms per square when measured after various conditions. Also, one hundred forty-five coating systems were found to possess and retain conductivities in the range of 10 to 100 megohms per square, when measured after similar conditions. The diffuse reflectances of these coatings are generally better than those of non-conductive white coatings, and their resistance to discoloration is substantially improved.

WADC TR 57-185  
ASTIA Document No. AD 131054  
OTS Release

September 1957

**SUBJECT:** CONDUCTIVITY TESTING OF ZINC DUST PIGMENTED COATINGS

**INVESTIGATOR:** Lt. D. S. Nantz

**ABSTRACT:** Zinc rich coatings have been in use for over 100 years as a protective pigment for steel. Since the effectiveness of these coatings are dependent upon the electrical conductivity from zinc particle to zinc particle and from zinc particle to the steel surface, it is important to have a good conductivity measurement test to determine the protective properties of any zinc rich coating system.

Several procedures were tried before a good conductivity test was developed. These conductivity tests can be then used as a scientific basis for formulation of zinc rich coatings which will provide good galvanic protection to steel.

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It was concluded that the conductivity test developed can be used to determine optimum zinc concentration, film thickness, and drying time for any given vehicle pigmented with zinc.

WADC TR 57-186  
ASTIA Document No. AD 142143  
OTS Release

November 1957

**SUBJECT:** THE EFFECTS OF NUCLEAR RADIATION ON MILITARY SPECIFICATION PAINTS  
**INVESTIGATOR:** 1/Lt. Lloyd A. Horrocks  
**ABSTRACT:** This report is an evaluation of the effects of gamma radiation and heat on several available types of specification and non-specification paints. Reflectance, adhesion, abrasion, humidity, and film condition data are presented on the effects of gamma radiation and heat on silicone-alkyd, fluorocarbon, alkyd, nitrocellulose, phenolic, and epoxy resin-based paints. Silicone-alkyd, alkyd, and phenolic resin-based paints appear to be satisfactory for use after exposure to  $1 \times 10^9$  roentgens.

WADC TR 57-195  
ASTIA Document No. AD 142146  
OTS Release

November 1957

**SUBJECT:** TEST METHODS FOR MAGNESIUM SURFACE TREATMENTS  
**INVESTIGATOR:** Frank W. Pfohl  
Howard T. Francis  
**CONTRACT:** AF 33(616)-2855  
**CONTRACTOR:** Armour Research Foundation  
**ABSTRACT:** A study was made of test methods whereby the quality of magnesium surface treatments could be determined. Methods which measured the corrosion-inhibiting power of the coating were found unsatisfactory. A method based on measurement of paint adherence after exposure to a corrosive environment, however, was found quite promising. The latter approach evolved the "Gauze-Peel Test", in which a gauze strip is embedded in the organic coating. After exposure to accelerated corrosion, the gauze is peeled from the surface. Both quantitative (peel strength) and qualitative (visual examination of the stripped area) evaluations are easily made. An important observation made during preparation of experimental specimens was that contact between magnesium and steel during Dow 7 treatment leads to rapidly deteriorating painted surfaces.

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WADC TR 57-296  
ASTIA Document No. 150985  
OTS Release

February 1958

**SUBJECT:** MARKING MATERIALS FOR RUBBER, PLASTIC FILMS AND  
COATED FABRICS  
**INVESTIGATOR:** Harry N. Vosmer  
**CONTRACT:** AF 33(616)-3624  
**CONTRACTOR:** National Cash Register Company  
**ABSTRACT:** A standard procedure for marking ink evaluation was developed based on the target properties of the contract and the laboratory techniques established by the contractor.

Six inks from commercial sources were found to be acceptable for use as marking inks on the majority of the coated fabrics and plastic films.

Four inks developed in the laboratories of NCR proved to be acceptable. Two of these are based on acrylic resins, and two on a polyvinyl butyral resin system.

None of the ten inks recommended are entirely satisfactory for use on polyethylene or Cotton-vinyl coated, Type II, Class C, Specification MIL-F-4143, since they fail the adhesion and immersion tests.

Titanium adhesion promoters were investigated but were found to contribute little toward the improvement of adhesion on the plastic films and coated fabrics.

Work with treated polyethylene indicated a slight increase of adhesion over the untreated material. Printing on polyethylene still remains a serious problem, since the inks developed and evaluated in this report have not proved satisfactory on this material.

WADC TR 57-317  
ASTIA Document No. AD 131070

September 1957

**SUBJECT:** THE DEVELOPMENT OF TESTING PROCEDURES AND THE EVALUATION  
OF COATINGS FOR TANTALUM, GRAPHITE, NICKEL AND COPPER  
**INVESTIGATOR:** W. A. Spraker J. L. Harp  
A. E. Weller F. L. Bagby  
R. E. Hess  
**CONTRACT:** AF 33(616)-3227  
**CONTRACTOR:** Battelle Memorial Institute  
**ABSTRACT:** Recent developments in the field of aeronautics have focused attention on the behavior of materials under conditions of high heat flux and high temperature. A number of materials, whose physical properties are of interest, must be protected from erosion and oxidation when exposed to a flow of high temperature air. The resistance of these materials and their coatings to thermal shock is also important.

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This report summarizes the results of the first year of a program to evaluate the resistance of a number of coatings for copper, nickel, tantalum, and graphite to erosion, oxidation, and thermal shock under high temperature service conditions. Specimens were tested in the throat of a hydrogen-oxygen rocket motor which could produce a maximum heat flux of approximately 3000 Btu/sec sq ft, and a maximum temperature of approximately 5500F.

The results of the research program indicate that several coatings have properties of promise. These coatings include Rokide A, chromium, and chromium-nickel coatings for copper; Rokide A and chromium coatings for nickel; and a Rokide C coating for graphite. In general, flame sprayed and electrodeposited types of coatings appeared to be the most successful. None of the coatings for tantalum that were tested were successful. The failure of coatings on tantalum appears to be the result of oxidation of the tantalum through the coatings; a successful tantalum coating, if developed, should have a low porosity. Tests of coatings applied by vapor deposition indicated that these coatings have relatively poor adhesion and are subject to erosion.

WADC TR 57-342  
ASTIA Document No. AD 142080  
OTS Release

October 1957

SUBJECT: EFFECTIVE THICKNESS OF CHROMIUM PLATE ON THE  
SENSITIVITY OF MAGNETIC PARTICLE INSPECTION

INVESTIGATOR: W. Steindorf  
B. Cohen

ABSTRACT: A survey was conducted on small cracked rods to determine the size crack which can be detected by magnetic particle inspection. Chromium plating 2.2, 4.4, and 6.6 mils thick were successively plated to these rods and the rods were examined after each plating. Scotch tape transfers were taken and studied in conjunction with the subsequently measured cracks. The summation of results indicate that 4.5 mils is a practical limitation of the effective depth of chromium plate for magnetic particel inspection of the plated body.

WADC TR 57-435  
ASTIA Document No. AD 142025

October 1957

SUBJECT: STUDY AND DESIGN OF SUPERSONIC ROTATING ARM RAIN  
EROSION TEST APPARATUS

INVESTIGATOR: John L. Beal  
Norman E. Wahl

WADC TR 53-373 Sup 5

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**CONTRACT:** AF 33(616)-3267  
**CONTRACTOR:** Cornell Aeronautical Laboratory, Inc.  
**ABSTRACT:** The feasibility of building a 2500 mile per hour rotating arm, rain erosion, test apparatus based upon economic aspects and engineering problems was investigated. It was found that such a rain erosion test facility was practicable and one was designed which consisted of a large, refrigerated chamber evacuated to 0.01 atmosphere pressure in which the test blade, 56 feet in diameter, would rotate.

The problems and calculations leading to the design, as well as the cost of the equipment of a rotating arm test apparatus for three speeds (i.e. 2200, 2270 and 2500 mph) are reviewed.

WADC TR 57-752  
ASTIA Document No. AD 151012

March 1958

**SUBJECT:** THE INVESTIGATION OF THERMOSTABILITY OF INHIBITIVE PIGMENTS  
**INVESTIGATOR:** Edward R. Allen  
Harold V. Lindstrom  
**CONTRACT:** AF 33(616)-5290  
**CONTRACTOR:** Rutgers, The State University  
**ABSTRACT:** This report is the result of a three months study of the heat stability of some corrosion inhibiting alkaline earth chromates. Certain chromates were collected, analyzed and compared as to behavior upon heating. As indicated in the literature, the presence of certain impurities, such as calcium oxide, tends to foster decomposition at high temperatures. The result is that the yellow chromate form is changed in part to a green chromic form with the loss of oxygen.

The behavior of a highly purified form of zinc phosphate was studied for its possible application as a corrosion inhibiting pigment.

WADC TR 58-4  
ASTIA Document No. AD 142330

February 1958

**SUBJECT:** DEVELOPMENT AND FABRICATION OF RAINFALL SIMULATION EQUIPMENT FOR SUPERSONIC WHIRLING ARM TEST APPARATUS  
**INVESTIGATOR:** Roy R. Lapp  
Norman E. Wahl  
**CONTRACT:** AF 33(616)-5316  
**CONTRACTOR:** Cornell Aeronautical Laboratory, Inc.  
**ABSTRACT:** The development and method of calibration of spray nozzles for rainfall simulator, operating in a vertical plane, are reviewed. The design of the spray ring and its fabrication are described.

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WADC TR 58-11  
ASTIA Document No. AD 150970  
OTS Release

February 1958

SUBJECT: PREPARATION OF PROTECTIVE COATINGS BY  
ELECTROPHORETIC METHODS

INVESTIGATOR: Allen C. Werner  
Robert J. Abelson

CONTRACT: AF 33(616)-5002

CONTRACTOR: Vitro Laboratories

ABSTRACT: An investigation was conducted of the preparation of oxidation-resistant coatings for molybdenum by electrophoretic methods. Multi-layer coatings of 80% nickel - 20% chromium and nickel-bonded columbium and titanium carbide provided good oxidation resistance and good erosion resistance. The ballistic impact resistance requirement was fulfilled by inclusion of a 50% dense, 80% nickel - 20% chromium layer. The effects of sintering and coating thickness on resistance to thermal shock were noted. Attempts to incorporate an intergranular diffusion barrier alloy layer for oxidation resistance at 2000°F proved unsuccessful, although a gold-chromium diffusion barrier layer proved resistant at 1800°F. The coating technique has been refined to permit application of a single layer in one operation instead of two.

## CORROSION

WADC TN 55-212 Sup 2  
ASTIA Document No. AD 131017

September 1957

SUBJECT: RESISTANCE OF ORGANIC FINISHES TO ROCKET BLAST

INVESTIGATOR: Sam E. Collis

ABSTRACT: Forty-seven test panels with various organic and inorganic coating systems were exposed to an impinging blast from free fired, T-214 Rockets. None of the coating systems offered complete protection for the metal. The systems offering the best protective properties were EC 843 (Minnesota Mining and Manufacturing Company) applied over zinc chromate primer or over wash primer-zinc chromate primer. Ceramic coatings or drying oil enamels and varnishes applied over zinc chromate primer, were found to rank second in protective properties. The other coating systems, which were, in most cases, applied without a corrosion inhibiting primer, provided only fair protection.

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WADC TR 56-380  
ASTIA Document No. AD 130820  
OTS Release

June 1957

**SUBJECT:** INVESTIGATION OF THE APPLICABILITY OF HIGH FREQUENCY SOUND WAVES (ULTRASONICS) FOR CLEANING OF PRECISION PARTS

**INVESTIGATOR:** Oskar E. Mattiat  
Paschal P. Zapponi

**CONTRACT:** AF 33(616)-3011

**CONTRACTOR:** Clevite Research Center

**ABSTRACT:** Grease and preservative compounds on bearings are easily removed by several methods. The important soils, AN-C-124 preservative compound and MIL-G-3278 grease are removed by a thorough spray treatment with Stoddard solvent or an ultrasonic treatment under various conditions using trichloroethylene as the coupling liquid.

The effect of the ultrasonic factors, frequency, intensity, pulse power, coupling fluid and container for holding the parts, upon the rate of removal of field and synthetic soils from complex parts with small openings is determined. Two new methods of study are developed for evaluating available ultrasonic systems and factors, namely, the steel-removal and probe methods.

Results obtained with these methods show that accessible soils of all kinds are easily removed by any of the ultrasonic systems studied; however, inaccessible soils, such as steel particles in bearings and grease soils in blind holes require high sonic intensities and a coupling fluid with optimum cavitating and solubility or dispersability properties for the particular soil.

Low frequency systems appear to be more effective than high frequency systems for removing most types of soils investigated. However, inability to control intensity at the point of cleaning for the high frequency systems make this evaluation uncertain.

The use of pulsed power does not improve the removal rates of the soils studied.

With the possible exception of an aqueous solution for the removal of steel particles from bearings, trichloroethylene is the most effective coupling fluid tested for the various soils.

Data submitted also show that while a coarse mesh basket for containing parts is not detrimental to soil removal at low frequencies it is at high frequencies.

It is shown by erosion measurements on bearings that the adverse effect of ultrasonic treatment on the starting torque is caused by cavitation erosion; however, the amount of erosion is so small that it

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may be neglected for the short cleaning time normally required.

Recommendations are made for the cleaning of bearings and other precision parts contaminated with all of the various soils submitted by the contracting agency.

WADC TR 57-302

April 1958

ASTIA Document No. AD 151116

SUBJECT: INVESTIGATION OF CORROSION INHIBITORS FOR FUMING  
NITRIC ACID

INVESTIGATOR: William H. Bergdorf  
Edward J. Kinsey, Jr.

CONTRACT: AF 33(616)-3056

CONTRACTOR: Bell Aircraft Corporation

ABSTRACT: The major effort of this investigation was to develop a corrosion inhibitor for fuming nitric acid that has corrosion-inhibiting and scale and sludge-formation properties superior to those of hydrofluoric acid. Ammonium hexafluorophosphate in amount equivalent to 0.30% by weight hydrofluoric acid was found to provide better inhibition to the corrosion of 61S-T6 aluminum alloy, and 17-7PH and AISI Type 304L stainless steels than 0.70% by weight hydrofluoric acid under the test conditions. Optimum inhibitor concentrations for Types I and III specification acids, and inhibitor depletion rates for eight inhibitors including hydrofluoric acid were determined. Effects of inhibitor up to five times optimum inhibitor concentration of each of the several inhibitors were measured in terms of slush or freezing point of inhibited acid media which had been subjected to wide variation in temperature and short time storage conditions. Measurements of the mechanical properties of welded specimens of the aluminum and stainless steel alloys that had been immersed in the liquid and vapor phases of eight inhibited acid systems for thirty days at 120°F showed that no significant change in properties was effected by these exposure conditions. Film-formation studies were conducted on aluminum and stainless steel specimens that had been exposed to the liquid and vapor phases of the several inhibited systems and subsequently rinsed in water and methylene chloride. Both rinses removed the inhibited acids from the specimens but methylene chloride failed to remove corrosion products.

Hydrofluoric acid phosphoric acid plus hydrofluoric acid, and ammonium hexafluorophosphate at optimum concentrations dissolved in specification Types I and III acid media were tested in vessels fabricated from the aluminum and stainless steel alloys for a period of 60 days at 70°, 120°, and 160°F. Ammonium hexafluorophosphate produced the greatest pressures during the test period. This inhibitor, however, suffered the smallest loss in concentration and in general provided the best inhibition.

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WADC TR 57-540  
ASTIA Document No. AD 142263  
OTS Release

December 1957

**SUBJECT:** THE DESIGN AND CONSTRUCTION OF A SPECIAL TEST  
FIXTURE FOR THE STATIC EVALUATION OF THE CORROSIVE  
EFFECTS OF BORON OXIDE AT HIGH TEMPERATURES

**INVESTIGATOR:** Charles R. Andrews

**CONTRACT:** AF 33(616)-3737

**CONTRACTOR:** University of Dayton Research Center

**ABSTRACT:** The design and construction of a specialized high-temperature corrosion test fixture is described herein. The fixture is arranged to provide for the long-time exposure of test materials to static environments consisting of cyclic immersion in molten boron oxide ( $B_2O_3$ ) at temperatures up to  $3000^{\circ}F$  under closely controlled conditions. Features of the test fixture include an extensive electrical heating control system and an automatic specimen cycling mechanism. The fixture can be operated on a continuous basis with a minimum of attention.

A description of the operating characteristics of the fixture and details of operating procedures are included in this report. A list of engineering drawings pertaining to the test fixture, as well as an outline of maintenance procedures to be followed, are contained in appendices.

WADC TR 57-662  
ASTIA Document No. AD 155516  
OTS Release

May 1958

**SUBJECT:** THIN METAL FILMS AS CORROSION INDICATORS

**INVESTIGATOR:** Richard B. Belser  
Frank E. Hankinson

**CONTRACT:** AF 33(616)-3879

**CONTRACTOR:** Georgia Institute of Technology

**ABSTRACT:** In order to investigate the feasibility of using thin metal films deposited on glass or plastic substrates as integrating indicators of corrosive conditions, the corrosion rates of films of nine metals exposed to air atmospheres of controlled temperature and humidity have been studied. Of the metals iron, manganese, copper, nickel, cobalt, strontium, calcium, magnesium and zinc, only films of iron and manganese proved worthy of more than a preliminary survey. A more intensive study of the corrosion properties and rates of iron and manganese films and of the bimetal pairs iron and copper and iron and gold was undertaken.

This investigation disclosed the important role in metal film corrosion of the absorbed gas layer normally present on glass substrates. This layer furnished the oxygen necessary for small particles of the oxides of iron to form at random sites in the film. These subsequently became elements of electrolytic cells in which the iron film acted as the anode in

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the presence of air at 25°C and 70 percent relative humidity. Destructive oxidation of the film thereafter proceeded by electrochemical processes.

Rates of corrosion for iron films were controllable to a degree by removal of the absorbed gas layer by pre-deposition heating of the substrate to 400°C or by more complete combination of the absorbed layer with the metal of the film induced by post-deposition heating of the film. The former method reduced corrosion rates because of the removal of the oxygen supply for the formation of oxide nuclei, and the latter method increased the corrosion rate because of the greater number of oxide nuclei formed.

Control of the corrosion rates of iron films under specific conditions of temperature and humidity appears feasible by control of the absorbed gas layer and by introduction of cathodic particles either in the form of oxides or in the form of small volumes of a more electrically positive metal deposited at selected sites.

## CREEP

WADC TR 57-96  
ASTIA Document No. 151114  
OTS Release

April 1958

SUBJECT: A SUMMARY OF COMPRESSIVE-CREEP CHARACTERISTIC  
OF METAL COLUMNS AT ELEVATED TEMPERATURES

INVESTIGATOR: R. L. Carlson  
G. K. Manning

CONTRACT: AF 33(616)-3317

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: A summary of an extensive study of the creep buckling of metal columns is presented. The column behavior prior to collapse is described and the column action at the time of buckling is interpreted in terms of stability.

Solutions to creep buckling are discussed. It is concluded that in spite of certain limitations, Shanley's time-dependent tangent-modulus method has several practical advantages over available analytical solutions.

An application of the time-dependent tangent-modulus method

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to four structural metals indicates that estimates are consistently conservative for small values of column imperfection. Imperfection variations were generally observed to have a very marked effect on the column lifetime.

The possible existence of a lower column-load limit below which time-dependent collapse will not occur is discussed. From a rational consideration of known creep behavior it is concluded that there may be a temperature below which finite lower limits exist and above which the lower limit is zero.

WADC TR 57-104  
ASTIA Document No. AD 130762  
OTS Release

May 1957

SUBJECT: ANALYSIS OF DYNAMIC CREEP CONSIDERING STRAIN RATE EFFECTS  
INVESTIGATOR: F. H. Vitovec  
CONTRACT: AF 33(616)-2803  
CONTRACTOR: University of Minnesota  
ABSTRACT: The dynamic creep behavior of a number of materials is discussed and classified. The creep properties under simultaneous action of static stress and alternating stress have been derived by several authors from the creep rate-stress relationship under static load. The use of this so derived stress range diagram for creep as a basis for determination of acceleration or retardation of dynamic creep is emphasized. Retardation of creep is attributed to aging effects, and to strain rate effects caused by alternating stress frequency. Acceleration of creep is attributed to metallurgical changes, and overtemperature at very high frequencies. Special consideration is given to the effect of superposition of strain rate and creep rate. The theoretical analysis of the strain rate effects in dynamic creep, presented here, conform with the experimental observation that retardation of creep increases with increasing temperature, and with increasing alternating stress frequency if metallurgical effects are negligible.

WADC TR 57-150 Pt I  
ASTIA Document No. AD 150956  
OTS Release

February 1958

SUBJECT: EFFECT OF PRIOR CREEP ON MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL METALS (2024-T86 Aluminum and 17-7 PH Stainless)  
INVESTIGATOR: Jeremy V. Gluck  
Howard R. Voorhees  
James W. Freeman  
CONTRACT: AF 33(616)-3368  
CONTRACTOR: The University of Michigan

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**ABSTRACT:** Tests have been performed on two typical aircraft structural sheet alloys in an investigation to study changes in mechanical properties brought about by prior exposure to elevated-temperature creep conditions. Specimens of 2024-T86 aluminum alloy and 17-7PH (TH 1050) precipitation hardening stainless steel were exposed for times of 10, 50, and 100 hours at stresses giving up to 3% total deformation, using temperatures of from 350° to 500°F for the 2024-T86 and 600° to 900°F for the 17-7PH.

Following the exposures, short-time tensile, compression, or tension-impact tests were run at either room temperature, the temperature of exposure, or both. The results indicate that the short-time strength of structural materials may be either raised or lowered. The changes in properties may approach as much as 50 percent of the original value. The direction of the change depends on the material, test temperature, creep exposure conditions, and property being measured.

From the standpoint of the structures designer the most important changes found to date are a large drop in strength for 2024-T86 after prior creep exposure for times of from 10 to 100 hours and an apparent decline in the room temperature ductility of 17-7PH (TH 1050 condition) after prior creep exposure for 100 hours at temperatures near 600°F.

WADC TR 57-150 Pt II  
ASTIA Document No. AD 151115  
OTS Release

April 1958

**SUBJECT:** EFFECT OF PRIOR CREEP ON MECHANICAL PROPERTIES  
OF AIRCRAFT STRUCTURAL METALS  
Part II - 17-7PH Alloy (TH 1050 Condition)

**INVESTIGATOR:** Jeremy V. Gluck  
Howard R. Voorhees  
James W. Freeman

**CONTRACT:** AF 33(616)3368  
**CONTRACTOR:** The University of Michigan

**ABSTRACT:** A study was carried out of the effect of exposure to elevated-temperature creep conditions on subsequent mechanical properties of aircraft structural metals. The present report considers a precipitation hardening stainless steel, 17-7PH (TH 1050 condition). Exposures were conducted for times of 10, 50, or 100 hours either unstressed or at stresses giving up to 3-percent total deformation at temperatures between 600° and 900°F.

Following the exposures, short-time tensile, compression, or tension-impact tests were run at either room temperature or the temperature of exposure. The principal effects found were a loss in ductility in tensile tests and a substantial loss in compression yield strength after exposure to creep at 600°F. In general, remarkably little change in the other mechanical properties was found as a result of exposure to creep, with any such changes primarily confined to increases in strength.

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WADC TR 57-297  
ASTIA Document No. AD 130802  
OTS Release

June 1957

**SUBJECT:** DESCRIPTIONS OF SOME CURRENT METHODS FOR DETERMINING  
CREEP PROPERTIES UNDER COMPRESSIVE, BEARING AND  
SHEAR TYPE OF LOADING

**INVESTIGATOR:** E. L. Horne

**ABSTRACT:** This report gives descriptions of several methods currently used for evaluating creep behavior in compressive, bearing and shear types of loading. Four methods for compressive creep testing are described. Two methods for bearing creep and four methods for shear creep, respectively, are also given. Only unclassified and non-proprietary information are discussed. Special mention is made of methods developed under WADC sponsorship. Future panel action will be based on recommendations by panel members and the organizations they represent resulting from a study of methods described in this report.

## DESIGN CRITERIA

AF TR 6517 Pt V  
ASTIA Document No. AD 142218  
OTS Release

December 1957

**SUBJECT:** DETERMINATION OF THE TENSILE, COMPRESSIVE AND  
BEARING PROPERTIES OF FERROUS AND NONFERROUS  
STRUCTURAL SHEET MATERIALS AT ELEVATED TEMPERATURES

**INVESTIGATOR:** Donald E. Miller

**CONTRACT:** AF 33(616)-3145

**CONTRACTOR:** Armour Research Foundation

**ABSTRACT** To provide data for the establishment of design criteria, the compressive and bearing properties of six aluminum alloys, three magnesium alloys, two titanium alloys, two stainless steels, and one alloy steel have been determined at elevated temperatures. Tensile data were also obtained for all materials except the stainless steels. Each material was tested at several temperatures as follows: 2014-T6, 7075-T6, and X7178-T6 aluminum alloys and M1-H24, AZ31-H24, and AZ31-0 magnesium alloys at 200°, 300°, 400°, 500°, and 600°F; 2024-T3 aluminum alloys at 212°, 300°, 400°, 500°, 600°, and 700°F; 2024-T81 and 2024-T86 aluminum alloys at 200°, 300°, and 400°F; annealed titanium, cold-rolled titanium, Type 301 half-hard stainless steel, Type 302 annealed stainless steel, and SAE 4130 alloy steel at 400°, 600°, 800°, and 1000°F. Specimens of all materials were aged for 0.5 hr at test temperature prior to testing.

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The test materials used in the program were leftover portions of sheets which had been tested previously. They had been in storage for periods ranging from 3 to 5 yrs. Analysis of the test data suggested that long room temperature aging has a substantial effect on the elevated temperature properties of certain hard-rolled and heat-treated alloys. The properties of the fully annealed materials, however, appeared to be unaffected by protracted aging at room temperature.

Test specimens, equipment, and procedures are described in detail, and test results are presented in both tabular and graphical form.

WADC TR 55-150 Pt V  
ASTIA Document No. AD 142069

October 1957

SUBJECT: MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS  
Part V. The Conventional Short-Time, Elevated-  
Temperature Properties of Selected Stainless  
Steels and Super Alloys

INVESTIGATOR: Ronald J. Favor  
William P. Achbach  
Walter S. Hyler

CONTRACT: AF 33(616)-2303

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: Presented in this report is a compilation of data on the conventional short-time, elevated-temperature properties of selected corrosion-resistant and high-temperature alloys applicable to airframe and missile fabrication. The resultant recommended design data obtained in this study have been presented in such form as to be directly applicable to the ANC-5 Bulletin (issued by the Air Force-Navy-Civil Panel) on "Strength of Metal Aircraft Elements".

WADC TR 55-150 Pt VI  
ASTIA Document No. AD 142043  
OTS Release

October 1957

SUBJECT: MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS  
Part VI - The Conventional Short-Time Elevated-  
Temperature Properties of Selected Light Alloys

INVESTIGATOR: W. P. Achbach  
R. J. Favor  
W. S. Hyler

CONTRACT: AF 33(616)-3965

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: Described in this report is a compilation of data on the conventional short-time elevated-temperature properties of selected light alloys applicable to airframe and missile fabrication. The resulting recom-

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mended design data obtained in this study has been presented in such form as to be directly applicable to Bulletin ANC-5 (issued by the Air Force-Navy-Civil Panel) on "Strength of Metal Aircraft Elements".

WADC TR 55-150 Pt VII  
ASTIA Document No. AD 142064

October 1957

**SUBJECT:** MATERIALS-PROPERTY-DESIGN CRITERIA FOR METALS  
Part VII. The Conventional Short-Time, Elevated-Temperature Properties of Selected Low-and Medium-Alloy Steels

**INVESTIGATOR:** Ronald J. Favor  
William P. Achbach  
Walter S. Hyler

**CONTRACT:** AF 33(616)-2303

**CONTRACTOR:** Battelle Memorial Institute

**ABSTRACT:** Presented in this report is a compilation of data on the conventional short-time, elevated-temperature properties of selected low-and medium-alloy steels applicable to airframe and missile fabrication. The resulting recommended design data obtained in this study have been presented in such form as to be directly applicable to the ANC-5 Bulletin (issued by the Air Force-Navy-Civil Panel) on "Strength of Metal Aircraft Elements".

WADC TR 55-199 Pt III  
ASTIA Document No. AD 142003  
OTS Release

September 1957

**SUBJECT:** TENSILE PROPERTIES OF AIRCRAFT-STRUCTURAL METALS  
AT VARIOUS RATES OF LOADING AFTER RAPID HEATING

**INVESTIGATOR:** William P. Roe  
J. Robert Kattus

**CONTRACT:** AF 33(616)-424

**CONTRACTOR:** Southern Research Institute

**ABSTRACT:** The purpose of the present phase of this program was to determine the effects of variations in strain rate and holding time at temperature on the tensile properties of several aircraft-structural metals after they had been heated within 10 sec to test temperatures approaching the melting points of the alloys involved. Major emphasis was placed on an accurate determination of these effects on the ultimate tensile strength and 0.2%-offset yield strength. The modulus of elasticity, percent elongation, and proportional limit were determined with less accuracy as by-products of the data for the purpose of establishing trends. This investigation covered strain rates from 0.00005 in./in./sec to 1.0 in./in./sec, holding times at test temperature from 10 sec to

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1800 sec, and the following sheet materials over the range of test temperatures indicated:

Annealed Stellite-25, 1600°F - 2250°F  
Heat-treated Inconel-X, 1600°F - 2250°F  
Full-hard 301 stainless steel, 1600°F - 2250°F  
Annealed Al10-AT titanium alloy, 1200°F - 2770°F  
Alclad 2024-T3 aluminum alloy, 800°F - 900°F

In general, the ultimate tensile strength and 0.2%-offset yield strength of all test materials increased appreciably with increasing strain rates at each test temperature. With only minor variations, the apparent modulus of elasticity showed a consistently increasing trend with increasing strain rates. The same was true for the percent elongation with the exception of annealed Al10-AT titanium alloy, which showed a decreasing trend with increasing strain rates.

Because of atmospheric attack, the properties of the Al10-AT alloy deteriorated with increasing times at the higher test temperatures. Holding times at test temperature had no significant effect on any of the properties of the other materials investigated.

At 2250 F, annealed Stellite-25 had the greatest strength, followed by heat-treated Inconel-X, annealed Al10-AT titanium alloy, and full-hard 301 stainless steel.

The oxidation resistance of annealed Stellite-25 and heat-treated Inconel-X was good at 2250°F, full-hard 301 stainless was fair to poor, and annealed Al10-AT titanium alloy was poor. Alclad 2024-T3 aluminum alloy showed good oxidation resistance at 900°F.

WADC TR 56-44  
ASTIA Document No. AD 130777  
OTS Release

June 1957

SUBJECT: ANALYTICAL METHODS FOR DETERMINING SPECIFIC DAMPING ENERGY CONSIDERING STRESS DISTRIBUTION  
INVESTIGATOR: E. R. Podnieks  
B. J. Lazan  
CONTRACT: AF 33(616)-2803  
CONTRACTOR: University of Minnesota  
ABSTRACT: Various energy and energy ratio units for expressing the damping properties of materials are discussed. Specific damping energy (in-lb/cu in/ cycle) is proposed as the most useful unit for comparing materials and performing engineering calculations. Analytical methods are presented for converting the total or average damping energy of a specimen to its specific damping. The role of the volume-stress function in this analysis is discussed in general and specific cases are illustrated. Additional factors involved in converting such energy ratio units as

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logarithmic decrement are analyzed. Data on the dimensionless damping and strain energy integrals are included for a range of conditions to facilitate data conversion. Caution is urged in interpreting damping data in which stress distribution and other factors, analyzed in this report, are not fully considered.

WADC TR 56-340  
ASTIA Document No. AD 131069  
OTS Release

September 1957

SUBJECT: DETERMINATION OF TENSILE, COMPRESSIVE, BEARING, AND SHEAR PROPERTIES OF FERROUS AND NON-FERROUS STRUCTURAL SHEET METALS AT ELEVATED TEMPERATURES

INVESTIGATOR: John V. Melonas  
J. Robert Kattus

CONTRACT: AF 33(616)-2741 and AF 33(616)-3224

CONTRACTOR: Southern Research Institute

ABSTRACT: The tensile, compressive, bearing, and shear properties of the following sheet metals were determined at various temperatures after exposure times from 1/2 to 1000 hours at the test temperature:

1. AISI-4130 alloy steel, 150, 000 psi nominal strength level
2. AISI-4130 alloy steel, 180, 000 psi nominal strength level
3. AISI-4130 alloy steel, 200, 000 psi nominal strength level
4. Al10-AT titanium alloy
5. HK31-H24 magnesium alloy
6. Type 301 stainless steel, full-hard

The magnesium alloy was tested over a temperature range from 75°F to 600°F, whereas the other test metals were tested at temperatures to 1000°F.

The test results show that all strength and modulus properties decreased to some degree with increasing temperature over the range studied. Increasing exposure time, which had no significant effects on modulus-of-elasticity values, resulted in some decreased strength properties in all test metals at the higher test temperatures but had no appreciable effect at the lower temperatures. In general, the effects of variations in temperature were greater than the effects of variations in exposure time.

A correlation and comparison of the test results indicated some crude relationships between various properties. These relationships provide a means of calculating rough estimates of certain properties on the basis of a different property determination. The accuracy of these calculations decreases as the range of materials and conditions, to which they are applied, increases. For the entire range of materials and conditions used in this work the accuracy of the various property relationships ranged from ±11% to ±55%. Precise and reliable data on the tensile, compressive, bearing, and shear properties of aircraft-structural materials can be obtained only by testing under the desired conditions.

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ELECTRODEPOSITION

WADC TN 57-178  
ASTIA Document No. AD 130821

June 1957

SUBJECT: EFFECT OF SHOT-PEENING PRIOR TO CHROMIUM PLATING ON THE FATIGUE STRENGTH OF HIGH STRENGTH STEEL

INVESTIGATOR: Bennie Cohen

ABSTRACT: The effect of shot-peening prior to chromium plating on the fatigue strength of high strength steel has been studied in an effort to minimize the drastic drop in fatigue strength, approximately 50%, which occurs as a result of chromium plating. Shot-peened, chromium plated steel, 220,000 psi UTS, had an endurance limit 12% higher than the bare unshot-peened steel. Shot-peened, chromium plated steel, 220,000 psi UTS, which was baked after plating, had an endurance limit 18% higher than the bare unshot-peened steel. Shot-peened, chromium plated steel, 280,000 psi UTS, showed only a slight decrease in fatigue strength in both the baked and unbaked conditions when compared to the fatigue strength of the unshot-peened bare steel.

WADC TR 57-514  
ASTIA Document No. AD 142316  
OTS Release

January 1958

SUBJECT: DEVELOPMENT OF ELECTROPLATING PROCESSES TO ELIMINATE HYDROGEN EMBRITTELEMENT IN HIGH-STRENGTH STEEL

INVESTIGATOR: J. E. Chilton

CONTRACT: AF 33(616)-3429

CONTRACTOR: Stanford Research Institute

ABSTRACT: The embrittlement of cadmium plated high-strength steels e.g., (SAE 4340) by hydrogen is of serious concern to the Air Force. Unexpected failures of highly stressed components have been experienced. It has been found that cadmium plating from a sulfamate, a perchlorate, or a fluoborate bath with addition agents can be accomplished without embrittling the steel, but that the quality and adherence of the cadmium plate emerging from these baths, as compared with the cadmium plate from a cyanide bath, indicates the need for further evaluation before use.

In order to evaluate the effects of various degrees of hydrogen embrittlement on the steel, a sensitive mechanical test method was required. The important criteria were that the test coupon be of such geometry that it could be plated uniformly and that the test would evaluate only the effect of hydrogen embrittlement. Simple bend tests and notched

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bar tensile tests have been commonly used for studying the embrittling effect. In this program, the bend test, torsion test, and static sustained load beam test were investigated. A static sustained load beam test, using a 9- x 1/4- x 1/2-inch specimen with the grain transverse to the length was adopted. The SAE 4340 specimens were heat treated to 280,000 to 300,000 pounds per square inch tensile strength and the fracture time, under a given stress, was taken as a measure of the degree of hydrogen embrittlement.

It was found that replicate specimens show a large deviation in fracture times in spite of the care that was given to the selection, preparation, and testing techniques. These deviations are due to the normal defects and variables that are present in commercial, air melted, aircraft quality steel.

WADC TR 58-5 Pt I  
ASTIA Document No. AD 151078

March 1958

SUBJECT: COATINGS OF NICKEL-ALUMINUM ALLOYS PREPARED BY ELECTROLYSIS  
INVESTIGATOR: Dwight E. Couch  
Jean H. Connor  
CONTRACT: AF 33(616)57-10  
CONTRACTOR: National Bureau of Standards  
ABSTRACT: The electrodeposition of nickel-aluminum alloy coatings for the protection of Thermold J Alloy steel was investigated because of certain properties of the alloy, namely, extreme hardness and oxidation resistance. The alloy was formed by plating aluminum upon a layer of nickel. When the fused salt baths operated above the melting point of aluminum, the alloy was formed directly. When the aluminum was deposited at lower temperatures, the samples were later heated to form the alloy by diffusion. Oxidation data and salt spray corrosion resistance of the alloy-plated steel are given. Optimum protection of the steel was afforded by the alloy formed at 700°C, consisting of 2 mils of nickel and 0.2 mil of aluminum.

## ELECTRONIC MATERIALS

WADC TN 57-241  
ASTIA Document No. AD 131074

September 1957

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**SUBJECT:** ELECTRICAL AND ELECTRONIC MATERIALS FOR HIGH SPEED  
MISSILES AND AIRCRAFT

**INVESTIGATOR:** Dr. R. N. Evans

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

WADC TN 58-54  
ASTIA Document No. AD 151183

April 1958

**SUBJECT:** HIGH TEMPERATURE ELECTRICAL INSULATION MATERIALS FOR  
MISSILES AND AIRCRAFT

**INVESTIGATOR:** Dr. R. N. Evans

**ABSTRACT:** A review on the development of special-purpose electrical materials is presented together with new areas of materials development which may profitably be exploited. The environmental factors to which high speed aircraft and missiles are exposed are discussed. A materials program designed to meet these measurements is presented and its relation to programs on end-item development is pointed out.

## FATIGUE

WADC TR 56-611  
OTS Release

March 1957

**SUBJECT:** INVESTIGATION OF MATERIALS FATIGUE PROBLEMS

**INVESTIGATOR:** H. N. Cummings  
F. B. Stulen  
W. C. Schulte

**CONTRACT:** AF 33(616)-2876

**CONTRACTOR:** Curtiss-Wright Corporation

**ABSTRACT:** The studies of high-hardness steels that were begun under

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# Contracts

Contract AF 33(616)-493 were continued. S-N curves were obtained for SAE 4340 air-melted steel of 230 ksi UTS and vacuum melted steel of 190 ksi UTS. Several steels of 300 ksi UTS were investigated by Prot tests, and S-N curves were obtained for a 4350 steel of the same UTS. Studies of the relation of non-metallic inclusions to fatigue life and strength of the steels were continued and exploratory studies were made of crack initiation and propagation. The applicability of the Prot method of testing to four non-ferrous metals was also included in the work done under the present contract.

WADC TR 57-310  
ASTIA Document No. AD 142118  
OTS Release

November 1957

**SUBJECT:** THE EFFECT OF VARIOUS MACHINING PROCESSES ON THE REVERSED-BENDING FATIGUE STRENGTH OF A-110 AT TITANIUM ALLOY SHEET

**INVESTIGATOR:** Robert J. Rooney

**ABSTRACT:** The results of reversed cantilever bending fatigue tests on A-110 AT (5% AL - 2.5% Sn) titanium alloy sheet, machined by various processes, are presented. The machining processes employed were the following: Ultrasonic, slab milling, chem-milling, grinding and electrical discharge machining. The effect of shot-peening on the fatigue strength of the "as-rolled" alloy is also presented. The effects of the various machining processes, as well as that of shot-peening, are shown by comparing the fatigue strengths obtained under these conditions with the fatigue strength of the material in the "as-rolled" condition. Results of the measurements of residual surface stress in the "as-rolled" material are also presented.

WADC TR 57-589  
ASTIA Document No. AD 151162  
OTS Release

April 1958

**SUBJECT:** FATIGUE STRENGTH REDUCTION FACTORS FOR INCLUSIONS IN HIGH STRENGTH STEELS

**INVESTIGATOR:** H. N. Cummings  
F. B. Stulen  
W. C. Schulte

**CONTRACT:** AF 33(616)-5182

**CONTRACTOR:** Curtiss-Wright Corporation

**ABSTRACT:** Tentative values of Fatigue Strength Reduction Factors for non-metallic non-malleable inclusions in single-nucleus fractures of R. R. Moore rotating beam specimens are determined by two methods. Data for the computations are taken from tests on 309 specimens of SAE 4340 and 4350 steel, of 140, 190, 230, 260 and 300 ksi UTS. Quantitative results

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are thought to be somewhere near the correct order of magnitude. Qualitatively, it is concluded that the values of the factors depend upon the size of the inclusions and upon the hardness level of the steel. Also, it is thought that for very small inclusions (less than 0.00025 inch) other inhomogeneities inherent in the steel itself dominate the failure of a specimen.

## FUELS

WADC TR 55-138  
ASTIA Document No. AD 118109  
OTS Release

March 1957

SUBJECT: ACCELERATED STORAGE STABILITY OF AVIATION FUELS  
INVESTIGATOR: R. W. Sneed  
O. M. Ballentine  
Capt. J. H. Winterhaulter

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

Wide variations were found in the rates of deterioration of the fuels. Phenylene diamine appears to be the best oxidation inhibitor and the C<sub>2</sub> and C<sub>4</sub> alkylate base stock is more stable than the straight run fuel; copper definitely accelerates gum formation.

## GRAPHITE

WADC TR 57-491 Pt I  
ASTIA Document No. AD 142022  
OTS Release

October 1957

SUBJECT: SiC-GRAPHITE BODY FOR ELEVATED TEMPERATURE SERVICE

WADC TR 53-373 Sup 5

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# Contrails

INVESTIGATOR: Carl F. Cline  
CONTRACT: AF 33(616)-3538  
CONTRACTOR: The Carborundum Company  
ABSTRACT: A graphite body bonded by silicon carbide has been developed which has physical properties superior to graphite alone. Data are presented for transverse rupture strength, density, chemical analysis, and tensile strength. One composition has an average transverse rupture strength of 12,000 pounds per square inch at 1500°C. as compared to 5,400 pounds per square inch for ATJ graphite.

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

WADC TR 57-602  
ASTIA Document No. AD 150962

February 1958

SUBJECT: GRAPHITE-BASED MATERIALS FOR HIGH TEMPERATURE APPLICATIONS  
INVESTIGATOR: M. Jones  
CONTRACT: AF 33(616)-3537  
CONTRACTOR: National Carbon Company  
ABSTRACT: Recent aerodynamic developments require a material which will endure under conditions of high heat flux with consequent high surface temperatures and high flow of an oxidizing gas relative to a body of the material. Surface temperature rise may be quite rapid, so that the material must possess resistance to thermal shock. Graphite has outstanding high temperature strength, a high sublimation temperature, and excellent thermal shock resistance in comparison to other agglomerate bodies. However, it is quite reactive with oxygen or other oxidizing gas at high temperatures and high relative gas flow rates.

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

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

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

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

## HYDRAULIC FLUIDS

WADC TR 55-89 Pt V  
ASTIA Document No. AD 118341

July 1957

SUBJECT: PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF SILICATE  
BASE HIGH TEMPERATURE HYDRAULIC FLUIDS  
Part V. Room Temperature Hydrolysis Studies

INVESTIGATOR: George Baum

ABSTRACT: Silicate-esters are being considered for use in 400°F  
hydraulic systems. Previous study had established evidence of the  
hydrolytic instability of the silicate-ester formulations.

The behavior of four silicate-ester base formulations toward extended storage with varying quantities of water was studied. The formulations presented varying degrees of hydrolytic stability. Solids formation was encountered with all formulations. However, the disiloxane formulation (MLO 8200) produced significantly lower quantities of solids than the other three formulations. Addition of a diester to this disiloxane formulation resulted in a large increase in the amount of solids formed. The solids formed were not a linear function of water present. Instead, maximum solids occurred at intermediate water concentrations.

WADC TR 56-168 Pt II  
ASTIA Document No. AD 150988  
OTS Release

February 1958

SUBJECT: SILICONE FLUID RESEARCH FOR THE DEVELOPMENT OF HIGH  
TEMPERATURE HYDRAULIC FLUID AND ENGINE OILS

INVESTIGATOR: Edgar D. Brown, Jr.  
Norman G. Holdstock

CONTRACT: AF 33(616)-2899

CONTRACTOR: General Electric Company

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# Contrails

**ABSTRACT:** Continued study of the chlorophenyl silicone formulation has resulted in development of an additive which greatly improves the lubricity of the base fluid (F-50) without impairing other desirable features such as viscosity-temperature characteristics and oxidation stability below 500°F. This additive is a straight chain linked tin-carbon-silicon-oxygen polymer and is soluble in the base fluid at all temperatures down to -65°F. The tin polymer-silicone formulation has been designated silicone fluid 81717 and will allow for prolonged use in the -65°F to 575°F temperature range. Under appropriate conditions of high pressure and wet atmosphere, hydraulic fluid use in the 650°F - 700°F range is possible.

Silicone fluids undergo thermal rearrangement above 600°F which causes large changes in viscosity properties. However, a change in structure to silicone carbon linkage has yielded fluids with excellent 700°F thermal stability. Further syntheses and evaluation study is required to produce fluids of the desired lubricity level.

The fluid with the best over-all oxidation stability is still the base-stock, F-50, with iron octoate as oxidation inhibitor. This is now available as silicone fluid 81644.

WADC TR 56-207 Pt III  
ASTIA Document No. AD 151166

April 1958

**SUBJECT:** \*CHAIN TYPE\* POLYPHENYL AND POLYNUCLEAR AROMATIC COMPOUNDS AS BASE MATERIALS FOR HIGH TEMPERATURE STABLE AND RADIATION RESISTANT LUBRICANTS AND HYDRAULIC FLUIDS

**INVESTIGATOR:** Josef J. E. Schmidt  
John A. Krimmel  
Thomas J. Farrell, Jr.

**CONTRACT:** AF 33(616)-2939

**CONTRACTOR:** Denver Research Institute

**ABSTRACT:** This report describes the preparation and investigation of alkyl-biphenyl and alkylterphenyl derivatives and their intermediates. In particular, homologous series of 4' -alkyl-m-terphenyl and 3-alkyl-m-terphenyl compounds have been prepared in connection with a closer investigation of the correlations between chemical structure of these classes of compounds and their physical properties, thermal stability, and their feasibility as thermally stable and radiation resistant fluids.

Two half-gallon samples of the more promising fluids, 4' -n-nonyl-m-terphenyl and 3-n-heptyl-m-terphenyl, have been prepared for WADC's Materials Laboratory under contractual terms for the purpose of additional testing.

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WADC TR 57-2  
ASTIA Document No. AD 131080

September 1957

**SUBJECT:** HIGH TEMPERATURE FLUID EVALUATION TEST STAND  
**INVESTIGATOR:** Donald B. Reinke  
**CONTRACT:** AF 33(616)-3126  
**CONTRACTOR:** Sundstrand Aviation Division  
**ABSTRACT:** A hydraulic fluid test stand with the capability of evaluating fluids at temperatures up to 700°F and pressures up to 3000 psi has been constructed. A survey of the industry showed several areas in which reliable aircraft type components were not available, and in such instances the best available non-aircraft components were adapted to complete the test stand.

This report describes the fluid evaluation parameters, the finalized test procedures to be used for fluid analysis, the selection of components, and the results of initial check-out tests. Evaluation of hydraulic fluids at the temperature level for which this stand is built represents a potential fire hazard. Safety and automatic control provisions are noted.

WADC TR 57-119  
ASTIA Document No. AD 130915  
OTS Release

July 1957

**SUBJECT:** DEVELOPMENT OF A HYDROLYTICALLY STABLE HIGH TEMPERATURE HYDRAULIC FLUID  
**INVESTIGATOR:** Robert L. Peeler  
Steve A. Kovacich  
**CONTRACT:** AF 33(616)-3476  
**CONTRACTOR:** California Research Corporation  
**ABSTRACT:** This report covers the first year's work on a contract to improve the hydrolytic stability of silicate based hydraulic fluids operating in the -65°F to 400°F temperature range.

A literature search was made of the mechanism and factors affecting the rate of hydrolysis of alkoxy silanes. Steric effects were found to be the most important factor.

Silanes, disiloxanes, trisiloxanes, and miscellaneous silicates, having primary, secondary, and tertiary alkoxy, alkyl, and phenyl groups, were synthesized. These compounds were evaluated for hydrolytic stability at 400°F. Of the compounds tested to date, silanes with two tertiary butoxy and two secondary alkoxy groups have the greatest hydrolytic stability. Di(tert-butoxy)-di(5-ethyl-2-nonyloxy) silane and di(tert-butoxy)di(2-octoxy)silane were the two most stable compounds prepared.

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Disiloxanes containing secondary alkoxy and two tertiary butoxy groups have not shown stability comparable to the silanes.

Two carboxylic acid esters and four silicones were evaluated for hydrolytic stability at 400°F for reference purposes. Contrary to the good hydrolytic stability shown by these synthetic materials at 200°F, they showed considerable deterioration at 400°F. Thus, the best silanes prepared in this program are more stable in this test than other types of synthetic base materials usually assumed to have satisfactory stability.

A small-scale, 400°F hydrolytic stability test method was developed which shows good correlation with the standard bomb method. Diesters, mono-esters, and hydrocarbons have been found which are more effective swelling agents for Neoprene than di(2-ethylhexyl) sebacate. Dimethyl silicones were found to be the most effective viscosity index improvers of those tested, although other high viscosity silicones, polymethacrylates, and polyvinyl ethers were also usable. With di(tert-butoxy)di(2-octoxy)silane, a fluid with 2500 cs viscosity at -65°F and over 1.0 cs at 400°F can probably be prepared. Oxidation tests at 400°F indicated the need for better inhibitors. Four-Ball Wear properties of the new hydrolytically stable base stocks were equivalent to the currently used hexa(2-ethylbutoxy)disiloxane.

WADC TR 57-151  
ASTIA Document No. AD 130764  
OTS Release

May 1957

**SUBJECT:** RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT HYDRAULIC FLUIDS

**INVESTIGATOR:** Michael G. Zabetakis  
Aldo L. Furno  
Joseph J. Miller, Jr.

**CONTRACT:** AF 18(600)-151

**CONTRACTOR:** Bureau of Mines

**ABSTRACT:** The results of minimum spontaneous ignition temperature tests conducted on seven hydraulic fluids while in contact with seven surfaces found in aircraft under conditions likely to be encountered in practice are presented here. These tests were conducted by members of the Branch of Gas Explosions, Division of Explosives Technology, U. S. Bureau of Mines between 1 November 1955 and 31 October 1956. Hydraulic fluids Esso Univis J-43, conforming to specification MIL-O-5606, MLO 53-446 (General Electric GE 81406), MLO 54-540 (Monsanto OS 45), MLO 54-540 (Monsanto OS 45), MLO 54-581, MLO 54-645 (85% Oronite 8200 + 15% Flexol), MLO 54-856 (Hollingshead 72073C), and MLO 8200 (Oronite 8200) were tested while in contact with heated aluminum, beryllium-copper, copper, magnesium, pyrex glass, stainless steel and titanium surfaces. The effects of both test chamber pressure and injection pressure variations were investigated.

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WADC TR 57-151 Sup 1  
ASTIA Document No. 155510  
OTS Release

May 1958

**SUBJECT:** RESEARCH ON THE FLAMMABILITY CHARACTERISTICS OF AIRCRAFT  
HYDRAULIC FLUIDS

**INVESTIGATOR:** Michael G. Zabetakis  
Agnes C. Imhof  
Frank W. Lang

**CONTRACT:** DO 33(616) 0-57-4

**CONTRACTOR:** Bureau of Mines

**ABSTRACT:** The results of spontaneous ignition temperature tests conducted on JP-4 and seven hydraulic fluids in air while in contact with seven surfaces found in aircraft are presented. Tests were conducted at reduced and elevated pressures using both low and high velocity injection techniques. They were conducted by members of the Branch of Gas Explosions, Division of Explosives Technology, U. S. Bureau of Mines between 1 November 1956 and 31 October 1957. Hydraulic fluids Esso Univis J-43 (Spec. MIL-O-5606), MLO 53-446 (General Electric GE 81406), MLO 54-540 (Monsanto OS 45), MLO 54-581, MLO 54-645 (85% Oronite 8200 + 15% Plexol), MLO 54-856 (Hollingshead 72073C) and MLO 8200 (Oronite 8200) were tested while in contact with heated aluminum, beryllium-copper, copper, magnesium, pyrex glass, stainless steel and titanium surfaces. In addition, the flash and fire points of these fluids were determined in a standard ASTM apparatus.

WADC TR 57-167  
ASTIA Document No. AD 131009  
OTS Release

August 1957

**SUBJECT:** HIGH TEMPERATURE HYDRAULIC FLUID DEVELOPMENT STATUS  
AND ENGINEERING DATA

**INVESTIGATOR:** George Baum  
Robert J. Benzing

**ABSTRACT:** A review of the status of extreme high temperature hydraulic fluid development is presented. Progress in research and development for fluids with a high temperature capability of 550°F to 700°F and a low temperature capability of 0°F to -65°F is described in detail. The fluids within this temperature range and described in this report fall into the following classes of compounds; Organosilicates, disiloxanes, silicones, mineral oils and silanes. The available data on properties of these research fluids, important to hydraulic system design are reported. These data include the following; viscosity-temperature, density-temperature, thermal stability, data for heat transfer calculations, compressibility, vapor pressure, and flammability measurements.

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The large amount of design data on  $-65^{\circ}\text{F}$  to  $400^{\circ}\text{F}$  type fluids obtained by industry and the military services is compiled and reported. This includes both previously published and unpublished data.

WADC TR 57-168  
ASTIA Document No. AD 142141  
OTS Release

March 1958

**SUBJECT:** LABORATORY EVALUATION OF SILANE FLUIDS AS POTENTIAL BASE STOCKS FOR HYDRAULIC FLUIDS AND LUBRICANTS

**INVESTIGATOR:** Harold W. Adams  
George Baum

**ABSTRACT:** A new class of potential base stocks for high temperature turbojet engine oils and hydraulic fluids has been developed at this laboratory. The physical and chemical properties of the alkyl and alkyl-aryl silanes show them to possess good thermal stability up to  $650^{\circ}\text{F}$ , good lubricity and marginal oxidative stability at  $500^{\circ}\text{F}$ .

An overall assessment of the properties of the silanes evaluated indicates that they are promising base materials for a 0 to  $500^{\circ}\text{F}$  engine oil or a 0 to  $700^{\circ}\text{F}$  hydraulic fluid.

WADC TR 57-266 Pt I  
ASTIA Document No. AD 130807  
OTS Release

June 1957

**SUBJECT:** EFFECTS OF NUCLEAR RADIATION ON ORGANIC FLUIDS  
Part I. Gamma Radiation Stability of Certain Mineral Oils and Diester Fluids

**INVESTIGATOR:** William L. R. Rice, 1/Lt  
James H. Way, 1/Lt

**ABSTRACT:** A study was made of the stability to gamma radiation of certain refined petroleum oils and diester fluids. Testing of the irradiated fluids by means of lubricant evaluation procedures, such as viscosity, flash point, and oxidation-corrosion resistance, indicated that for the tests conducted, the mineral oils of the type studied should have general resistance to gamma radiation up to a dosage of about  $1 \times 10^8$  roentgens. The diester fluid di-2-ethylhexyl sebacate (Plexol 201) had very poor stability to gamma radiation over the same range. Addition of 0.5% phenothiazine improved the radiation resistance of this base fluid, except for the loss of oxidative stability experienced at dosage levels of  $1 \times 10^7$  roentgens and lower.

Examination was made of the test data for the irradiated fluids to determine if any property changes followed the radiation exposure history. This was to discover if either the mineral oil or diester type

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fluids could be used as secondary reference standards for radiation calibration of non-uniform geometries exposed to nuclear radiation, such as bearing assemblies or circulating fluid loops. The changes offering the most promise were the increase in neutralization number of the diester fluid and the gas evolution of the mineral oils.

WADC TR 57-294  
ASTIA Document No. AD 151001  
OTS Release

February 1958

SUBJECT: RESEARCH ON LIQUID METALS AS POWER TRANSMISSION FLUIDS  
INVESTIGATOR: Richard H. Blackmer  
CONTRACT: AF 33(616)-3698  
CONTRACTOR: General Electric Company  
ABSTRACT: The eutectic alloy of sodium (23 weight-percent) and potassium (77 weight-percent), known as NaK 77, has been determined from a technical survey of liquid metals and salts as the most feasible liquid known for 10°F to 1000°F hydraulic system applications. A single cylinder test pump in an inert atmosphere glove-box has pumped NaK-77 up to 3600 psi at 100°F and up to 2000 psi at 1000°F. A total of about 100,000 cycles at 1 cycle per second and an average pressure of 1500 psi have been accumulated. Results of literature survey, consultation, and laboratory tests are included in this report.

WADC TR 57-437  
ASTIA Document No. AD 142188

December 1957

SUBJECT: HIGH-TEMPERATURE BASE STOCK FLUIDS  
INVESTIGATOR: Edward S. Blake William C. Hammann  
James W. Edwards Thomas E. Reichard  
CONTRACT: AF 33(616)-3819  
CONTRACTOR: Monsanto Chemical Company  
ABSTRACT: A limited study was made on the variables involved in the production of a quantity of bromine-free non-crystallizable mixed bis-(methylphenoxy)benzene, by the Ullmann reaction.

Thermal stability and other physical data are reported on 61 compounds which include seven classes not previously reported, namely amides, sulfones, aromatic amines, ketones, silicon fluorides, triaryl derivatives of Group V-B elements below nitrogen and ferrocenes. Twenty chemical classes are compared in thermal stability, and the useful life for eight potential base stocks is estimated.

The limits of error for the WADC TR 55-449 oxidation and corrosion test were determined using esters without antioxidants as

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test fluids. This test was used to determine the oxidation susceptibility of certain compounds and substituent groups in aromatic ethers. An attempt was made with some success to adapt a modified Warburg apparatus for simplified oxidation studies.

Some eutectic and crystallization studies on various aromatic ethers are reported. Numerous syntheses were made.

WADC TR 57-573  
ASTIA Document No. AD 142262  
OTS Release

December 1957

**SUBJECT:** GAMMA RADIATION STABILITY OF OS-45 AND OS-45-1  
HYDRAULIC FLUIDS

**INVESTIGATOR:** William L. R. Rice, 1/Lt

**ABSTRACT:** The hydraulic fluids OS-45 and OS-45-1 were exposed to gamma radiation over the range  $4.36 \times 10^8$  ergs/gm C to  $8.71 \times 10^{10}$  ergs/gm C. Changes in the physical and chemical characteristics of the fluids were determined following the static irradiations. It was observed that major changes took place in most of the properties at the higher exposure levels. Relationships were derived between the change in certain properties and the gamma dosage. It was concluded that although the effects of gamma radiation on the fluids indicated possible difficulty in their use in a radiation field, dynamic in-source testing in a mock-up hydraulic system would be necessary to establish their use limitations.

WADC TR 57-581  
ASTIA Document No. AD 151074

March 1958

**SUBJECT:** RESEARCH AND DEVELOPMENT ON THE SYNTHESIS OF TETRAALKYL SILANES

**INVESTIGATOR:** Edward E. Harris                      Robert J. Rayner  
Frank J. Milnes                                      Roman Dombachewsky

**CONTRACT:** AF 33(616)-3430

**CONTRACTOR:** Olin Mathieson Chemical Corporation

**ABSTRACT:** One-gallon samples of nine tetraalkyl silanes and one diaryl dialkyl silane have been synthesized for testing as high-temperature lubricants. The diaryl dialkyl silane and three of the tetraalkyl silanes have been produced in five-gallon quantity. Finally, fifteen-gallon quantities of diphenyl di-n-octyl silane and di-n-dodecyl di-n-octyl silane have been produced. A Grignard process has been used for all syntheses, both of intermediates and final products.

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WADC TR 58-84  
ASTIA Document No. AD 151191

May 1958

**SUBJECT:** DEVELOPMENT OF HIGH-TEMPERATURE STABLE LIQUID UREAS  
AND AMIDES  
**INVESTIGATOR:** Cecil C. Chappelow, Jr.  
Robert N. Clark  
**CONTRACT** AF 33(616)-5129  
**CONTRACTOR:** Midwest Research Institute  
**ABSTRACT:** A program for the development of thermally stable liquid ureas was undertaken. This program consisted of the syntheses and evaluation of new compounds which contain only carbonly groups, nitrogen atoms and organic radicals. The first class of compounds to be investigated has been the tetra-substituted ureas.

During the syntheses program, a total of 26 tetra-substituted ureas were prepared, 23 of which are new compositions of matter not reported in the literature. These compounds are representative of the following types of tetra-substituted ureas: tetraalkylureas, dialkyl-diarylureas, tetraalkarylureas, dialkaryldiarylureas, N-(substituted phenyl)triphenylureas and tetraarylureas.

During the evaluation program, primary emphasis was placed upon thermal stability. As a result of this work, it was found that the basic urea structure was very stable. Also, it was found that the following substituent groups were thermally stable: diphenylene, phenyl, 4-methylphenyl, 4-biphenyl, 4-methoxyphenyl, 2,4-dimethylphenyl, 1- and 2-naphthyl, n-octadecyl, methyl, 4-phenoxyphenyl, n-butyl.

On the basis of preliminary experimental data, tetra-substituted ureas as a class of compounds show promise as base materials for high-temperature hydraulic fluid and lubricant applications.

## LUBRICANTS

WADC TR 53-83 Pt VI  
ASTIA Document No. AD 151053

March 1958

**SUBJECT:** DEVELOPMENT AND EVALUATION OF HIGH TEMPERATURE GREASE  
THICKENERS  
**INVESTIGATOR:** John C. Goossens  
**CONTRACT:** AF33(038)-23687

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**CONTRACTOR:** Standard Oil Company (Indiana)  
**ABSTRACT:** This work was directed toward the development of new grease thickeners operable at temperatures above 450°F and as high as 700°F. During this period a variety of substances were prepared and tested as thickeners for silicone and ester fluids. Those which proved to be effective thickeners in less than 40% concentration were given preliminary screening tests and the better samples were run in the ABEC-NLGI bearing tester.

The general types of materials tested as high temperature thickeners were polar organic compounds, polymers, and organo-metal compounds. Selection of materials for test was based on thermal stability, ease of preparation, and ability to prepare the substance in small particle size. Of the many materials tested, those which have shown the greatest promise are, in decreasing order of their effectiveness, a new substituted pteridine, derivatives of hydrazine with bitolylene diisocyanate, phthaloyl melamine and some derivatives of cyanuric acid. However, at present none of these appears as good a high temperature thickener as the arylureas developed previously.

In addition to the usual 450°F high-speed bearing tests, 600°F bearing tests were conducted on silicone greases thickened with the two better thickeners, arylurea and a pteridine derivative. The tests were run both in air and under a slow stream of nitrogen. Both greases ran 85 to 120 hours under nitrogen. In air the arylurea had better oxidative stability running 40 to 60 hours in contrast with approximately 20 hours for the pteridine thickened grease.

WADC TR 53-293 Pt VIII  
ASTIA Document No. AD 150994  
OTS Release

February 1958

**SUBJECT:** HIGH TEMPERATURE ANTIOXIDANTS FOR SYNTHETIC BASE OILS  
Part VIII. Evaluation of Antioxidants in Synthetic Fluids  
**INVESTIGATOR:** James W. Cole, Jr.  
**CONTRACT:** AF 33(616)-3234  
**CONTRACTOR:** University of Virginia  
**ABSTRACT:** This is a continuation of a laboratory program on the evaluation of the oxidation patterns of synthetic fluids in the presence and absence of inhibitors and metals in the temperature range 400°-700°F. Two methyl chlorophenyl silicones, F-50 and F-60 were examined at 500°F and 600°F. N,N'-di-2-naphthyl-p-phenylenediamine in 0.1 - 0.2% was the most interesting additive. Work with a mineral oil, MLO 57-30, did not reveal additives of outstanding activity. Substances containing sulfur and selenium showed promise, but some attack on silver and copper.

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The experiences with four tetra-substituted silanes showed that these substances did not have outstanding response to additives. A series of runs with a pentaerythritol ester, MLO 55-584, indicate that ring substituted aryl amines have considerable antioxidant activity over the range 400°-500°F. Some additional data for bis-(2-ethylhexyl) sebacate are included to compare the promising amines with the phenothiazine type. The former retain the inhibition of activity over a wider temperature range. Some attention was given to determining the nature of the components in an oxidized diester which contributes to the acidity. It appears that some improvement may be achieved in a partial oxidized fluid by washing with hydrocarbon solvents. The limitations of a laboratory test procedure are discussed, especially with respect to the evaluation of the effects of the test metals, aluminum, silver, copper, titanium and several steels.

WADC TR 53-426 Pt IV  
ASTIA Document No. AD 118307  
OTS Release

May 1957

SUBJECT:           ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE  
                  LUBRICANTS AND RELATED MATERIALS

INVESTIGATOR:    Henry Gilman  
                  Richard D. Gorsich

CONTRACT:        AF 33(616)-3510

CONTRACTOR:     Iowa State College

ABSTRACT:        This project is concerned primarily with the syntheses of new compounds to be examined as potential hydraulic fluids and lubricants. The syntheses and preliminary screening of organo-metallic and organo-metalloidal compounds has been extended. New cyclic compounds continue to be promising. It has also been found that the meta-biphenyl group can be added to those groups which impart desirable physical characteristics. The methyl-di-benzylsilylmethyl group is also of significant promise.

In the experimental part, there is included a table which outlines the preliminary screening of thirty-six compounds.

The security classification of this report is unclassified.

WADC TR 53-426 Pt V  
ASTIA Document No. AD 142211

December 1957

SUBJECT:           ORGANO-METALLIC AND ORGANO-METALLOIDAL HIGH-TEMPERATURE  
                  LUBRICANTS AND RELATED MATERIALS

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# Contrails

INVESTIGATOR: Henry Gilman  
Richard D. Gorsich  
Bernard J. Gaj  
CONTRACT: AF 33(616)-3510  
CONTRACTOR: Iowa State College  
ABSTRACT: Several novel synthetic procedures have been developed in connection with studies concerned with the preparation of organometallic and organometalloidal compounds to be examined as potential hydraulic fluids and lubricants. The promising cyclic compounds have been extended by making available cyclic-Si-M types. Procedures have been developed for the synthesis of non-cyclic unsymmetrical  $R_3SiM$  compounds. One of the new developments involving tetrahydrofuran as a solvent in organosilicon chemistry is the stepwise introduction of a wide variety of R groups by reactions between the Si-H group and some RM compounds. This provides the opportunity of synthesizing  $R_4Si$  compounds, where all R groups may be unlike, and where there is a maximum promise of obtaining liquids.

The experimental part contains a table which describes the preliminary screening of twenty-eight compounds.

The security classification of this report is unclassified.

WADC TR 54-464 Pt IV  
ASTIA Document No. AD 130922

July 1957

SUBJECT: DEVELOPMENT OF SCHEMATIC ANALYTICAL PROCEDURES FOR SYNTHETIC LUBRICANTS AND THEIR ADLITIVES  
Part IV. Laboratory Manual for the Analysis of Synthetic Lubricants, Greases and Their Additives  
INVESTIGATOR: Francis S. Bonomo  
Joseph J. E. Schmidt  
CONTRACT: AF 33(616)-3336  
CONTRACTOR: Denver Research Institute  
ABSTRACT: Laboratory methods and techniques for the analysis of certain synthetic lubricants, greases, and their additives are described in detail, including drawings and photographs of equipment when necessary. As stipulated by WADC, none of the methods includes optical or elaborate instrumental techniques of analysis. Most of the methods employ simple wet chemical manipulations, or such techniques as adsorption or partition paper or column chromatography. The individual methods are numbered and patterned after Federal Test Method Standard No. 791.

The manual is divided into five major sections:

- (1) Preliminary qualitative examination of the lubricant

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# Contrails

or grease, including solubility, elemental analysis, identification of grease type, and behavior of the lubricant (or base-oil from a grease) upon adsorption on a column of silica gel.

- (2) Qualitative detection and quantitative determination of antioxidants.
- (3) Qualitative detection and quantitative determination of corrosion preventive compounds.
- (4) Separation and quantitative determination of gelling agents and thickeners.
- (5) Separation, identification, and determination of base-oils from lubricants and greases.

In a number of instances parts of existing ASTM methods have been incorporated into the analytical procedures, where applicable, to avoid the use of parallel methods to the same goal.

WADC TR 54-576 Pt II  
ASTIA Document No. AD 150977  
OTS Release

February 1958

SUBJECT: EFFECT OF METALS ON LUBRICANTS  
Part II. Corrosion and Oxidation Stability at 400°  
Fahrenheit

INVESTIGATOR: John B. Christian

ABSTRACT: This report presents data which deals with the general effects which silicates and siloxanes have on various metals; and the effects which these metals have on the fluids under severe conditions.

The corrosion and oxidation stability of a diester blend, a siloxane, and a silicate in the presence of various metal specimens is discussed.

WADC TR 55-30 Pt V  
ASTIA Document No. AD 130907  
OTS Release

July 1957

SUBJECT: FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS

INVESTIGATOR: E. Erwin Klaus  
Merrell R. Fenske

CONTRACT: AF 33(616)2851

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**CONTRACTOR:** Pennsylvania State University  
**ABSTRACT:** This report describes work carried out on a continuing project directed toward the development of improved hydraulic fluids and jet engine lubricants for use in the high temperature range of 400° to 700°F. With few exceptions these studies are equally applicable to hydraulic fluids and jet engine lubricants.

Efforts have been concentrated on the study of mineral oils, hydrocarbons, and improved stability esters for application to high temperature systems. Super-refining of mineral oils and hydrocarbons by hydrogenation, acid extraction, and silica gel percolation has been shown to improve materially the overall quality of these materials as hydraulic fluids and lubricants. Selection of the molecular configuration of esters has been shown to affect their thermal stability and hence their overall utility as high temperature fluids.

A number of new and improved test techniques have been developed or applied to the high temperature evaluation of hydraulic fluids and jet engine lubricants. These tests include: (1) high temperature density measurement, (2) the controlled atmosphere panel coker test, (3) a modified thermal stability test including catalyst metals, (4) a high temperature PRL thin film oxidation test, and (5) a single pass high temperature lube rig.

A number of large scale blends designed for use in bench, mock-up, and actual service equipment (jet engines and high temperature hydraulic systems) have been formulated for evaluation by WADC and industry. Used samples of fluids from actual tests in jet engines and high temperature hydraulic systems have been evaluated. Results of these evaluations offer some insight into the actual behavior of fluids in service and the degree of realism in various laboratory tests in policing fluid quality necessary for adequate service behavior.

Some additional miscellaneous duties have been performed at the request of the Wright Air Development Center. These duties include: (1) distribution of Laboratory reports, (2) fluid formulation, (3) distribution of standard fluid samples, (4) preparation of WADC Technical Reports and papers on specific topics of interest, and (5) evaluation of specific fluid properties.

WADC TR 55-240 Pt III  
ASTIA Document No. AD 130808  
OTS Release

June 1957

**SUBJECT:** BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP LUBRICATING GREASES

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INVESTIGATOR: John J. Chessick  
Albert C. Zettlemyer  
CONTRACT: AF 33(616)-2440  
CONTRACTOR: Lehigh University  
ABSTRACT: This project is aimed at the determination of the fundamental factors in the formation of non-soap greases, and the relation of these factors to their thermal, chemical and mechanical stability. The vehicle-thickener interface was early recognized as the seat of important properties in non-soap greases. In order to index the nature and extent of this interface, first the physical properties of several oils and the surface characteristics of a variety of thickening agents were measured. These properties of the oils and solids were correlated with the behavior of grease systems formulated with them. Modification of the interfacial region by water and its effect on grease building by inducing flocculation was then investigated.

The influence of concentration and surface area of hydrophobic thickeners on the consistencies of various greases was determined specifically. Above  $200 \text{ m}^2/\text{g.}$ , grease building varies directly with surface area, but falls off sharply below this level. To give greases of a specified consistency, the amounts of thickeners required varies as the square of their surface areas. It appears also that the effect of area is similar for all thickeners.

Grease consistency was found to be directly proportional to the amount of water adsorbed at the oil-thickener interface for hydrophilic thickeners. As might be expected, the hydrophilic thickeners adsorb relatively large amounts of water and yield much larger changes than greases built with hydrophobic thickeners. The amount of water in a grease varies considerably with temperature, amount of working and the relative humidity.

While water enhances the gel forming ability of hydrophilic solids in nonpolar vehicles, somewhat surprisingly, the opposite effect is found for these solids dispersed in polar liquids miscible with water. The thickening effect of hydrophobic thickeners is not nearly so sensitive to the polarity of the vehicle, since very little water is adsorbed to develop flocculation.

These and related data were used to develop a mechanism for the flocculation of hydrophobic and hydrophilic solids in grease vehicles to form non-soap gels. Of great importance was the finding that in gelling pure liquids, the particle size of the thickener is more important than the polarity of the surface. However, in the presence of trace amounts of water, commonly present under practical conditions, grease consistency is strongly dependent on the polar nature of the solid surface. For thickeners dispersed in oils containing both water and

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additives the polarity of the surface determines whether the water or additive will be preferentially adsorbed.

Further studies of the influence of heptyl additives, their benzene analogues, as well as more complex additives were carried out. In addition, rheological measurements of a number of thickener solids in several grease vehicles were made. The preparation of arylurea thickeners was also recently begun. These studies have not yet proceeded to a point where a quantitative discussion can be offered.

Low angle x-ray scattering techniques have been used to study grease structure. Marked diffraction maxima have been found corresponding to spacings of the order of 100 to 2000 Å units for copper phthalocyanine thickened grease.

WADC TR 55-240 Pt IV  
ASTIA Document No. AD 151120

April 1958

**SUBJECT:** BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP LUBRICATING GREASES

**INVESTIGATOR:** John J. Chessick  
Albert C. Zettlemyer  
James P. Wightman

**CONTRACT:** AF 33(616)-3999

**CONTRACTOR:** Lehigh University

**ABSTRACT:** Rheological measurements of dispersions of grease thickeners in pure vehicles or in vehicles containing water, additives or both these components have been carried out. Previous studies were made with the systems Aerosil--Plexol 201 and Santocel C2-paraffin oil. These types of measurements have been extended to include both polar and nonpolar solids dispersed in grease vehicles. The solids studied now include Copper Phthalocyanine, G. S. Hydrophobic Silica, HiSil, Aerosil, Santocel and Permagel. It was demonstrated that surface hydrophilicity and the presence of trace water far overshadows the influence of particle diameter and shape in the formation of gel structure by solid thickeners.

Further studies of the behavior of commercial additives have been made by measuring the change in consistency of a grease with increased concentration of additive. Because of the very complex nature of additive behavior a fundamental program has been developed to learn more of this phenomenon. This study has begun and includes measurements of additive adsorption from solution by selected solids as well as heat of immersional wetting measurements of solids in additive solutions at various concentrations. Studies thus far indicate that the heat of solution of additive in the grease vehicle and the heat of wetting of the solid thickener by pure additive relative to the vehicle appear to govern adsorption from solution. With Aerosil, a weakly polar solid, the

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heats of solution of the additives studied appears to predominate, and only those additives which dislike the vehicle (high endothermic heats of solution) adsorb and fluidize Aerosil-built greases. With rutile-built greases, however, the surface polarity of the thickener and consequent heat of adsorption is sufficiently high to influence adsorption of additives ineffective in Aerosil-built greases.

The program for the preparation of arylurea thickened greases has progressed satisfactorily. Several variables have been found to influence the particle diameter of the thickener solid formed. These include type of reactant, method of addition of reactants and the temperature of the reaction. Trace or larger quantities of water drastically increase particle size of arylurea thickeners prepared in solvents from which they can be isolated. The effect on greases prepared "in situ" is believed to be similar. This point is being checked.

WADC TR 55-449 Pt VI  
ASTIA Document No. AD 142210  
OTS Release

December 1957

SUBJECT: MICRO LUBRICANT TEST METHODS  
Part VI. Cloud Point, Pour Point, and Cloud Intensity  
INVESTIGATOR: John B. Christian  
ABSTRACT: Miniaturized methods have been developed for the determination of cloud point, pour point, and cloud intensity. The methods described herein require a 5-milliliter sample for the three determinations.

WADC TR 56-646 Pt II  
ASTIA Document No. AD 151176

April 1958

SUBJECT: EFFECTS OF RADIATION ON AIRCRAFT LUBRICANTS AND FUELS  
INVESTIGATOR: R. O. Bolt  
J. G. Carroll  
CONTRACT: AF 33(616)-3184  
CONTRACTOR: California Research Corporation  
ABSTRACT: The objectives of the work reported included the development of radiation resistant lubricants and a survey of the radiation stability of jet fuels.

Aromatic base materials were needed in the lubricant work, and most of these had to be synthesized. Exploratory synthesis evolved alkaryl ethers, alkaryl esters, alkylbenzenes, alkyl diphenyl ethers, and diarylalkanes. Chemical structure was correlated with physical properties and also with oxidation, thermal, and radiation stability. Although esters were inferior, each class had members with good radiation stability. Over-all, the alkyl diphenyl ethers offer the most promise for

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lubricant development. Polymers were also synthesized. These were used in an alkyl diphenyl ether to improve viscosity index and to increase viscosity. Compared to conventional thickeners, alkylated poly( $\alpha$ -methylstyrenes)(APAMS) and poly(alkylphenoxyethyl methacrylates) (APEMS) show promise for use in future radiation resistant lubricants.

The grease research produced CALRESEARCH 159, a material of enhanced radiation stability. It is a selenide-inhibited alkylbiphenyl gelled with an aromatic salt. Promise of improved products was shown in work on new oils, e. g., tris(phenoxyphenyl)dodecylsilane; on new gelling agents, e.g., sodium N-p-tolyterephthalamate of mixtures of aromatic salts; and on new additives, e.g., N, N'-di-2-naphthyl-p-phenylenediamine.

Many formulations of various hydraulic fluids were evaluated before and after irradiation. The alkyl diphenyl ethers looked best in radiation stability and in thermal stability at 700°F. CALRESEARCH 216 evolved from the formulation work. It is an alkyl diphenyl ether inhibited with a selenide and thickened with a polybutene. Chemical inhibitors improved oxidation stability both before and after gamma irradiation. Aromatic hydrocarbon additives in MLO 8200 fluid reduced viscosity change and gassing caused by irradiation. This fluid was shown to be usable to about  $10^{10}$  ergs/g C of gamma radiation. Original and irradiated aromatic base fluids operated satisfactorily in aircraft piston pumps at 275°F. Isothermal bulk modulus was found to decrease markedly in a gas-liquid system such as would prevail under irradiation.

The alkyl aromatics were found best as base materials for gas turbine oils. Esters, mineral oils, and polyglycols tested were deficient in radiation or thermal stability. CALRESEARCH 230 was developed in the work; it is a selenide-inhibited alkyl diphenyl ether containing a petroleum bright stock and other additives. The alkyl diphenyl ethers showed good oxidation stability before and after irradiation. Their tendency towards high coking and low lubricity was improved by additives. Irradiation lowered coking in these bases and also in alkylbiphenyls. In all base stocks, foaming increased after irradiation, with or without silicone being present. Coking was reduced by blending with high boiling materials, e.g., bright stocks. Certain oil soluble dyes showed synergism with the beneficial dialkyl selenides in oxidation tests.

The thermal stability of nine jet fuels (five JP-4's, three JP-5's, and an RP-1) was studied in the CFR coker. Original stocks and samples irradiated for three different levels were tested. Low level irradiation ( $0.8 \times 10^{10}$  ergs/g C) impaired thermal stability. Higher level radiation ( $8 \times 10^{10}$  ergs/gC) improved thermal stability. An equation was developed by which it was possible to predict increase in viscosity with irradiation at any dosage once a single dosage point was known.

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WADC TR 57-61 Pt I  
ASTIA Document No. AD 142187  
OTS Release

December 1957

SUBJECT: LUBRICATION OF TITANIUM  
INVESTIGATOR: Richard O. Lee  
Nicholas Fatica  
CONTRACT: AF-33(616)-3350  
CONTRACTOR: Clevite Research Center  
ABSTRACT: The susceptibility of titanium to galling and seizure prevent it from replacing many steel parts involving sliding contacts in spite of the favorable strength-weight ratio of titanium. Particularly in aircraft, where one pound of steel replaced may result in gains of up to five pounds in pay load, the lubrication of moving parts involving titanium is an urgent problem. Past efforts to solve this problem have uncovered special lubricants and various surface treatments that have shown promise.

This investigation sought to explain the role of the oxide film on titanium, to obtain some information about the frictional properties of modified titanium coatings, and to make a direct comparison of the wear resistance of the best surface treatment in the presence of various lubricants using equipment generally acceptable for wear tests under conditions of boundary lubrication.

Studies on the stick-slip machine have shown that the oxide layer on titanium is easily penetrated even at moderate loads while the friction coefficient remains practically constant through this transition.

Other results on this machine show that moisture normally present in air is an important factor in determining the frictional properties of titanium. Although its effects depend on both the treatment of the metal and the lubricant used, generally speaking the moisture acts as a lubricant.

Temperature was found to be an important factor in the frictional properties evaluated on the stick-slip machine but its effect cannot be predicted even qualitatively without taking into account the surface treatment, the lubricant and the test condition.

Tests on the stick-slip apparatus also showed Halocarbon 11-14 to be generally effective as a lubricant on both untreated and treated titanium surfaces.

Studies on the Shell Four-Ball Wear Tester also showed Halo-carbon 11-14 to be the most generally effective lubricant on both

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treated and untreated surfaces. There were no significant differences among the wear properties of untreated metals (Ti 75A, Ti 6Al 4V, Ti C-130 AM) using Halocarbon 11-14. Nitriding improved the wear properties and generally lowered the friction coefficient in the presence of practically all of the lubricants tested.

Carburizing improved the wear and frictional properties of Ti 75A in most of the lubricants tested. Carburizing Ti 6Al 4V lowers the friction coefficient but seems to be without effect on the wear resistance.

Polypropylene glycol 1025 and Halocarbon 11-14 have practically identical frictional and wear properties for any of the nitrided metals. The same may be said for carburized Ti 75A. However Halocarbon 11-14 is definitely superior to polypropylene glycol 1025 for carburized Ti 6Al 4V.

In general, it may be said that all the other lubricants used were inferior to Halocarbon 11-14 and polypropylene glycol 1025 in performance with any metal surface. Where it was possible to compare the performance of the individual members of the inferior group (Nujol + 1% fatty acid addition, Carbowax, di-iso-octyl sebacate, Versilube F-50, Silicone (Dow F-60), Dow #41 grease, and silicate ester (MLO 8200), the results showed that they were practically equivalent.

Bronze sliding against titanium is an effective answer to the frictional and wear properties of Ti 75A for any lubricant tested. However, the use of this material partially defeats the objectives for using titanium in the first place.

The best wear values listed for any titanium specimen in the best lubricant are still about twice as large as those found for steel.

WADC TR 57-93  
ASTIA Document No. AD 130815

June 1957

SUBJECT: PROCEEDINGS OF AIR FORCE-NAVY-INDUSTRY CONFERENCE ON AIRCRAFT LUBRICANTS  
INVESTIGATOR: Mrs. Opal M. Fielder  
CONTRACT: AF 33(616)-3820  
CONTRACTOR: Southwest Research Institute  
ABSTRACT: The Annual Air Force-Navy-Industry Conference on Aircraft Lubricants was held at the Hilton Hotel, San Antonio, Texas, on November 5, 6, and 7, 1956. Approximately 300 representatives from the Department of the Air Force, The Department of the Navy, other government agencies, and the industry, attended. A total of 36 papers were read: 20 on gas turbine lubricants, 7 on greases, 5 on hydraulic fluids, and 4 on solid film lubricants.

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WADC TR 57-166  
ASTIA Document No. AD 130953  
OTS Release

July 1957

SUBJECT: HIGH TEMPERATURE WEAR EVALUATION TECHNIQUES AND DATA  
INVESTIGATOR: Robert J. Benzing  
ABSTRACT: A general description is given of the modifications required to increase the operating temperature potential of the following four lubricant testers: Shell Four-Ball Wear Tester used for evaluating the lubricity of fluids; Shell Roll Tester used to examine the effects of mechanical shearing on grease consistency; bearing endurance grease tester for evaluation of the high speed and high temperature performance of greases; and hydraulic fluid pump stand for determination of dynamic effects of circulation on shear stability of fluids. The British IAE gear machine, recently acquired for evaluation of the anti-scuffing properties of lubricants, is described.

Lubricity data obtained with the four-ball tester are reported for Specification MIL-O-5606 hydraulic fluid and MIL-L-7808 turbine engine oil. The wear scar data for a wide variety of experimental fluids are compared to the wear patterns for the two specification materials.

WADC TR 57-177  
ASTIA Document No. AD 130925

July 1957

SUBJECT: ENGINE OIL DEVELOPMENT  
INVESTIGATOR: C. Lynn Mahoney Emmett R. Barnum  
William W. Kerlin Karl J. Sax  
CONTRACT: AF 33(616)-3182  
CONTRACTOR: Shell Development Company  
ABSTRACT: Inhibited lubricants, made from ester or aliphatic-hydrocarbon base oils, are highly sensitive to radiation damage even in absence of oxygen. The effect on the base oil itself, measured by such properties as viscosity change or formation of acidic products, is generally relatively small at radiation doses less than  $10^8$  reps. The antioxidant, however, acts as a radical scavenger. While protecting the base oil against radiation-induced change, it is selectively inactivated. Loss of the antioxidant activity leaves the lubricant unprotected against attack by oxygen, which leads to formation of corrosive materials and deposits. Degradation products formed during irradiation of ester oils also greatly reduce the oxidation stability of inhibited lubricants. For example, a di-2-ethylhexyl sebacate blend, containing 0.5% phenothiazine as an inhibitor, had all antioxidant activity destroyed during irradiation to  $5 \times 10^7$  reps ( $\gamma$ -rays, ambient temperature, nitrogen atmosphere). With a MIL-L-7808C diester lubricant, containing 1% phenothiazine and 5% tricresyl phosphate, formation of strong phosphoric acid derivatives accelerated destruction of the inhibitor. Complete loss of antioxidant activity occurred

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in this lubricant after irradiation to  $10^7$  reps. The strong phosphoric acid derivatives increased decomposition of the ester, leading to very high deposit levels in panel coke tests. The high panel coke deposits found with other irradiated ester lubricants were also caused by the presence of tricresyl phosphate.

Auto-oxidation proceeds through free radical chain reactions. Therefore, oxidation reactions would be accelerated during irradiation. The stability of inhibited oils, in oxidation-corrosion tests conducted in a  $\gamma$ -ray source, decreased rapidly as the radiation dose rate was increased. At  $400^\circ\text{F}$ , a dose rate of  $1.5 \times 10^4$  reps/hr shortened the stable life of a wide variety of antioxidant-oil combinations to approximately 70% of that obtained in absence of radiation. When the dose rate was increased to  $7.2 \times 10^5$  reps/hr, only 25% of the original stability was retained. In oxidation tests at  $347^\circ\text{F}$  and dose rates of  $7.2 \times 10^5$  reps/hr, only 12% of the unirradiated stability was found for various types of antioxidant-oil blends. The stable lives of all inhibited lubricants tested, in irradiated oxidation-corrosion tests at both  $347^\circ\text{F}$  and  $400^\circ\text{F}$ , appeared to be approaching zero at an estimated dose rate of  $2-5 \times 10^6$  reps/hr. The total  $\gamma$ -ray dose absorbed during the stable life of the various antioxidant-oil blends depended on the relative stability of the blend, the temperature of the test and the radiation dose rate, but in no case was it greater than  $2 \times 10^7$  reps.

No protection against antioxidant radiation damage was found through use of radical scavengers or energy transfer agents (various iodine-containing compounds or aromatic materials). Base oil damage could be reduced through use of such materials but the reactivity of antioxidants towards radicals was considerably greater than compounds added as protectors.

The dialkyl selenides show some promise for use with ester or hydrocarbon base oils at temperatures and radiation levels somewhat above those attainable with the more usual antioxidants. On an equal weight basis, the dialkyl selenides are slightly less effective than phenothiazine in imparting oxidation stability or in protecting ester base oils from radiation damage. However, they can be used in much higher concentrations without greatly affecting the deposit-forming tendency of the lubricant. The selenides must be kept from contact with copper and silver to avoid corrosion and loss of activity.

For use at bulk oil temperatures above  $400^\circ\text{F}$  or at total radiation doses of  $10^8$  reps or higher, inherently-stable, aromatic-containing materials will probably be needed. Preliminary work with specifically designed compounds has been directed towards determining the advantages and disadvantages of the various types of substituents and linkages that can be used with aromatic compounds. Tertiary butyl and carbomethoxy groups are promising substituents for use with benzene derivatives.



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Silyl, ether, and ester groups can be used to link aromatic nuclei without large reduction in oxidation or radiation resistance. Preliminary examination of some of these synthetic compounds shows that their useful life, determined in oxidation tests at 400°F, would be at least 10-20 times longer than the most stable of the MIL-L-7808C ester lubricants. Additional aromatic derivatives are being prepared and examined in various high temperature screening tests.

WADC TR 57-199  
ASTIA Document No. AD 131050

September 1957

**SUBJECT:** EVALUATION OF AN ARYLUREA-PENTAERYTHRITOL ESTER GREASE  
IN WHEEL BEARINGS OF INTERNAL TYPE BRAKE WHEEL ASSEMBLIES

**INVESTIGATOR:** D. T. Kjerland, 2/Lt

**ABSTRACT:** The use of Specification MIL-L-3545 greases in wheel bearings of internal type brake assemblies has proven unsatisfactory. High temperatures encountered in wheel bearings as the result of heat dissipation from the brakes during aircraft take-off and landing operations have caused MIL-L-3545 greases to run out of the wheel bearings. An arylurea-pentaerythritol grease (MLG-9373) developed by Standard Oil Company (Indiana) under Contract AF 33(038)-23687 was subsequently evaluated in wheel bearings of laboratory test rigs of internal type brake assemblies and in wheel bearings of aircraft using the internal type brake assembly. Complete physical, chemical, and mechanical laboratory tests were performed on MLG-9373.

MLG-9373 grease gave satisfactory performance in all the wheel bearings of the various internal type brake assemblies tested from -65°F to temperatures in excess of 350°F while under heavy loads.

Specification MIL-L-25760 (USAF) has been written covering this type of grease. In the near future, greases qualified under Specification MIL-L-25760(USAF) will be required as wheel bearing lubricants for internal type brake assemblies and other wheel bearing applications requiring a lubricant having a wider temperature range than Specification MIL-L-3545 greases.

WADC TR 57-255  
ASTIA Document No. AD 131065

September 1957

**SUBJECT:** NUCLEAR RADIATION RESISTANT TURBINE ENGINE LUBRICANTS

**INVESTIGATOR:** Alfred H. Matuszak

**CONTRACT:** AF 33(616)-3181

**CONTRACTOR:** Esso Research and Engineering Company

**ABSTRACT:** This report covers the results of work aimed at developing radiation resistant turbine engine lubricants. The work has been divided

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into two parts: (1) Phase I covering finished lubricants and (2) Phase II covering potential lubricating oil base stocks, all screened at dosages up to  $10^8$  roentgens.

In the Phase I program, twenty-four oils were screened. These included finished or slightly modified mineral-based aviation oils, ester-based synthetic lubricants and a polyalkylene glycol mono ether lubricant. At  $10^7$  roentgens, no appreciable changes occurred in lubricant properties. At  $10^8$  roentgens, certain modifications of the mineral oils evaluated showed good retention of original physical and chemical properties. With uncompounded oils (Esso Aviation Oil 65 and 100) borderline performance was obtained in the MIL-L-7808 foam test and in the  $347^\circ\text{F}$ . oxidation-corrosion stability test. These deficiencies can be partially overcome through the incorporation of additives. For improved oxidation-corrosion stability, phenyl-alpha-naphthylamine is beneficial. However, silicones are not very effective in maintaining foam suppression after irradiation. Additional work will be required to improve the foaming tendencies of mineral oils. This work will be undertaken using Barosa 56 as the base oil because of its slightly better test performance characteristics.

WS-3019, an MIL-L-25336 type synthetic lubricant, showed somewhat greater stability toward irradiation than any of the other synthetic materials evaluated. It retained most of its original properties and generally met the MIL-L-25336 requirements deviating only slightly in the  $347^\circ\text{F}$ . oxidation-corrosion stability test. It exhibited, however, an undesirable flash point degradation (from  $450$  to  $355^\circ\text{F}$ . after  $10^8\text{r}$ ) which can be attributed to a small amount of irradiation-produced contaminants.

A third oil, LO-2062, an MIL-L-7808 type adipate oil was indicated to have some promise providing its copper corrosion tendencies after irradiation can be corrected. The addition of about 1% tricresyl phosphate appears to offer a possible fix for improved stability up to gamma dosages of  $10^8$  roentgens. The effect of tricresyl phosphate in reducing copper corrosion at higher dosages will be investigated.

In the Phase II program twenty-three base stocks of varying chemical structures were screened at  $10^7$  and/or  $10^8$  roentgens. As in the case of Phase I lubricants the Phase II lubricant base stocks were evaluated against Military specification tests, notably MIL-L-7808 and MIL-L-9236. The most attractive materials evaluated were the inhibited mineral oil base stocks of which Barosa 56, a paraffinic base, is representative. Evaluation of this material at higher gamma dosages ( $5 \times 10^8$  and  $10^9$  roentgens) is contemplated. A sample of this base stock has been sent to WADC for more extensive laboratory and engine evaluations.

In the ester field, di-tridecyl carbonate appeared to be the most resistant to  $10^8$  roentgens. It exhibited good retention of physical properties but showed moderately high panel coking deposits.

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Additional work is planned with other carbonates at higher screening dosages. Higher flash diesters, such as di-tridecyl adipate, azelate and sebacate also showed promise of approaching a reasonable resistance to  $10^8$  roentgens. Their tendency to corrode copper and magnesium will require some additional attention. In this connection, the ameliorating effect of tricresyl phosphate will be investigated.

In the future program this initial effort to develop a radiation resistant lubricant will be expanded to include the evaluation of other synthetic and petroleum type finished oils and base stocks. Particular stress will be placed on higher flash base stocks, many of which are available commercially. Others may have to be synthesized. Any promising materials coming out of this screening program will be tested at higher gamma dosages. The most attractive base stocks will then be blended into finished oil formulations and finally evaluated further as radiation resistant engine lubricants.

WADC TR 57-283  
ASTIA Document No. AD 130902  
OTS Release

July 1957

SUBJECT: EVALUATION OF ANTI-WEAR AND EXTREME PRESSURE CHARACTERISTICS OF NEW HETEROCYCLIC COMPOUNDS  
INVESTIGATOR: Robert J. Benzing  
ABSTRACT: A series of nineteen compounds were studied as possible anti-wear additives or extreme pressure additives. The materials were evaluated in Di(2-ethylhexyl)sebacate in the Shell Four-Ball Wear Tester and Shell Extreme Pressure Tester.

The basic types studied were compounds of heterocyclic structure containing one or more of the following heteroatoms: nitrogen, oxygen, selenium and sulfur. Some gave promise as good wear and extreme pressure additives.

WADC TR 57-455  
ASTIA Document No. AD 150982  
OTS Release

February 1958

SUBJECT: HIGH TEMPERATURE SOLID DRY FILM LUBRICANTS  
INVESTIGATOR: Melvin T. Lavik  
CONTRACT: AF 33(616)-3684  
CONTRACTOR: Midwest Research Institute  
ABSTRACT: This report covers the development and evaluation of a dry lubrication test machine and the testing of several dry film lubricants. In order to meet the very high temperature and pressure requirements currently placed on lubricants in the aircraft industry, it was

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necessary to devise a suitable test instrument. The first part of this program was spent in designing, constructing and calibrating such a device capable of testing dry film lubricants at pressures as high as 50,000 psi and at temperatures up to 800°F. Several minor revisions have been made during the program leading up to the machine as reported here.

Several commercial dry film lubricants and also some materials prepared at the Institute were investigated for both friction coefficient and wear life at the various temperatures and pressures available with the test machine. In general, the materials worked with, while effecting reasonable lubrication, did not exhibit the wear life which is desired. The lubricant properties of several materials are discussed in this report, with the discussion serving mainly to point out areas in which further information is needed to realize the full benefits available from these materials.

## MATERIALS PHYSICS

WADC TR 56-4 Pt I  
ASTIA Document No. AD 131096

September 1957

SUBJECT: THE THERMODYNAMIC PROPERTIES OF MOLTEN SALTS:  
Part I. Molybdenum Hexafluoride

INVESTIGATOR: A. P. Brady  
J. K. Clauss  
O. E. Myers

CONTRACT: AF 33(616)-2558

CONTRACTOR: Stanford Research Institute

ABSTRACT: Assembling and testing of apparatus for measuring heat capacities from about 50°K to room temperature has been completed, including calibration of thermocouples by comparison with a platinum resistance thermometer and with a copper one. This apparatus has been used to determine the absolute entropy of molybdenum hexafluoride vapor at 298.16°K. The result,  $79.7 \pm 0.6$  cal/mole·deg, agrees well with that calculated from its spectrum, 80.05 cal/mole·deg. A previously unreported solid-solid transition was found to occur at 263.6°K (-9.6°C). A solution calorimeter has also been assembled and tested. By comparison of the heat of solution in sodium hydroxide of molybdenum hexafluoride with that of the oxide, the heat of formation of MoF<sub>6</sub> (l) at 298.16°K was found to be -390.9 kcal/mole. From these values the corresponding standard free energy of formation is computed to be -363.1 kcal/mole. These data, together with data in the

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literature, have allowed calculation of the desired thermodynamic functions over the temperature range of interest.

WADC TR 56-4 Pt II  
ASTIA Document No. AD 131097

September 1957

SUBJECT: THE THERMODYNAMIC PROPERTIES OF MOLTEN SALTS:  
Part II. Tungsten Hexafluoride, Niobium Pentafluoride,  
and Vanadium Trifluoride

INVESTIGATOR: Orlo E. Myers

CONTRACT: AF 33(616)-2558

CONTRACTOR: Stanford Research Institute

ABSTRACT: Assembly and adjustment of apparatus for measuring enthalpies from 50 to 1200°C has been completed. A new solution calorimeter suitable for use with metallic ampoules for measurements with fluorides has been constructed. An improved low temperature calorimeter has been built with the aid of electroforming techniques. This equipment has been used in conjunction with low temperature equipment previously described to obtain thermodynamic data for the following:

Tungsten hexafluoride vapor (from heat of solution and spectra):

$$\begin{array}{lll} \Delta H_{\text{form,}} & 298.16 & = -416.3 \text{ kcal/mole} \\ \Delta F_{\text{form,}} & 298.16 & = -394.8 \text{ kcal/mole} \\ S_{298.16} & & = 81.75 \text{ cal/mole}\cdot\text{deg} \end{array}$$

Niobium pentafluoride (from heat capacity, enthalpy, and heat of solution):

$$\begin{array}{lll} \Delta H_{\text{form,}} & 298.16 & = -431 \text{ kcal/mole} \\ \Delta F_{\text{form,}} & 298.16 & = -404 \text{ kcal/mole} \\ S_{298.16} & & = 38.30 \text{ cal/mole}\cdot\text{deg} \\ \Delta H_{\text{fusion}} & & = 2.92 \text{ kcal/mole} \end{array}$$

Vanadium trifluoride (from heat capacity and enthalpy):

$$S_{298.16} = 23.05 \text{ cal/mole}\cdot\text{deg}$$

Excellent agreement was found between the last entropy and the predicted value from the theory of the late Prof. Latimer concerning the summation of ionic entropy contributions. Values of the above properties at temperatures up to 1400°K have been calculated when possible or estimated from experimental data and standard methods.

WADC TR 56-19 Pt I  
ASTIA Document No. AD 130855  
OTS Release

June 1957

**SUBJECT:** ANALYSIS OF FLUORINATED ORGANO-METALLICS  
Part I - Determination of Fluorine and Silicon in Organic Fluoro-Silicon Compounds

**INVESTIGATOR:** Otto Schwarzkopf  
Rosemarie Heinlein  
Nora E. Srp

**CONTRACT:** AF 33(616)-3067

**CONTRACTOR:** Schwarzkopf Microanalytical Laboratory  
Materials Laboratory

**ABSTRACT:** Development of analytical methods to determine fluorine and silicon in organic fluoro-silicon compounds was the object of this project. A method having a precision of  $\pm 1\%$  for fluorine and  $\pm 0.7\%$  for silicon was developed. The test sample is reacted with potassium metal in a pure nickel bomb heated to  $650^{\circ}\text{C}$  for two hours. The fusion mixture is dissolved in water and after filtering, fluorine is precipitated as  $\text{PbClF}$  and filtered. The filtrate containing silicon is used for the determination of silicon in the form of  $\text{SiO}_2$ .

This method needs more testing with a variety of organic fluoro-silicon compounds.

WADC TR 56-19 Pt II  
ASTIA Document No. AD 130781  
OTS Release

June 1957

**SUBJECT:** ANALYSIS OF FLUORINATED ORGANO-METALLICS  
Part II. Determination of Fluorine, Boron and Silicon in Organic Fluoro-Silicon or Fluoro-Boro-Silicon Compounds

**INVESTIGATOR:** Otto Schwarzkopf  
Rosemarie Heinlein

**CONTRACT:** AF 33(616)-3552

**CONTRACTOR:** Schwarzkopf Microanalytical Laboratory

**ABSTRACT:** Development of analytical methods, for the determination of the elements silicon, fluorine and boron present in organic compounds containing two or three of the elements mentioned, was the object of this project.

For the determination of fluorine and silicon present in organo-fluoro-silicon compounds a method has been devised wherein fluorine is determined as  $\text{PbClF}$  and silicon as  $\text{SiO}_2$  in the filtrate from  $\text{PbClF}$ .

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A more satisfactory method was the determination of fluorine as  $PbClF$  and the determination of silicon as oxine-silicomolybdate. It was possible to determine silicon either in the presence of fluorine after converting fluorine to fluoboric acid, or to precipitate  $PbClF$  and determine silicon in the filtrate.

The three methods were tested with a variety of test samples. The preferred method is the determination of fluorine as  $PbClF$  and the determination of silicon as oxine salt of silicomolybdic acid after conversion of fluoride to fluoborate. The precision of this method was found to be  $\pm 0.3\%$  (absolute).

The presence of boron did not interfere in the determination of fluorine or of silicon carried out by the above mentioned method.

While silicon was without influence, fluorine interfered in the usual determination of boron by the boric acid mannitol titration method. Fluoride and boric acid react in acid medium forming fluoboric acid. This reaction made it impossible to eliminate carbon-dioxide completely prior to the addition of mannitol. The first neutralization endpoint was uncertain and high results for boron were the consequence.

Satisfactory results were obtained in the following manner. Fluorine is precipitated as  $PbClF$  at  $pH=7$ , and filtered. To the filtrate sulfuric acid is added precipitating the excess lead and bringing the  $pH$  below  $pH=1$ . The aqueous solution is concentrated in a distilling apparatus. Volatile weak acids - organic and inorganic are distilled and the distillate is discarded. Methanol is added to the residue and by keeping the water content low, boric acid is converted into methylborate which is distilled, hydrolyzed by  $NaOH$  and titrated by the mannitol titration method.

WADC TN 56-183  
ASTIA Document No. AD 130926

July 1957

SUBJECT: THE CONSOLIDATED ANALYTICAL MASS SPECTROMETER AND ITS APPLICATIONS  
INVESTIGATOR: George Howan  
ABSTRACT: The mass spectrometer principle of separating ionized molecules according to their mass forms the basis of one of today's most effective methods of analyzing highly complex gas or liquid mixtures. Routine control analyses, purity determinations, exploratory analyses, and fundamental research investigations are all proven applications of the analytical mass spectrometer.

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The scope is extremely broad as low vapor pressure materials can be analyzed by using the heated sample inlet at temperatures up to 150°C. An overall range of 1 to 700 mass units is provided for use by the operator. High sensitivity detects impurities in concentrations as low as 5 ppm. Sample size can be as little as 0.1 ml gas and less than 0.001 cc liquid. Deviation normally is within the range of 0.05 to 1.0 mol per cent and usually less than might be expected from most other methods of analysis.

The instrument described in this report is the Consolidated Electroynamics Corporation (CEC) 180° Analytical Mass Spectrometer. Details regarding the scope, theory of operation, and various applications of the mass spectrometer are included.

WADC TN 56-191 Pt I  
ASTIA Document No. AD 130864  
OTS Release

June 1957

SUBJECT: CALCULATION OF THE RADIATION DOSE DELIVERED BY BETA  
EMITTING ISOTOPES

Part I - Point and Thin Plane Sources in Air

INVESTIGATOR: Edward A. Burke, 1/Lt

ABSTRACT: This report is the third in a series concerned with the development of methods for calculating the radiation dose delivered by beta emitting isotopes. This report extends the basic equations previously developed for all beta emitting isotopes. The publication by R. Loevinger of extensive experimental data on beta isotopes has been a major factor in making the present study possible since the equations developed are empirical and depend solely upon such data for their validity.

An empirical function for the dosage distribution around a point source of phosphorous -32 presented in WADC TN 56-101 has been extended to include all other beta emitting isotopes and found to be in excellent agreement with available experimental data.

WADC TR 56-436  
ASTIA Document No. AD 151081

March 1958

SUBJECT: THE RETENTION OF FISSION PRODUCTS BY SOILS UNDERLYING  
THE NUCLEAR ENGINEERING TEST FACILITY AT WADC

INVESTIGATOR: Bruce Raby  
Dr. George John

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**ABSTRACT:** This report primarily concerns the retention of fission products by soils underlying the site for the Nuclear Engineering Test Facility. The purpose of the study was to provide some data which would be of value in planning for the contingency of a reactor "run-away" and for possible seepage of contaminated water during normal operation.

Three types of experiments were conducted: (1) total capacity studies of the soils, (2) equilibrium sorption of ions by the soil in a static system, and (3) elution of ions from the soil by water. These experiments were of an exploratory nature, and although not complete, do give an indication of the soils' ability to retain various fission products. However, on the basis of the few experiments performed, it is possible to provide only qualitative information and to indicate the direction and extent of future studies.

WADC TR 56-455  
ASTIA Document No. AD 130797  
OTS Release

June 1957

**SUBJECT:** STUDIES OF QUANTITATIVE METHODS FOR THE SEPARATION AND DETERMINATION OF ZIRCONIUM AND THORIUM IN MAGNESIUM ALLOYS

**INVESTIGATOR:** B. A. Raby, 1/Lt

**ABSTRACT:** This paper describes the results of a comparative study of analytical methods suitable for the determination of thorium and zirconium in magnesium base alloys.

In addition, this paper describes an attempted amalgamation of selected analytical methods and ion exchange techniques into a scheme for the quantitative analysis of thorium and zirconium in these alloys. Manganese and zinc, common constituents of magnesium base alloys, were carried in the experiments, but only to study their interference in the proposed scheme.

The sample is dissolved in 12N-HCl and the resulting solution is percolated through a column containing Dowex 2-X8 anion exchange resin. Magnesium and thorium pass through the column while zirconium, zinc, and manganese are absorbed. The latter elements are removed by eluting the column with water. The thorium and zirconium in the separated fractions can be determined by means of the complexometric titration methods described by J. S. Fritz.

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WADC TR 56-465  
ASTIA Document No. AD 130801  
OTS Release

June 1957

**SUBJECT:** ELECTRICAL PROPERTIES OF IRRADIATED POLYMERS  
**INVESTIGATOR:** Ralph E. Woodard  
**ABSTRACT:** A brief discussion is given of the electronic nature of solids as it applies to insulators and then polymers. The fundamental theory of nuclear radiation effects is discussed and shown to be associated with the laws of energy dissipation of these radiation particles within a material. A survey is given on the role that nuclear radiation plays in polymer kinetics. Work of various investigators on the electrical properties of irradiated polymers is reported. An analogy is drawn between photoconductivity in semi-conductors and nuclear radiation induced conductivity in certain polymers. The instantaneous nature of this induced conductivity is emphasized. The change produced in the dielectric constant of an irradiated polymer is considered in terms of crosslinking and dipole moments. A survey is included of the available engineering data for irradiated polymers. Factors affecting radiation to the electrical properties of two tetraarylmmonosilanes is given.

WADC TR 56-515  
ASTIA Document No. AD 142343  
OTS Release

February 1958

**SUBJECT:** EFFECTS OF GAMMA RADIATION ON LINEAR POLYETHYLENE  
**INVESTIGATOR:** William B. Terrell, Captain  
Jack T. Humphries, 1/Lt  
**ABSTRACT:** Linear polyethylenes have recently been developed which have empirical chemical formulae identical to those of common polyethylenes, but which differ in the molecular arrangement of the polymer chain. The physical properties of two linear polyethylenes were measured before and after gamma irradiation to afford a comparison with conventional polyethylene.

Measurements were made of the following properties as functions of total radiation dosage up to  $10^9$  roentgens: tensile strength, elongation, hardness, impact strength, specific gravity, water absorption, heat distortion temperature, dielectric constant, arc resistance, color, and infrared absorption. In addition, densities were determined as functions of both radiation dosage and temperature.

WADC TR 56-548  
ASTIA Document No. AD 118062  
OTS Release

May 1957

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**SUBJECT:** MEASUREMENT OF RADON GAS IN CYLINDRICAL IONIZATION CHAMBERS  
**INVESTIGATOR:** Edward A. Burke, 1/Lt  
**ABSTRACT:** Three of the fundamental problems encountered in the measurement of radon gas using cylindrical ionization chambers are discussed and solutions presented. These are: variation of ionization current with short half-life radon daughters, effect of dielectric absorption, and calibration of the ionization chamber.

WADC TR 56-623  
ASTIA Document No. AD 131030

July 1957

**SUBJECT:** DEVELOPMENT OF A TRITIUM IMPREGNATED METAL FOR USE AS THE ACTIVATOR IN A SELF-LUMINOUS PHOSPHOR AND FOR USE AS AN IONIZATION SOURCE  
**INVESTIGATOR:** D. L. Prosser J. G. MacHutchin  
C. H. Wright H. H. Dooley  
J. E. Atherton  
**CONTRACT:** AF 33(616)-3036  
**CONTRACTOR:** United States Radium Corporation  
**ABSTRACT:** A description of the methods and equipment used in the preparation of tritiated foils is presented. The results of the experimental studies indicate that maximum beta emission is obtained from extremely thin layers of titanium tritide which have been prepared on the surface of sandblasted stainless steel backing foils.

Phosphor response studies have indicated that the main factors effecting the response of ZnS type phosphors to tritium beta radiation are (a) phosphor particle size (b) type and concentration of activator and (c) cadmium content.

Six research models of tritium foil-activated self-luminous sources have been prepared for submission to WADC. A description of the research models is provided.

WADC TR 57-10  
ASTIA Document No. AD 131032  
OTS Release

August 1957

**SUBJECT:** MEASUREMENTS OF THE THERMAL PROPERTIES OF VARIOUS AIRCRAFT STRUCTURAL MATERIALS  
**INVESTIGATOR:** Perry C. Covington  
Sabert Oglesby, Jr.

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CONTRACT: AF 33(616)-3328  
CONTRACTOR: Southern Research Institute  
ABSTRACT: This report describes the measurements of the thermal properties of a group of structural panels and cores. Thermal expansion, thermal conductivity, and specific heat were determined for three honeycomb cores, one foamed-core sandwich panel, one laminated panel, and five sandwich panels with various honeycomb cores and facing materials. In addition, thermal conductivity determinations were made for two silicone-resin laminates to establish a check for data previously reported on these panels.

The equipment and procedures used in these thermal property determinations are essentially the same as described in WADC TR 54-306 Parts I and II with the exception of additional components necessary to obtain temperatures below ambient temperature.

WADC TR 57-92  
ASTIA Document No. AD 130920  
OTS Release

July 1957

SUBJECT: EXPANSION CHARACTERISTICS OF MARLEX 20 AND MARLEX 50  
INVESTIGATOR: Hyman Marcus  
Frank V. Zaleski, Capt.  
ABSTRACT: A suitable technique was developed for volume dilatometry of plastic materials and the thermal behavior of two commercial linear polyethylene plastics, Marlex 20 and Marlex 50, was observed.

The technique consisted of sealing a test sample in a glass tube with mercury as the confining fluid. The change in volume of mercury as evidenced by its rise in a capillary tube was noted at different temperatures. The volume expansion of the test sample was calculated from the known expansivities of glass and mercury.

The dilatometers used were immersed in an electrically heated oil bath equipped with a motor driven stirrer to insure a uniform temperature distribution.

Test data were obtained for a series of six samples of each material, each series containing one non-irradiated sample and five samples which had received increasing doses of gamma radiation at the Materials Test Reactor Facility. The doses were of the values  $10^6$ r,  $10^7$ r,  $10^8$ r,  $5 \times 10^8$ r, and  $10^9$ r for the irradiated samples.

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WADC TN 57-114  
ASTIA Document No. AD 142066  
OTS Release

October 1957

SUBJECT: X-RAY DIFFRACTION STUDY OF CRYSTALLINE SILANES  
INVESTIGATOR: W. L. Baun, 1/Lt  
ABSTRACT: X-Ray Diffraction patterns for nineteen crystalline silanes are presented. These compounds are of interest because of their high thermal stability and low vapor pressure. They are particularly well suited for X-Ray Diffraction analysis because of their crystalline nature and the individualistic patterns obtained.

WADC TR 57-141  
ASTIA Document No. AD 130857  
OTS Release

June 1957

SUBJECT: A COMPARISON OF HIGH-ENERGY ELECTRON AND GAMMA IRRADIATION EFFECTS ON ORGANIC LIQUIDS  
INVESTIGATOR: Edwin L. Zebroski  
Edwin M. Kinderman  
CONTRACT: AF 33(616)-3738  
CONTRACTOR: Stanford Research Institute  
ABSTRACT: It has been proposed that high-energy electron irradiation be substituted for gamma irradiation in testing the radiation resistance of aircraft materials, components, and systems. To determine the feasibility of the proposal, the effects of electron and gamma radiation on selected organic liquids were compared. Results of this study and the experimental work reported by others indicate that there is qualitative similarity between electron and gamma radiation effects on simple compounds. Some differences in quantitative yields are observed, but these may be a result of differences in time of irradiation rather than of differences in radiation type.

WADC TN 57-207  
ASTIA Document No. AD 142179  
OTS Release

December 1957

SUBJECT: STANDARD INSTRUMENTATION TECHNIQUES FOR NUCLEAR ENVIRONMENTAL TESTING  
INVESTIGATOR: Walter R. Burrus, 1/Lt  
ABSTRACT: The ANP Advisory Committee for Nuclear Measurements and Standards was established to standardize nuclear measurements for all Air Force programs supporting the development of nuclear weapon systems.

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The first phase of this Committee's effort is nearly complete and is concerned with the standardization of nuclear measurements for radiation damage studies. This Technical Note presents and discusses Committee recommendations for standardizing radiation damage measurements. A minimum number of measurements were selected which can provide a consistent basis for comparison of experiments and correlation of data. A measurement of "absorbed dose" in carbon is recommended as the minimum measurement of the gamma radiation field. In addition, determination of the activation of U-238, Pu-239 (boron shielded) and sulphur is recommended as the minimum measurement for neutron radiation. Consistent units and symbols for reporting these measurements are recommended. Additional techniques will be recommended in the future.

WADC TR 57-212  
ASTIA Document No. AD 131063  
OTS Release

September 1957

**SUBJECT:** X-RAY EMISSION LINES AND 2θ VALUES FOR LITHIUM FLUORIDE ANALYZING CRYSTAL  
**INVESTIGATOR:** W. L. Baun  
R. E. Brocklehurst  
**ABSTRACT:** A complete table of x-ray emission lines and 2θ values, using a lithium fluoride analyzing crystal, is presented. This table includes all the lines of the K, L, and M series, order 1 to order 9.

WADC TR 57-226  
ASTIA DOCUMENT NO. AD 142093  
OTS Release

November 1957

**SUBJECT:** MEASUREMENT OF THERMAL DIFFUSIVITY OF VARIOUS MATERIALS BY MEANS OF THE HIGH INTENSITY ELECTRIC ARC TECHNIQUE  
**INVESTIGATOR:** Charles Sheer Donald L. Rothacker  
Lawrence H. Mead Leonard H. Johnson  
**CONTRACT:** AF 33(616)-3669  
**CONTRACTOR:** Vitro Laboratories  
**ABSTRACT:** Studies have been made of the tail flame of a high-intensity electric arc to determine its suitability as a medium for testing materials under sustained high-temperature gaseous flow. The first year effort reported herein centered on the establishment of an electrode geometry for stable operation, measurements of the temperature and velocity distribution in the tail flame at varying ambient pressures, and qualitative observations of the electrodynamic, magnetic and thermodynamic properties of the tail flame.

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The electrode geometry giving optimum flame stability consists of a triple cathode arrangement symmetrical with respect to the anode. A somewhat less favorable configuration utilizes a single cathode forming an acute angle with the anode axis.

The axial temperature distribution in the tail flame was measured by spectral line reversal technique for temperatures below 3200°K, and by spectral band analysis above this temperature. The data obtained by line reversal show that the temperature decreases with decreasing ambient pressure and increasing distance from the crater. The spectrographic data are currently in the process of reduction.

Flame velocity was measured by a modification of Rohloff's method, involving a temporary interruption of the arc and observation of the downstream propagation of the resulting disturbance by high-speed cinematography. The velocities measured thus far were 47, 78 and 150 m/sec at pressures of 1.0, 0.5 and 0.1 atmospheres, respectively. The increase of velocity with decreasing pressure is in general accordance with classical laws although the variation here appears to be less than linear, probably due to an increase in flame diameter with decreasing pressure.

Miscellaneous exploratory observations have shown the tail flame to be markedly diamagnetic and to possess a statistical dipole distribution of positive ions and free electrons. Also observed were strong plasma oscillations in the order of 2 kc and with peak amplitudes of 20 volts; this effect suggests the possibility of measuring charge density of both signs in the flame.

Surface heat flux rates were measured at 1.0 and 0.1 atmospheres on copper bodies in thermal equilibrium. The values obtained were 4.0 and 1.0 kw/cm<sup>2</sup> respectively, but it is emphasized that further measurements are required to corroborate these figures. The appearance of the gaseous flow about blunt and conical wedges in the presence and absence of coulomb potentials showed a distinct separation zone between body and flame, the thickness depending on ambient pressure and electrical polarity. This phenomenon is believed to be associated with a magneto-hydrodynamic effect.

Finally, diffusivity measurements were made in the tail flame on OFHC copper and graphite plates. The data obtained thus far agree with literature values in order of magnitude. Closer refinements in the instrumentation and evaluation techniques appear necessary before the tail flame can be used for measurements of this type.

WADC TN 57-294  
ASTIA Document No. AD 142186  
OTS Release

December 1957

**SUBJECT:** DETERMINATION OF HYDROGEN IN ZIRCONIUM HYDRIDE  
**INVESTIGATOR:** Wade H. Jones  
**ABSTRACT:** It is now generally recognized that control of the hydrogen content in zirconium is important in the manufacture, melting, fabrication and subsequent use of the metal. It is therefore highly desirable to have a simple, rapid, precise method for determination of hydrogen in zirconium. Such a method is described. It is based on the measurement of the equilibrium pressure of hydrogen over the metal in a closed system under predetermined conditions.

This report covers the analysis of thirty-five samples of zirconium hydride with temperatures ranging from 1900°C to 1000°C and collection times from 10 to 30 minutes.

WADC TN 57-298 Pt I  
ASTIA Document No. AD 142342  
OTS Release

February 1958

**SUBJECT:** PROCEEDINGS OF THE ANP SPECTROSCOPY INFORMATION MEETING HELD AUGUST 6-7 AT WRIGHT AIR DEVELOPMENT CENTER  
**INVESTIGATOR:** Robert E. Brocklehurst  
**ABSTRACT:** This report is a compilation of the unclassified papers given at the ANP Spectroscopy Information Meeting held at Wright-Patterson Air Force Base, August 6 and 7, 1957. The subjects covered in these proceedings are instrumentation for gamma and neutron spectral measurements, treatment of experimental data, characteristics of scintillation crystals, and the use of beam collimators.

WADC TN 57-308  
ASTIA Document No. AD 142095  
OTS Release

November 1957

**SUBJECT:** CRITIQUE ON THE ANALYTICAL REPRESENTATION OF SPECIFIC HEAT DATA  
**INVESTIGATOR:** Richard Pawel, 1/Lt  
**ABSTRACT:** A considerable lack of agreement exists in the literature for high temperature specific heat values. Since the majority of these data have been obtained indirectly through enthalpy measurements, the problem of data treatment is added to those of the experiment. It is

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thought that the present uncertainties in the methods of curve fitting the enthalpy data can yield misleading trends to the resulting specific heat curves. For this reason, it appears wise to consider the final result in the light of simple specific heat theory and the accuracy and reproducibility of the original enthalpy data. The complexity of the analysis should be based upon the reliability of the enthalpy data to avoid "over-fitting". It appears that in the absence of exceptionally precise enthalpy data, a quadratic expression will satisfactorily describe the data, resulting in a linear specific heat representation. This simple representation is sometimes more compatible than those obtained by other means.

WADC TR 57-319

February 1958

ASTIA Document No. AD 150961

SUBJECT: DETERMINATION OF THE THERMAL CONDUCTIVITY OF MIXED AND SYNTHETIC ENGINE OILS BY THE UNSTEADY-STATE METHOD  
INVESTIGATOR: E. E. Kohnke  
A. R. Schmidt  
CONTRACT: AF 33(616)-3544  
CONTRACTOR: Oklahoma Agricultural and Mechanical College  
ABSTRACT: Results are reported for the thermal diffusivities of five mineral and synthetic oils as measured by an unsteady-state method at temperatures between 80°F and 430°F. Maximum measurement temperatures vary depending upon the chemical nature of the various oils.

Experimental apparatus is described and methods of data analysis discussed. A critical evaluation of the potentialities of unsteady-state methods in general and of the particular experimental procedure employed are presented. Suggestions for improvement are offered.

WADC TR 57-334

April 1958

ASTIA Document No. AD 151163

SUBJECT: EVALUATION OF THE MECHANISMS WHICH AFFECT THE PERFORMANCE OF "THERMAL RADIATION RESISTANT COATINGS"  
INVESTIGATOR: Louise E. Moore Edwin H. Tompkins  
Matthew Prastein Donald O. Van Ostenburg  
CONTRACT: AF 33(616)-3595  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: This is the final report on Contract AF 33(616)-3595, ARF Project C 097, for Wright Air Development Center, covering the period from May 1, 1956 through April 30, 1957. The theory of absorption,

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reflection, and scattering of electromagnetic radiation is presented in a classical review and for harmonically bound electrons and spheres. The absorption spectra of possible pigments and vehicles are considered in terms of incident radiation from 3000°K and 6000°K black body radiators, with possible modification by atmospheric absorption. Although refractive indices of vehicles are largely between 1.4 and 1.7, pigments may have high refractive indices, and calculations are made to determine indices of pigments. In systems less than 6% pigment by volume, the statistical distribution of the particles is largely singly or in groups of two. Procedures to determine the size distribution of the particles are illustrated with rutile. A first approximation to an optimum size distribution is a modified black body curve. The wavelength axis, contracted by the factor 0.41, is the diameter axis for rutile in linseed oil.

WADC TR 57-334 Sup 1  
ASTIA Document No. AD 151164

April 1958

**SUBJECT:** EVALUATION OF THE MECHANISMS WHICH AFFECT THE PERFORMANCE OF "THERMAL RADIATION RESISTANT COATINGS"

**INVESTIGATOR:** C. Roland McCully      F. V. Schossberger  
Louise E. Moore      Edwin H. Tompkins

**CONTRACT:** AF 33(616)-3595

**CONTRACTOR:** Armour Research Foundation

**ABSTRACT:** The work covered in this report was done for the Wright Air Development Center under Contract No. AF 33(616)-3595 over the period from May 1, 1956 to May 1957. This report is a supplement to the Final Report: Theory, which gives an analysis of the mechanisms of scattering and absorption in highly-reflective paint coatings. This supplement is intended to serve as a guide in the formulation of reflective coatings.

The thermal sources for the energy to be reflected were specified in the contract to be 3000°K and 6000°K black bodies, which yield peak intensities at 1.0 micron and 0.50 micron, respectively. The modification of the energy distributions from these sources by atmospheric absorption is discussed to show how such absorption might be used to protect an aircraft.

The optical characteristics required in vehicles and pigments suitable for reflective coatings are summarized, and tentative recommendations are made. For vehicles, fluoroethylenes (Kel-F, Teflon), silicones, vinylidene chloride-acrylonitrile copolymers, and epoxy resins are considered suitable; for pigments, rutile and colorless silicon carbide are outstanding, but boron nitride and colorless barium titanate also have possibilities. Since titanium dioxide is readily available its methods of manufacture are discussed in some detail to show how particle size and impurity content are controlled.

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On the basis of several approximations, first-order particle size distributions required in rutile pigments for the reflection of 3000°K and 6000°K radiation are presented. Centrifugal sedimentation and elutriation techniques are described for the classification of pigments and the formulation of specified size distributions. The presence of considerable infrared in the radiations to be reflected requires the presence of larger particle sizes than are normally present in conventional white pigments. This is particularly true for 3000°K radiation, where the median particle size should be about 0.45 micron instead of the 0.25 micron size present in commercial rutile pigments. If a coating having a median size of 0.25 micron were exposed to 3000°K radiation, about half of the radiation would penetrate to the base of the coating. For 6000°K radiation the penetration would be about 20%, which is still seriously large.

WADC TN 57-335  
ASTIA Document No. AD 142249  
OTS Release

December 1957

SUBJECT: FEASIBILITY OF A GRAPHITE-CARBON DIOXIDE IONIZATION CHAMBER TO MEASURE CARBON DOSE AT HIGH DOSE RATES  
INVESTIGATOR: Robert L. Hickmott  
ABSTRACT: This report discusses the feasibility of designing a graphite-carbon dioxide ion chamber for the absolute measurement of the carbon dose rate from a pure gamma field up to  $10^{10}$  erg/g/hr. There is insufficient data on neutron response to judge the feasibility of using the ion chamber in mixed fields with large neutron components.

WADC TN 57-342  
ASTIA Document No. AD 142277  
OTS Release

January 1958

SUBJECT: THE QUANTITATIVE ANALYSIS OF THE FUNGICIDE 3,3'-DIFLUORO 4,4'-DIHYDROXYBIPHENYL  
INVESTIGATOR: Myron L. Dunton  
ABSTRACT: A quantitative method is described for determining the quantity of the fungicide 3,3'-difluoro 4,4'-dihydroxybiphenyl which has been added to white duck cloth. This method involves extraction with methanol and subsequent ultra-violet spectrophotometric measurement. It also is suggested that this method may be applied to the analysis of other related fungicides.

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WADC TR 57-359  
ASTIA Document No. AD 142010  
OTS Release

September 1957

**SUBJECT:** ANALYTICAL APPLICATIONS OF FAR INFRARED SPECTRA I  
HISTORICAL REVIEW, APPARATUS AND TECHNIQUES

**INVESTIGATOR:** Freeman F. Bentley Nora E. Srp  
Eugene F. Wolfarth Wilbert R. Powell

**ABSTRACT:** The infrared spectrum of a molecule is one of its most unique physical properties, and perhaps for this reason a large amount of spectral data have been amassed over the last few decades. Most of the work has been limited to the fundamental region of the spectrum since characteristic group frequencies capable of giving structural information of great value and precision occur in this region, and because it has been the easiest region to study accurately and rapidly.

Many characteristic group frequencies are also observed in the long wave length region of the spectrum which are useful in analytical and structural determinations. Furthermore, spectra obtained in this region are particularly useful in material characterization. Slight changes in structure produce considerable changes in spectra, giving a more specific "fingerprint". Investigations in the far infrared region have been limited due to instrumental difficulties such as high atmospheric absorption, low energy, stray radiation, and the lack of suitable prism materials.

A review of the work accomplished to date in the far infrared region is presented in this paper, and most of the available literature is cited. A double beam double pass spectrophotometer equipped with cesium bromide optics is described. This instrument extends the useful spectral range to 35 microns. The general features of the instrument's construction and its performance capabilities are discussed.

Conventional infrared techniques are used, and in some instances simplified in obtaining infrared spectra in the cesium bromide region. Many common solvents such as carbon disulfide, furan, benzene, and dioxane, have large "open windows" in the long wavelength region and the infrared spectra of these and other solvents are given. The advantages of far infrared spectra, and some of the special techniques of obtaining infrared spectra in the cesium bromide region, are also discussed.

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WADC TN 57-362

ASTIA Document No. AD 142168

November 1957

SUBJECT: RESISTANCE OF AIR CREW CLOTHING MATERIAL TO THERMAL RADIATION

INVESTIGATOR: Ben J. Lisle  
Richard L. Peckham, 2/Lt

ABSTRACT: Twelve assemblies of flight clothing materials presently in use were tested to determine their resistance to thermal radiation. The assemblies were irradiated at intensities of 10 cal/cm<sup>2</sup> sec and 30 cal/cm<sup>2</sup> sec. The results were evaluated with respect to the total amount of energy necessary to produce the visible effects of scorch, char, and burn. These evaluations were aimed at determining the relative damage caused by a specific quantity of thermal energy incident upon the assembly.

WADC TR 57-374 Pt I

ASTIA Document No. AD 142119

November 1957

SUBJECT: PHYSICAL PROPERTIES OF HIGH TEMPERATURE MATERIALS  
Part I. New Apparatus for the Precise Measurement of Heat Content and Heat Capacity from 0° to 1500°C

INVESTIGATOR: Thomas B. Douglas  
William H. Payne

CONTRACT: AF 33(616)-56-21

CONTRACTOR: National Bureau of Standards

ABSTRACT: There is described in detail new apparatus for the accurate measurement of the heat content and heat capacity of solids and liquids over the temperature range 0° to approximately 1500°C (32° to 2750°F). For use of the "drop" method, a new Bunsen ice calorimeter and resistance-heated furnace were designed, and have been partly constructed. The ice calorimeter is similar to that developed earlier at the National Bureau of Standards, but embodies several minor modifications. To achieve regions of known and uniform temperature, the furnace was designed to have three concentric cores wound with 17 independently controlled wire-heater sections. Temperatures will be measured by several platinum-rhodium thermocouples, which are arranged to permit their periodic recalibration in place. After the performance of the apparatus has been tested, the first measurements undertaken are to be on standard-sample synthetic sapphire (aluminum oxide). These data will augment the Bureau's earlier measurements on this material from 0° to 900°C (32° to 1650°F), and permit its use as a heat-capacity standard up to approximately 1500°C (2750°F).

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WADC TR 57-374 Pt II

August 1957

**SUBJECT:** PHYSICAL PROPERTIES OF HIGH TEMPERATURE MATERIALS  
Part II. The Heat Capacity of Zirconium, Several  
Zirconium Hydrides, and Certain Cladding Materials from  
0° to 900°C; Relation to Other Thermodynamic Properties  
of the Zr-H System

**INVESTIGATOR:** Thomas B. Douglas  
Andrew C. Victor

**CONTRACT:** AF33(616)56-21

**CONTRACTOR:** National Bureau of Standards

**ABSTRACT:** Using accurate apparatus and a "drop" method, the heat content (enthalpy) of zirconium, five zirconium hydrides ( $N_H$  from 1.34 to 4.14), and Stainless Steel Type 316 were measured over the range from 0° to 900°C. Using the values for the stainless steel and those from the literature for molybdenum and niobium, the heat capacities of typical clad samples of the hydrides may be computed additively. Thermal hysteresis of the hydrides was investigated in several cases. Corrections were applied for the impurities in the samples measured, but the two sets of hydrides gave heat values believed to be somewhat inconsistent in the range 550° to 800°C through systematic differences in phase compositions. The heat content data were extensively correlated with certain published equilibrium data for the zirconium-hydrogen system in order to extend knowledge of the heats of hydriding, equilibrium hydrogen pressures, and limits of solid solubility to wider ranges of temperature and composition than those covered by direct measurements. The results are discussed critically, and several structural implications are pointed out.

WADC TR 57-375

October 1957

ASTIA Document No. AD 142029  
OTS Release

**SUBJECT:** A METHOD FOR DETERMINING NEUTRON FLUX SPECTRA FROM  
ACTIVATION MEASUREMENTS

**INVESTIGATOR:** Sven R. Hartmann, 1/Lt

**ABSTRACT:** A method for the determination of neutron spectra which approximates the neutron flux by a linear combination of the response functions (cross sections) of the detectors used in the spectral measurement is discussed. This linear combination is shown to approximate the neutron flux in the mean and to provide a calculated activation, for each detector, identical with that found in the experiment. Some practical cases are considered herein. A set of seven cross sections are used to approximate a fission, constant, and monoenergetic spectrum. A discussion of uncertainty propagation is given with illustrative examples for the

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fission and constant spectras. The integral neutron flux is also calculated and shown to be more adequately representable than the neutron flux.

WADC TR 57-381

ASTIA Document No. AD 142344

OTS Release

February 1958

SUBJECT: TABLES OF INTERPLANAR SPACINGS COMPUTED FOR THE CHARACTERISTIC RADIATIONS OF COPPER, MOLYBDENUM, IRON, CHROMIUM AND COBALT

INVESTIGATOR: H. J. Garrett  
R. E. Brocklehurst

ABSTRACT: Computations of interplanar spacings, "d" values, obtained from solutions of the Bragg equation  $n\lambda = 2d \sin \theta$ , are listed for the radiations normally used in X-ray diffraction analysis. All angular measurements are expressed as  $(2\theta)$  values. Interplanar spacings are calculated for the  $K_{\alpha}$ ,  $K_{\alpha 1}$ ,  $K_{\alpha 2}$  and  $K_{\beta}$  radiations of the elements copper, molybdenum, iron, chromium, and cobalt.

WADC TR 57-465

OTS Release

July 1957

SUBJECT: EFFECTS OF HIGH-ENERGY, HIGH-INTENSITY ELECTROMAGNETIC RADIATION ON ORGANIC LIQUIDS

INVESTIGATOR: E. M. Kinderman

CONTRACT: AF 33(616)3738

CONTRACTOR: Stanford Research Institute

ABSTRACT: Effective development of practical nuclear power required that materials be developed to withstand its attendant nuclear radiation. For example, among the materials which must withstand radiation in a nuclear-propelled aircraft are hydraulic fluids and lubricants. A knowledge of the effects of structure on the radiation stability of organic materials is of advantage in designing and producing such radiation-resistant hydraulic fluids and lubricants. This investigation attempts to provide such background information.

A series of organic esters, hydrocarbons, and miscellaneous compounds were subjected to radiation. A quantitative measurement of the amounts and kinds of radiation products produced was made. From the observed products and their amounts, some inferences were made as to the stability of these materials and the influence that their structure had on their stability.

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Radiolysis of organic liquids is influenced by structure. Molecules containing branch chain and benzyl groupings are more susceptible to radiolytic damage than are molecules containing only straight chain groupings. This greater damage may be explainable in terms of the greater inductive effects possible with the branch chain and benzyl groupings. Greater radiation stability is observed with higher molecular weight compounds. This effect may be explained in terms of the mechanisms of energy absorption and disintegration by the molecule.

Comparison of the radiolytic with mass spectrographic data shows some correlation between the modes of molecular disintegration induced in the two cases. The correlation is best when conditions of radiolysis are similar to those in the mass spectrograph, that is when radiolysis occurs in an essentially low pressure condition through activated species whose half-life is short and whose diffusion constants or reaction times are long.

Both electron and gamma radiation was used. The former received emphasis because of its speed and convenience in the test program. Comparison of the effects of the two types of radiation in six cases shows some quantitative differences in radiolytic yield. The products identified are the same, and the same structural effects are evident. The available data do not permit definite statements of equivalence between electron and gamma effects, although this is indicated.

WADC TR 57-487  
ASTIA Document No. AD 150954  
OTS Release

February 1958

SUBJECT: THERMAL PROPERTIES OF HIGH TEMPERATURE MATERIALS  
INVESTIGATOR: I. B. Fieldhouse J. I. Lang  
J. C. Hedge T. E. Waterman  
CONTRACT: AF 33(616)-3701  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: The objective of this program was the measurement of the high temperature thermal properties of materials. The materials investigated were Hastelloy B, Hastelloy C, Stellite 21, Stainless Steel type 17-7 P.H., Stainless Steel type 446, Silicon Carbide, 60-15 Cr (ASTM B83-46), and beryllium. The thermal conductivity, specific heat, and linear coefficient of thermal expansion were measured from 1000°F to 3000°F, or the melting point of the material, whichever was lower. All measurements were made in the Heat Transfer Laboratory of the Propulsion and Fluid Mechanics Research Department, Armour Research Foundation of Illinois Institute of Technology. Both the experimental measurements and the results of the conversion of these measurements to the desired physical properties are given.

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WADC TR 57-488  
ASTIA Document No. AD 142138

November 1957

SUBJECT: MEASUREMENT OF THE HEAT CAPACITY AND DENSITY OF LIQUID COPPER  
INVESTIGATOR: J. I. Lang  
CONTRACT: AF 33(616)-3795  
CONTRACTOR: Armour Research Foundation  
ABSTRACT: The density and specific heat of molten electrolytic tough-pitch copper meeting Federal Specification QQ-C-576 was determined for the temperature range 2000 to 4000°F.

WADC TR 57-498  
ASTIA Document No. AD 142157  
OTS Release

November 1957

SUBJECT: DESIGN AND USE OF A 23,000 CURIE COBALT-60 FACILITY  
INVESTIGATOR: Marvin C. Atkins, 1/Lt Donald R. Smith, 1/Lt  
Kurt Wolfsberg, 1/Lt  
William N. Lorentz, 1/Lt  
ABSTRACT: A two-chambered hot cell has been designed, built, and used. The hot cell is suitable for radioactive materials testing, hot chemistry, or handling of radiation sources. A 23,000 curie cobalt-60 source has been assembled and is being used for materials irradiations. The source in its normal form is 11.6 inch I. D. x 28 inches long. The activity of each of the 2450 slugs comprising the source was individually calibrated. Slugs of different activities were arranged to give the desired flux distribution inside the cylindrical source. Dose rates in and around the source have been measured with an ion chamber and glass dosimeters. An engineering approach to design of radiation sources has been formulated and tested with the 23,000 curie source. The report includes a complete description of the hot cell, design and assembly of the source, dosimetry methods, and mathematical design of cylindrical sources.

WADC TR 57-512  
ASTIA Document No. AD 151060  
OTS Release

March 1958

SUBJECT: STATE OF DEVELOPMENT OF THERMAL RADIOMETER  
INVESTIGATOR: Arnold Pfenninger Maurice Godet  
Harold E. Henry Conrad A. Wogrin  
CONTRACT: AF 33(616)-3628

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CONTRACTOR: Quantum, Incorporated  
ABSTRACT: The objective of the work covered by this report was the development and construction of a thermal radiometer.

This report describes the progress made to date on the design and manufacture of the components of the instrument and includes mathematical analyses of both the heat transfer and control system problems where these were necessary and feasible. It also includes a discussion of the specifications as originally outlined and revisions suggested on the basis of analytical and experimental work performed on this contract.

WADC TR 57-667

October 1957

SUBJECT: HIGH TEMPERATURE X-RAY DIFFRACTION CAMERA  
CONTRACT: AF 33(600)-32214  
CONTRACTOR: Herrick L. Johnston, Inc.  
ABSTRACT: A high temperature furnace was designed for simple attachment to a Norelco diffractometer. The furnace is constructed so as to permit X-ray diffraction studies of solid or powder specimens at temperatures up to 1800°C in vacuum or under pressures of 2 atmospheres or less. Scanning from  $2\theta$  of 20° to 122° is possible. Heating elements are made of .005 inch tantalum and temperature is measured by a Pt-(87% Pt, 13% Rh) thermocouple or by an optical pyrometer. Temperatures up to 1600°C are automatically controlled to within  $\pm 0.1\%$ . Automatic control is also possible from 1600° to 1800°C. Measurements of the lattice parameters of tantalum at various temperatures, including 1800°C, were in agreement with previously published values.

WADC TR 57-771  
ASTIA Document No. AD 151084  
OTS Release

March 1958

SUBJECT: APPROXIMATION OF ALL ORDERS OF THE EXPONENTIAL INTEGRAL  
INVESTIGATOR: Francis Clark  
CONTRACT: AF 33(616)-5187  
CONTRACTOR: Trg, Incorporated  
ABSTRACT: Approximations are sought for the exponential integral functions, i.e.

$$E_m(p) = p^{m-1} \int_p^\infty \frac{e^{-x} dx}{x^m}, \quad m > 0.$$

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The function  $E_m(p)$  is set equal to

$$\frac{e^{-p}}{p^m + q_m(p)}$$

and suitable algebraic approximations to the function  $q_m(p)$  are then found. Consideration is given to ways of estimating the error of the approximations.

WADC TR 58-42

January 1958

**SUBJECT:** PRODUCTION OF HOMOGENEOUS TITANIUM ALLOY STANDARDS  
**INVESTIGATOR:** Donald H. Turner  
**CONTRACT:** AF 33(616)-3098  
**CONTRACTOR:** Armour Research Foundation  
**ABSTRACT:** Three compositional variations of each of nine commercial titanium alloys were prepared by a triple arc melting procedure for use as standards for spectrographic and X-ray fluorescent analysis of titanium alloys. Analysis of each triple-melted, as-cast ingot has been carried out by three laboratories.

The material was supplied to the National Bureau of Standards as 1 1/4 inch diameter, centerless-ground bar. In general, there is good agreement between analyses and nominal composition. Where there are differences, trends are similar.

## METALS, GENERAL

WADC TR 56-330 Pt III  
ASTIA Document No. AD 130865  
OTS Release

June 1957

**SUBJECT:** THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS  
Part III. The Plastic Bending of Tapered Members  
**INVESTIGATOR:** Montgomerie C. Steele  
Hassan A. Hassan  
**CONTRACT:** AF 33(616)-2753  
**CONTRACTOR:** University of Illinois  
**ABSTRACT:** This report contains an exploratory investigation of the plastic bending of tapered members. Two analytical approaches are employed. The first considers a rigorous treatment by the classical theories of elasticity and plasticity while the second resorts to the more tractable

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mechanics of materials methods. A limited amount of experimental work is presented in support of theory.

WADC TR 56-330 Pt IV  
ASTIA Document No. AD 130765  
OTS Release

May 1957

**SUBJECT:** THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS  
Part IV. Eccentrically-Loaded Columns Having Angle- and T-Sections

**INVESTIGATOR:** M. E. Clark  
O. M. Sidebottom

**CONTRACT:** AF 33(616)-2753

**CONTRACTOR:** University of Illinois

**ABSTRACT:** This paper presents the results of an analytical and experimental investigation for the determination of the load necessary to produce any given depth of yielding in angle- and T-section columns subjected to eccentric loads. This load is found analytically from the intersection of a constant depth of yielding interaction curve and the moment-load curve (see Part I of this Report). The collapse load of the column can be taken as the maximum load given by the moment-load curve.

In the experimental investigation, tests were made on eccentrically-loaded angle- and T-section columns made from type 304 stainless steel. The angle-section columns were so oriented that some buckled about the minimum axis of inertia and some about the maximum axis of inertia. The effects of variations in initial eccentricity and slenderness ratio on the collapse load were investigated. In all cases the experimental and theoretical moment-load curves were of the same general shape; the experimental collapse load being less than the theoretical by 2 to 16 percent. Since the theory is based on assumptions which make it conservative, the fact that the experiments showed it to be unconservative was attributed to the time-sensitive behavior of the stainless steel material. The stress-strain properties obtained from tension and compression tests did not represent the stress-strain behavior of the material in the column. When these stress-strain properties were adjusted by a 28 percent reduction in the modulus of elasticity, the experimental collapse load varied from 9 percent greater to 11 percent less than the theoretical value with most of the values comparing very favorably.

WADC TR 56-330 Pt V  
ASTIA Document No. AD 130768  
OTS Release

May 1957

**SUBJECT:** THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS

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## Part V. Inelastic Behavior of Aluminum Alloy I-Beams with Elliptical Web Section Cutouts

INVESTIGATOR: Will J. Worley  
CONTRACT: AF 33(616)-2753  
CONTRACTOR: University of Illinois  
ABSTRACT: This report represents an extension of WADC Technical Report 56-330, Part II, April, 1956.

This extension was undertaken to investigate the plastic bending behavior of aluminum alloy I-beams with elliptical web section cutouts. As in the above report, the mechanism method of analysis employing the Upper Bound Theorem was used in predicting the ultimate loads of the various beams. This procedure could not be applied in all cases since several of the beams failed by buckling failures in the web sections. The I-beams were loaded in pure bending using quarter point loading and in combined shear and bending using center point loading. The span lengths were varied for a given depth of I-beam in order to investigate the effects of shear on the failure mechanism. Where the mechanism analysis applies, it permits the prediction of loads which are in good agreement with the experimental results.

WADC TR 56-330 Pt VI  
ASTIA Document No. AD 118300  
OTS Release

May 1957

SUBJECT: THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS  
Part VI. A Digital Computer Analysis of Bending Moment-Axial Load Interaction Curves

INVESTIGATOR: Y. Maeda O. M. Sidebottom  
P. Van Lierde M. E. Clark  
CONTRACT: AF 33(616)-2753  
CONTRACTOR: University of Illinois  
ABSTRACT: This paper presents the results of a study undertaken to determine the feasibility of using the digital computer to make the time-consuming calculations necessary to construct moment-load interaction curves. A strip method of numerical integration was set up to solve the equations of equilibrium for the members, this method allowed a generalization of the procedure to all shapes of cross-section.

In setting up the problem for the digital computer, it was not convenient to work with the actual stress distributions. Instead, it was found that the actual stress distribution could be represented by three linear stress distributions having the same stress gradient. Load- and moment-functions were computed from these stress distributions and the actual load and moment in the section were determined from these load- and moment-functions.

Load- and moment-functions for some 86 different conditions for T-, angle-, and channel-section members were determined using the digital computer. The great number of additional computations

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required to construct the interaction curves once the load- and moment- functions are available renders the digital computer method reported herein of questionable use. Further study of the problem could possibly produce methods by which these additional computations could be made by the computer and thereby make the method more efficient and useful.

WADC TR 56-330 Pt VII  
ASTIA Document No. AD 142217  
OTS Release

December 1957

SUBJECT: THE EFFECTS OF INELASTIC ACTION ON THE RESISTANCE TO VARIOUS TYPES OF LOADS OF DUCTILE MEMBERS MADE FROM VARIOUS CLASSES OF METALS  
Part VII. Inelastic Behavior of Aluminum Alloy I-Beams with Elliptic-Type Web Section Cutouts

INVESTIGATOR: Will J. Worley  
Fred D. Breuer

CONTRACT: AF 33(616)-2753

CONTRACTOR: University of Illinois and Convair

ABSTRACT: This report represents a continuation of WADC Technical Report 56-330, Part II, April, 1957 and Part V, May, 1957.

This continuation was undertaken to investigate the fully plastic bending behavior of aluminum alloy I-beams with elliptic-type web section cutouts. As in the two earlier reports, the mechanism method of analysis employing the Upper Bound Theorem was used in predicting the ultimate loads of the various beams. The I-beams were loaded as simple beams with center loading. The mathematical relations were developed to enable solution by means of an electronic digital computing machine, the Illiac. The results obtained on the Illiac indicated that a diamond shape web section cutout was the most favorable type for resisting fully plastic bending. Tests were conducted which substantiated the results obtained on the Illiac.

WADC TR 57-131  
ASTIA Document No. AD 130910  
OTS Release

July 1957

SUBJECT: THE SAFETY OF AIRCRAFT STRUCTURES

INVESTIGATOR: A. M. Freudenthal

CONTRACT: AF 33(616)-2729

ABSTRACT: The concept of structural safety of airframes is analyzed on the basis of its relation to the probability of structural failure, with a view of establishing procedures of quantitative evaluation of safety factors for a predetermined "acceptable" risk of failure.

The difference in the approach to the concept of safety for ultimate strength and for fatigue is discussed, considering recent developments in fatigue research, particularly the results of fatigue tests under random loading, and methods of safety analysis for both conditions are proposed.

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In this analysis the "limit load" or "limit load factor" is a basic concept. It should, however, be noted that this concept is not identical with the conventional structural design criterion of the same name. The difference is fundamental: while in conventional design the "limit load" is a derivative concept obtained simply by dividing the "ultimate load" by an arbitrary "safety" factor, usually 1.5, the concept as used here is the primary load criterion defining, independently of the ultimate load, a limiting condition of service by the aid of which fatigue design and design for limit load can be correlated.

Since it is a purpose of the present analysis to discuss and develop procedures for the rational evaluation of safety factors, expediently defined in terms of ratios of the ultimate to the limit load factor, it is obvious that both load factors have to be independently derived from operational criteria. Thus, while the meaning of the concept of "ultimate load" used in this report does not differ significantly from its meaning in conventional design, the "limit load" concept is significantly different.

WADC TR 57-311  
ASTIA Document No. AD 131098  
OTS Release

September 1957

SUBJECT: EFFECT OF LOADING RATE ON THE STRENGTH OF SINGLE AND MULTIPLE RIVETED JOINTS  
INVESTIGATOR: Richard F. Klinger  
ABSTRACT: Slow and rapid loading tensile shear tests were conducted at room temperature on single and multiple rivet lap and butt joints. The joints were manufactured with a standard pneumatic type rivet squeezer using 2117-T3 aluminum alloy rivets, 1/8 inch diameter, and Alclad 2024-T3 aluminum alloy sheet, 0.064" thick. The ultimate strengths of the joints were determined with times to the ultimate load of 0.02 and 60 seconds. All failures were by rivet shear.

The results indicate that the effect of rapid loading is to increase the strength of single riveted joints, that this effect is diminished as the number of rivets in the joint increases and that when the number of rivets in the joint is four and above the effect of rapid loading is to decrease the joint strength slightly.

WADC TR 57-695  
ASTIA Document No. AD 151165  
OTS Release

April 1958

SUBJECT: DETERMINATION OF THE MECHANICAL PROPERTIES OF A HIGH PURITY LEAD AND A 0.058% COPPER-LEAD ALLOY  
INVESTIGATOR: Thomas E. Tietz  
CONTRACT: AF 33(616)-3785  
CONTRACTOR: Stanford Research Institute  
ABSTRACT: The mechanical properties of a high purity lead and a 0.058% copperlead alloy were determined at test temperatures of 100, 175, 250, and 325°F.

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Tensile properties evaluated included the ultimate strength, elongation, modulus of elasticity, proportional limit, and yield strength. Compression properties evaluated were the modulus of elasticity, proportional limit, and yield strength. Ultimate shear strength and the bearing yield strength and ultimate bearing strength were determined. Stress-creep time curves were obtained for total strain values of 0.2, 0.5, 1.0, and 2.0%, for creep times of from 1 hour to 500 hours.

The data obtained are summarized in graphical and tabular form, in the Experimental Results section of this report.

## METALS, FERROUS

July 1957

WADC TR 56-205  
ASTIA Document No. AD 110474  
OTS Release

SUBJECT: FATIGUE INVESTIGATION ON HIGH STRENGTH STEEL  
INVESTIGATOR: J. K. Childs  
M. M. Lemcoe  
CONTRACT: AF 33(616)-2513  
CONTRACTOR: Southwest Research Institute  
ABSTRACT: A single heat of aircraft quality SAE 4340 steel, heat-treated to 190,000, 260,000 and 300,000 psi ultimate tensile strength, was tested by the increasing load method (similar to Prot's method) in axial loading to determine the variation in fatigue properties under each of the three mean stress conditions, zero, 60,000 and 90,000 psi. Conventional S-N curves were determined by axial load tests at 190,000 psi ultimate tensile strength, under zero mean stress and 90,000 psi mean stress, to provide factors for adjusting the Prot-type failure stresses to standard mean endurance limit values. In addition, Prot rotating beam tests were performed at 190,000 psi ultimate tensile strength.

The endurance limit under completely reversed axial stress increased with an increase in ultimate strength from 190,000 psi to 260,000 psi, but appeared to decrease when the same material was heat-treated to 300,000 psi. Under high mean tensile stress the 300,000 psi material may have a fatigue strength superior to that of the 260,000 psi material.

May 1958

WADC TR 56-555  
ASTIA Document No. AD 130755  
OTS Release

SUBJECT: DEVELOPMENT OF NEW MECHANISMS AND TECHNIQUES FOR OBTAINING STEELS IN THE ULTRAHIGH STRENGTH RANGE

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INVESTIGATOR: Damian Gullotti  
Erwin Eichen  
Joseph W. Spretnak  
CONTRACT: AF 33(616)-2879  
CONTRACTOR: Ohio State University Research Foundation  
ABSTRACT: Two possible methods of obtaining steels in the high strength range were investigated, namely, the superimposition of plastic deformation on the transformation of austenite to martensite, and the production of high nitrogen-low carbon (0.25% maximum) martensite. In the deformation of austenites prior to transformation, increases in tensile strength up to 10% and in elastic limit up to 30% were obtained. In steels of base composition C-0.20, Si-1.60, Mn 0.75, Cr-1.40, increasing the nitrogen content from 0.004 to 0.009% increased the 0.2% offset yield strength about 6% and the tensile strength about 3.5%. The ductility was essentially unaffected by this increase in nitrogen content.

WADC TR 56-598

November 1956

SUBJECT: AN INVESTIGATION OF ABSORBED HYDROGEN IN ULTRA-HIGH-STRENGTH STEEL  
INVESTIGATOR: Charles R. Simcoe  
Arthur R. Elsea  
George K. Manning  
CONTRACT: AF 33(616)-3202  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: A study was made of the relationship between current density and the permeation of hydrogen through a thin, steel membrane which was the cathode in an electrolytic cell. The established relationship was used to study the effect of the variation in hydrogen-absorption rate on the time for rupture to occur under sustained stress. It was found that the relationship between the rupture time and the rate at which hydrogen was absorbed by the steel could be expressed by the equation  $T_f = KP^{-2/3}$  where  $T_f$  was the rupture time, K was constant for each applied stress, and P was the permeation rate.

A study was made of the incubation times for crack nucleation and of crack growth rates under various experimental conditions. Incubation times increased with decreasing applied stress. Crack growth rates also increased with decreasing applied stress, whereas, it was expected that they would decrease.

The effect of other factors, such as austenitizing temperature, prestrain, precharging, composition of the steel, and torsion loading, upon the rupture time also was studied.

WADC TR 57-501

December 1957

ASTIA Document No. AD 142265

SUBJECT: MECHANICAL PROPERTIES OF SOME SILICON-MODIFIED HIGH STRENGTH STEELS

WADC TR 53-373 Sup 5

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INVESTIGATOR: Carl J. Altstetter  
Morris Cohen  
B. L. Averbach  
CONTRACT: AF 33(616)-2012  
CONTRACTOR: Massachusetts Institute of Technology  
ABSTRACT: The mechanical properties of 43XX steels, containing 0.15%, 0.25% and 0.37% carbon, with and without 3% silicon, were determined as a function of tempering temperature. 4337 steels with 1.5% silicon and with variations in nickel, molybdenum and boron were also tested. The chief advantage of the silicon addition appears at high strength levels. Silicon retards the third stage of tempering, and an ultimate strength of 300,000 psi is developed in 4337 (3.0 Si) even after tempering as high as 600°F. The ductility and impact properties are not adversely affected at this strength level by the silicon. Silicon was also found to increase the as-quenched hardness, especially in the two lower carbon grades.

WADC TR 58-108 Pt I  
ASTIA Document No. 151075  
OTS Release

March 1958

SUBJECT: THE EFFECT OF CADMIUM PLATING ON SAE 4340 STEEL IN THE PRESENCE OF STRESS CONCENTRATIONS AT ELEVATED TEMPERATURES  
INVESTIGATOR: E. M. Kennedy, Jr., Major  
ABSTRACT: An investigation was carried out to determine the effect of cadmium plating on the tensile and stress rupture properties of high strength steel at elevated temperatures. Tests were conducted on SAE 4340 steel, and properties of this material were investigated in the annealed and heat treated conditions. The annealed material was investigated at 600°F and 611°F, effects on properties of the heat treated material were investigated at 500°F, 600°F and 611°F. The properties of cadmium plated steel in both conditions, annealed and heat treated, were compared with the properties of unplated steel subjected to the same test conditions.

## METALS, HIGH TEMPERATURE

WADC TR 55-243  
ASTIA Document No. AD 131071

September 1957

SUBJECT: DEVELOPMENT OF ALLOYS HAVING GOOD HIGH TEMPERATURE PROPERTIES THROUGH POWDER METALLURGY TECHNIQUES  
INVESTIGATOR: Dr. R. Kieffer  
Dr. F. Benesovsky  
CONTRACT: AF 61 (514)-741-C

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CONTRACTOR: Metallwerk Plansee  
ABSTRACT: Pure titanium carbide and titanium carbide with molybdenum carbide, vanadium carbide and chromium carbide have been alloyed with refractory binding agents such as iron-aluminum alloys with a content of 8 to 14 % of aluminum as well as Fe-Al-Cr, Fe, Cr-Al and Fe-Al-Mo. The carbide phase has been varied from 5 to 35%.

The most promising alloys found out among the 12 series of alloys consisted of a binding agent of iron-aluminum with 12 or 14% of aluminum and of mixed crystals of titanium carbide - chromium carbide 90/10.

All alloys examined can be shaped to a certain extent at high temperatures.

WADC TR 57-242  
ASTIA Document No. AD 142086  
OTS Release

November 1957

SUBJECT: INVESTIGATION OF Fe-Mn-Cr-N-C SYSTEM FOR HEAT RESISTANCE AND OXIDATION RESISTANCE BETWEEN 1200°F and 2000°F.  
INVESTIGATOR: Chi-Mei Hsiao  
Edward J. Dulis  
CONTRACT: AF 33(616)-3318  
CONTRACTOR: Crucible Steel Company of America  
ABSTRACT: A comprehensive study of wrought Cr-Mn-C-N steels was conducted with the objectives, (1) of developing outstanding elevated-temperature steels that contain a minimum amount of strategic alloying elements, and (2) of advancing the existing knowledge of this new type of steel. Based on the results of the present investigation phase boundaries of the base compositions of stable austenitic Cr-Mn-C-N steels have been established, and the effects of these elements and of V, W, Mo, and Cb on the microstructures, and the room- and elevated-temperature properties of the steels have been evaluated. A nomograph was prepared to facilitate the designing of the base compositions of these steels, and the estimating of their room- and elevated-temperature properties.

These steels are not as resistant to oxidation as are the Cr-Ni steels at comparable Cr levels. However, Cr-Mn-C-N base steels that have properly balanced compositions possess an excellent combination of strength and ductility at both room- and elevated-temperatures. For example a steel containing .6C, 12 Mn, 24 Cr, .7 N, .4V, 2 W, and .9 Cb, had a 185,000 psi tensile strength, a 120,000 psi yield strength, a 48% elongation, and a 40% reduction of area. Also, its rupture strength was superior to those of 16 Cr - 25 Ni - 6 Mo, N155, 19-9DL, and A-286 in the temperature range of 1200 to 1400 F.

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WADC TR 57-298 Pt I  
ASTIA Document No. AD 130823

May 1957

**SUBJECT:** IRON-ALUMINUM ALLOY SYSTEMS  
Part I - Fundamental Studies and Alloy Development

**INVESTIGATOR:** F. X. Kayser

**CONTRACT:** AF 33 (600)-32448

**CONTRACTOR:** Ford Motor Company

**ABSTRACT:** An extensive program is being carried on to study the mechanical and physical behavior of recrystallized iron-aluminum alloys containing up to 18.5 weight percent aluminum. Data are presented illustrating the effect of aluminum on the stress-rupture and tensile properties. It is concluded that the recrystallized binary alloys are too low in hot strength to consider for use in moderate stress application at temperatures in excess of 1000°F. Special attention has been given to the development of higher hot strength alloys. The effect of titanium on hot strength is demonstrated.

The effects observed of order-disorder transformations on mechanical properties are of fundamental interest. Some interesting and unique magnetic phenomenon have been discovered in the binary alloys among which is a continuous change from ferro-magnetic to anti-ferromagnetic behavior.

WADC TR 57-298 Pt II  
ASTIA Document No. AD 130824

May 1957

**SUBJECT:** IRON-ALUMINUM ALLOY SYSTEMS  
Part II - Some Investigations in Air-Melting and Application of Iron-Aluminum Alloys

**INVESTIGATOR:** Joseph J. Clark  
William J. Zager

**CONTRACT:** AF 33(600)-32448

**CONTRACTOR:** Ford Motor Company

**ABSTRACT:** During the period April 1956 to April 1957 the Process Development Department of the Manufacturing Engineering Office of Ford Motor Company air-melted a series of heats of iron-aluminum alloys in the 6 to 10% aluminum range. The melting and decoxidation of these heats, performed in indirect-arc type furnaces, are described. Mechanical properties obtained on bar stock rolled from ingots cast from a number of these heats are compared with established properties of vacuum-melted alloys of similar composition. Results of some exploratory heats involving changes in basic melting stock, as well as some with small additions of third elements, are presented.

Initial attempts to produce sound castings in sand molds are described.

Performance of certain iron-aluminum alloy samples and fabricated articles in service is described.

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WADC TR 57-298 Pt III  
ASTIA Document No. AD 130825

May 1957

SUBJECT: IRON-ALUMINUM ALLOY SYSTEMS  
Part 3 - Welding of Iron-Aluminum Alloys  
INVESTIGATOR: R. Burthwick  
S. Goodman  
CONTRACT: AF 33(600)-32448  
CONTRACTOR: Ford Motor Company  
ABSTRACT: Investigations were conducted to develop fusion welding and resistance welding procedures for three (3) heats of iron-aluminum alloy. Successful fusion welding procedures were developed using the tungsten inert gas shielded arc process. Variables of power type, current, voltage, shielding, travel speed, cleaning, preheat and postheat were investigated. No definite resistance welding schedule was established, although, successful welds were realized with several schedules. Three fabrications were made using the fusion welding process in order to evaluate weld and material performance under service condition. The service evaluations had not been completed at the time of writing this report.

WADC TR 57-309

August 1957

SUBJECT: JOINING OF MOLYBDENUM  
INVESTIGATOR: W. N. Platte  
CONTRACT: AF 33(616)-3524  
CONTRACTOR: Westinghouse Research Laboratories  
ABSTRACT: An examination of available metallurgical data on molybdenum base alloys indicated that additions of aluminum, titanium, carbon, and zirconium should provide good welding properties. A group of the alloys selected was welded in a chamber containing an atmosphere of argon. Mechanical and metallurgical tests showed that aluminum additions in the range of 0.18 to 0.22% gave welds that could be deflected 120° at room temperature. However, all the alloys containing aluminum showed fine porosity. Titanium additions of 0.5% produce welds which can be deflected 120° at temperatures between 80 and 120°F depending upon the amount of carbon in the alloy. Welds in 0.5% Ti with 0.05% C produced the best results. No porosity was observed in titanium alloys. Carbon in molybdenum-carbon alloys was shown to improve the weld metal ductility by reducing the oxygen content of the base material and by a protective deoxidization action during welding. Molybdenum with 0.06% carbon showed the best bend ductility within the carbon range examined.

The effect of welding speed on the grain size of the weld metal was examined. While it is theoretically possible to reduce the weld metal grain size by increasing the welding speed, experimental difficulties were encountered due to retraction of the weld metal at speeds of 2 cm/sec. Chilling the weld failed to overcome these difficulties.

Post-welding residual stresses were measured and it was found that these stresses could be eliminated by a vacuum heat treatment at

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1832°F.. The residual stresses found in the welds prior to heat treatment are sufficiently large to produce fracture without additional applied stress. The post-welding stress relief was accomplished before the weld had cooled to the transition range. Crater cracking was effectively prevented by this heat treatment.

WADC TR 57-343  
ASTIA Document No. AD 142117  
OTS Release

November 1957

**SUBJECT:** A STUDY OF THE METALLURGICAL PROPERTIES THAT ARE NECESSARY FOR SATISFACTORY BEARING PERFORMANCE AND THE DEVELOPMENT OF IMPROVED BEARING ALLOYS FOR SERVICE UP TO 1000 F

**INVESTIGATOR:** Gopal K. Bhat  
Alvin E. Nehrenberg

**CONTRACT:** AF 33(616)-3318

**CONTRACTOR:** Crucible Steel Company of America

**ABSTRACT:** The use of bearings made from hot work steels and other tool steels in experimental engines has resulted in a few premature engine failures. Unfortunately, very little has been known about the elevated temperature properties such as hot hardness, compressive yield strength, resistance to softening and structural and dimensional stability of these hot work and other tool steels. This report describes the work done to obtain these material properties for 29 steels ranging in type from SAE 52100, its modifications, to hot work and other tool steels. An analysis of the data obtained shows that Halmo, VSM, M50, M10, T1, M2, M1 and two experimental compositions one, Steel B, containing 0.70 carbon, 4.20 chromium, 0.60 vanadium, and 5.30 molybdenum, and the other, Steel G, containing 1.31 carbon, 4.07 chromium, 4.13 vanadium, 5.75 tungsten, and 4.87 molybdenum, are suitable for elevated temperature aircraft bearing application. From a point of view of temperature range of application these steels have been classified as follows:

Room Temperature up to 700 F	Halmo 1
Room Temperature up to 800 F	VSM, M50, M10 and Steel B
Room Temperature up to 900 F	T1, M2, M1 and Steel G

None of the steels investigated appeared suitable for application at 1000 F.

WADC TR 57-426  
ASTIA Document No. AD 142237  
OTS Release

December 1957

**SUBJECT:** INVESTIGATION OF THE EFFECTS OF INCONGRUOUS ELEMENTS

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## ELEMENTS AND THE INTERACTION EFFECTS OF THESE ELEMENTS ON HIGH TEMPERATURE STRENGTH OF FE-CO- NI-CR ALLOYS

INVESTIGATOR: J. H. Sye  
T. L. Robertshaw  
F. M. Richmond

CONTRACT: AF 33(616)-2777

CONTRACTOR: Universal-Cyclops Steel Corporation

ABSTRACT: An investigation was made of the effect of five combinations of incongruous elements on the high temperature properties of vacuum melted alloys with a base composition of 60 atomic % Ni, 20 atomic % Cr, 10 atomic % Fe, 10 atomic % Co. Balanced experimental designs were used for each group.

In the group of alloys containing additions of Al, Ti and W to the base, a cast alloy had a stress-rupture life of 133.9 hours at 1800F and 15,000 psi. A forged alloy of this series obtained a life of 149.1 hours at 1750F and 15,000 psi.

The value and limitations of balanced experimental designs are discussed.

A metallographic study of cast and wrought alloys is appended.

WADC TR 57-535  
ASTIA Document No. AD 150971  
OTS Release

February 1958

SUBJECT: A STUDY OF THE POSSIBILITY OF REINFORCING HIGH-  
TEMPERATURE ALLOYS BY ADDITION OF REFRACTORY  
POWDERS

INVESTIGATOR: John D. Burney

CONTRACT: AF 33(616)-2959

CONTRACTOR: P. H. Mallory & Co., Inc.

ABSTRACT: This investigation was concerned with attempting to reinforce 80 Ni-20 Cr alloy by the addition of such refractory oxides as  $TiO_2$  and  $Al_2O_3$ .

Several powder metallurgical fabrication techniques were investigated such as (1) pressing and sintering, (2) pressing and sintering followed by various working procedures, (3) internal oxidation and (4) liquid phase sintering.

In the press-sinter method of fabrication, the best stress-rupture characteristics at 1600°F were obtained with additions of 3.0 - 4.5%  $TiO_2$ . An addition of 4.5%  $TiO_2$  resulted in a rupture time

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of 29.6 hours as compared to a rupture time of 1.0 hour obtained on bars containing no  $TiO_2$ .

Various hot and cold working procedures did not result in any improved stress-rupture properties.

The liquid phase sintering technique produced the best stress-rupture properties. Stress-rupture tests at 1800°F showed a stress of 6200 psi for an 80 Ni-20 Cr + 1.0%  $TiO_2$  + C material which was more than three fold better than wrought Nichrome V. Stress-rupture tests on the same materials at 1975°F showed the liquid phase material to have twice the strength of Nichrome V. The impact strengths varied from 10 to more than 50 inch pounds. Room temperature tensile strengths which were fairly consistent, averaged about 75,000 psi. The thermal shock properties were excellent and the oxidation resistance, although not quite as high as Nichrome V, was considered good.

WADC TR 57-649 Pt I  
ASTIA Document No. 142284  
OTS Release

January 1958

**SUBJECT:** DETERMINATION OF THE MECHANICAL PROPERTIES OF AIRCRAFT-STRUCTURAL MATERIALS AT VERY HIGH TEMPERATURES AFTER RAPID HEATING

**INVESTIGATOR:** James B. Preston  
William P. Roe  
J. Robert Kattus

**CONTRACT:** AF 33(616)-3494

**CONTRACTOR:** Southern Research Institute

**ABSTRACT:** Structural components in high-speed aircraft and in missiles must function for short periods of time at high temperatures and at high stresses. The heating time and loading time in conventional tests for design data usually exceed those that apply in high-speed aircraft and in missiles. A study was made, therefore, of the mechanical properties --- tensile, creep, fracture, compression, shear, and bending --- of several structural materials under conditions of moderate to rapid rates of heating and of loading

The materials involved in this investigation included electrolytic-tough-pitch copper, oxygen-free high-conductivity copper, A-nickel, ingot iron, molybdenum, tantalum, Type GEH graphite, and composite OFHC copper plus 316 stainless steel sheet. The testing temperatures ranged from room temperature to the melting points of the metals and to 5750°F for the graphite. The total heating, holding, and loading times to failure ranged from about three seconds to about 30 minutes. Many of the tests were carried out both in air and in inert atmospheres.

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It was found that the strength of the test metals decreased continuously with increasing temperatures. The graphite, however, increased in strength up to 4500°F and then decreased in strength with further increases in temperature. With constant load and constant temperature and with transient load or transient temperature, the inherent load-carrying capacity of the metals increased with decreasing times under load. The inherent strength of the graphite was not significantly affected by variations in time under load.

The mechanical properties of the copper and of the nickel test materials were not significantly affected by exposure to air at temperatures up to their melting points within the time limitations of the various tests. Because of oxidation, the apparent strength of the iron, molybdenum, tantalum, and graphite decreased with increasing exposure times to air at elevated temperatures.

WADC TR 57-678

March 1958

ASTIA Document No. AD 151035

OTS Release

SUBJECT: STUDIES AND COMPARISON OF THE PROPERTIES OF HIGH TEMPERATURE ALLOYS MELTED AND PRECISION CAST BOTH IN AIR AND IN VACUUM

INVESTIGATOR: Milo J. Stutzman

CONTRACT: AF 33(616)-3468

CONTRACTOR: Westinghouse Electric Corporation

ABSTRACT: The objective of the research program for USAF Contract No. AF 33(616)-3468 was the investigation of the potentialities for improvement of the mechanical properties at elevated temperature of heat-resisting alloys by varied practices of melting and investment casting. Two cobalt and two nickel base alloys were prepared under the following casting conditions:

1. Stellite 31 was melted and investment cast in air and in vacuum, with an argon atmosphere for pouring.
2. He 1049 was melted and investment cast in air and in vacuum, with an argon atmosphere for pouring.
3. Utimet 500 was melted and investment cast in vacuum with and without additions of zirconium and boron.
4. Guy Alloy was melted and investment cast in vacuum and in argon.

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The effects of melting and casting procedures upon the gas (hydrogen, oxygen, oxygen and nitrogen) contents, the tensile properties at room and elevated temperatures, the stress-rupture properties in the range of 1350 to 1800 F, and the oxidation resistance to air at constant temperature of 2000 F and intermittent exposure to air at room temperature and 2000 F were studied.

The data obtained on this program failed to support general conclusions applicable to the four alloys under investigation. Tests of specimens of one alloy failed to substantiate data obtained from other tests for related properties. The data must be evaluated for each alloy with close correlation to service conditions for design applications.

WADC TR 57-717  
ASTIA Document No. AD 142258  
OTS Release

December 1957

SUBJECT: INTERMEDIATE PHASES IN THE IRON-TUNGSTEN AND COBALT-TUNGSTEN BINARY SYSTEMS  
INVESTIGATOR: Edward C. Van Reuth  
ABSTRACT: Fifty-three alloys have been examined in the Fe-W and Co-W alloy systems in an attempt to verify the finding of sigma phases in these systems as reported by Goldschmidt. Selected regions of both binary equilibrium diagrams were investigated by examining alloys metallographically and by X-ray diffraction techniques. The results of this investigation indicate that neither of these systems contains a sigma ( $\sigma$ ) phase. However, they do contain an xi ( $\xi$ ) phase ( $M_3W_2$ ), originally labeled ( $\chi$ ) by Arnfelt and Westgren, and Magneli and Westgren ( $M_7W_6$ ).

It was also found that some portions of the existing equilibrium diagrams should be changed.

WADC TR 58-28  
ASTIA Document No. AD 151161  
OTS Release

April 1958

SUBJECT: THE STRENGTHENING OF AUSTENITIC SOLID SOLUTIONS  
INVESTIGATOR: F. Eberle E. J. Rozic  
J. H. Hoke W. E. Leyda  
CONTRACT: AF 33(616)-2413 and AF 33(616)-5208  
CONTRACTOR: The Babcock & Wilcox Company Research Center  
ABSTRACT: Among vacuum-melted wrought carbon-free alloys of Fe-Cr-Ni, Fe-Cr-Co, and Fe-Cr-Ni-Co, the compositions 10Fe-20Cr-70Ni and 20 Cr - 80 Co have shown highest 100-hour rupture strength at 1800°F. Strengthening susceptibility studies with these two base compositions by

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solid solution hardening, complex carbide hardening, and intermetallic compound hardening revealed that the nickel base responds most effectively to intermetallic compound hardening with Ti and Al, less so to complex carbide hardening, and not at all to solid solution hardening. The cobalt base responds to all three methods of strengthening; however, it does not attain as high a level of rupture strength at 1800°F as the nickel-base alloys.

## METALS, NONFERROUS ALUMINUM

WADC TR 56-585 Pt I  
ASTIA Document No. AD 142007  
OTS Release

September 1957

SUBJECT: EFFECTS OF TEMPERATURE-TIME-STRESS HISTORIES ON THE MECHANICAL PROPERTIES OF AIRCRAFT STRUCTURAL METALLIC MATERIALS  
Part I. Temperature-Time Studies for 2024-T3 and 7075-T6 Alclad Sheet

INVESTIGATOR: Robert E. Fortney  
Charles H. Avery

CONTRACT: AF 33(616)-3028

CONTRACTOR: Northrop Aircraft, Inc.

ABSTRACT: In order to establish realistic design criteria applicable to aerodynamically heated materials and their complex combinations of temperature, time and stress exposure and inspection criteria for materials after exposure to complex service conditions, the tensile properties of 2024-T3 alclad and 7075-T6 alclad sheet were determined at room temperature, 200, 300 and 400°F after single and sequential multiple exposure in the range 250 through 600°F. In addition, the Rockwell hardness properties at room temperature after the above exposure conditions were determined to provide a basis for inspection of aircraft after service exposure to aerodynamic or engine heating.

Five tensile properties were determined for each exposure and test condition. Three of these, the proportional limit, modulus of elasticity, and percent elongation were tabulated and graphed in a non-dimensional form to generalize the data with respect to test material variability. Since the yield and ultimate strengths determine the load carrying ability, these tensile properties were analyzed carefully and generalizations with respect to exposure temperature and time and testing temperature were accomplished. Statistical calculations were made to

determine the accuracy of the various analyses. The conclusion was reached that the yield and ultimate strength analysis is adequate for establishing design criteria in the range room temperature through 400°F, after complex exposures to times from 1.0 to 1000 hours at temperatures from 250 to 600°F.

Material, equipment, specimens and procedures are described in detail. Test results are presented in the form of tables and curves to illustrate the effect of the exposure and test conditions on the materials under investigation and the effect of normalization analyses on the generalization of the data.

## METALS, NONFERROUS MAGNESIUM

WADC TR 56-415  
ASTIA Document No. AD 131042  
OTS Release

September 1957

SUBJECT: DEVELOPMENT OF ZM41 MAGNESIUM SHEET ALLOY  
INVESTIGATOR: H. A. Johnson  
R. D. Masteller, 1/Lt

ABSTRACT: A very promising magnesium alloy system was developed which appears superior to AZ31 sheet alloy. This alloy has been designated as ZM 41 and has a nominal composition of 4 Zinc + 1.0 Manganese + 0.7 Mischmetal balance Magnesium. Some of the features of ZM41 alloy compared to AZ31 are higher strength, more uniform longitudinal and transverse tensile properties, and a much greater tolerance for iron without the corrosion resistance of the alloy being adversely affected.

WADC TR 56-478  
ASTIA Document No. AD 142008

September 1957

SUBJECT: LITERATURE SURVEY OF THE CORROSION OF MAGNESIUM AND  
MAGNESIUM ALLOYS  
INVESTIGATOR: Henri S. Sack  
Bryon P. Roe  
CONTRACT: AF 33(616)-3032  
CONTRACTOR: Cornell University

ABSTRACT: This report represents a literature survey of the corrosion of magnesium and magnesium alloys, and emphasizes papers published since 1940. In view of the complexity of the phenomena and controversial or insufficiently documented experimental results only very few generalizations concerning the corrosion process can be made.

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WADC TR 57-194 Pt I  
ASTIA Document No. AD 151016

March 1958

SUBJECT: INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES  
Part I - Shop and Service Characteristics of Several Promising Wrought Magnesium Alloys

INVESTIGATOR: F. W. Hyslop  
G. S. Foerster

CONTRACT: AF 33(616)-2337

CONTRACTOR: The Dow Chemical Company

ABSTRACT: The shop and service characteristics of several promising wrought magnesium alloys were determined. The evaluation of HM21XA and ZH11X1, outstanding sheet alloys at high and moderate temperatures, respectively, includes mechanical properties, formability, weldability, corrosion resistance, and finishing. The effect of alloy modifications on mechanical properties, bending characteristics, and corrosion resistance of ZK60XB pellet extrusions was evaluated. The room temperature tensile properties of ZE41XA, a high strength sheet alloy, are also included.

WADC TR 57-194 Pt II  
ASTIA Document No. AD 131034  
OTS Release

September 1957

SUBJECT: INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES  
Part II Thermal and Electrical Properties of Magnesium Base Alloys

INVESTIGATOR: H. Baker

CONTRACT: AF 33(616)-2337

CONTRACTOR: The Dow Chemical Company

ABSTRACT: The electrical resistivities for cast AZ31A & B, AZ63A, AZ81A, AZ91C, AZ92A, AM100A, EK30A, EK41A, EZ33A, HK31A, rolled HK31A and HM21XA, and cast HZ32A were determined at room temperature and in some cases up to 500F. The values at 68F ranged from 17.2 microhm/cm<sup>3</sup> for AM100A-T4 to 9.2 microhm/cm<sup>3</sup> for AZ31A & B -F & -T4 and from 7.7 microhm/cm<sup>3</sup> for cast HK31A-T6 to 5.0 microhm/cm<sup>3</sup> for rolled HM21XA-T8. At 500F the values ranged from 11.6 microhm/cm<sup>3</sup> for EK41A-T6 to 9.1 microhm/cm<sup>3</sup> for HM21XA-T8 for the RE and Th containing alloys. Room temperature resistivity measurements on cast EM31 and EK31 ranged between 7.8 and 5.5 microhm/cm<sup>3</sup> for the -T6 and -T7 tempers respectively. The temperature coefficients of electrical resistivity were fairly constant ranging from 0.8 to 1.0 microhm/cm<sup>3</sup>-°F. Calculations of thermal conductivity from electrical data are reported for AZ31A & B, AZ63A, AZ81A, AZ91C, AZ92A, AM100A, EK30A, EK41A, EZ33A, HK31A, HM21XA, and HZ32A. The temperature coefficients of thermal conductivity were all positive

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and decreased with increasing temperature. Enthalpy, specific heat and heat of fusion were measured for AZ31B, HK31A, HM21XA, HM31XA, and ZK60A. The heat of fusion seemed to decrease with increasing alloy content from 82 cal/gm for HM21XA to 76 cal/gm for ZK60A. At temperatures around 500F the specific heats of the alloys were about the same as that of pure magnesium except the specific heat of AZ31B which was somewhat higher.

WADC TR 57-194 Pt III  
ASTIA Document No. AD 131035  
OTS Release

September 1957

SUBJECT: INVESTIGATION OF ALLOYS OF MAGNESIUM AND THEIR PROPERTIES  
Part III Development of Preferred Orientation in Wrought Magnesium Alloys

INVESTIGATOR: S. L. Couling  
CONTRACT: AF33(616)-2337  
CONTRACTOR: The Dow Chemical Company

ABSTRACT: A polarized-light metallographic technique capable of supplying information on the orientation of individual grains in a polycrystalline magnesium aggregate has been developed and used as a tool in several orientation studies. The distribution of preferentially oriented grains in various extruded alloys has been examined and it was found that elongated clusters of grains of one orientation alternate with clusters of another orientation throughout much of the extrusion thickness. Inhomogeneous flow or "banding" has been found to be an important deformation mechanism during the cold rolling of certain alloys and the hot compression of alloy cylinders. The bands are believed to be formed by a fracture-glide mechanism. Banding may be operative during hot rolling with large reductions per pass and could account for the observed double peak in the preferred orientation of most rolled alloys. Polycrystalline material with a preferred orientation close to the ideal can be twinned almost completely by the application of a sufficient compressive stress parallel to the sheet surface; on annealing, the heavily twinned metal recrystallizes in a preferred orientation markedly different from the initial ideal orientation.

WADC TR 57-241 Pt I  
ASTIA Document No. AD 131018  
OTS Release

August 1957

SUBJECT: DEVELOPMENT OF A CORROSION RESISTANT MAGNESIUM ALLOY  
Part I. Development of Magnesium Alloys for Better Corrosion Resistance

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INVESTIGATOR: M. Balicki  
C. D'Antonio  
A. Kravic  
CONTRACT: AF 33(616)-2917  
CONTRACTOR: Polytechnic Institute of Brooklyn  
ABSTRACT: A novel way for finding alloys of magnesium with better corrosion resistance has been evolved. This scheme relies upon alloying magnesium with elements which, owing to their lower surface tension, are expected to concentrate on the surface and thus alter the corrosion behavior. From a number of elements having a lower surface tension than magnesium the following: Hg, Pb, Ge, In, Bi, Sn, Cd, Ca have been added to it to form very pure, dilute, binary alloys. Since this was a feasibility study, insufficient data was obtained to allow a statistical analysis of results. The preliminary results obtained in general, however, indicate that magnesium alloys containing Sn, Sb, Ge, Cd, and In compare favorably with pure magnesium in the QQ-M-151A Salt Spray Test.

WADC TR 57-241 Pt II  
ASTIA Document No. AD 131010  
OTS Release

August 1957

SUBJECT: DEVELOPMENT OF A CORROSION RESISTANT MAGNESIUM ALLOY  
Part II Surface Tension Data of Elements  
INVESTIGATOR: V. P. Siuta  
M. Balicki  
CONTRACT: AF 33(616)-2917  
CONTRACTOR: Polytechnic Institute of Brooklyn  
ABSTRACT: Experimental and estimated values of surface tension of 75 elements have been gathered. This unique collection was of value in elucidating the magnesium alloying program and should be of use to workers in other fields.

Correlations for estimating surface tension values of elements have been scrutinized, augmented and used. Extensive bibliography on the subject of surface tension is presented.

WADC TR 57-518  
ASTIA Document No. AD 142197

December 1957

SUBJECT: DETERMINATION OF THE FORGING CHARACTERISTICS OF NEW AND HIGH TEMPERATURE MAGNESIUM ALLOYS  
INVESTIGATOR: R. E. Hook  
J. F. Pashak  
CONTRACT: AF 33(616)-3578  
CONTRACTOR: The Dow Chemical Company

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**ABSTRACT:** This laboratory forging program was established for the purpose of selecting the best magnesium alloy or alloys for elevated temperature service. The alloys investigated were three Mg-Th alloys (HM21XA, HK31A, HZ32A) and four Mg-RE alloys (EK31(MM), EZ33A, EK31(Di), EM41XA); two commercial alloys (AZ80A, ZK60A) were included for comparative purposes. Both cast ingot and extruded billet were used as forging stock. Static properties and creep resistance of the alloys were determined on forged discs of 90% reduction. The data, therefore, cannot be considered as representative of die forgings.

Of the alloys investigated, HM21XA-T5 exhibits the best combination of properties in the range of 500 to 700F while EK31XA-T6 presents the optimum values at 70 to 500F. Pre-forging cast ingot has no significant effect on the properties of forgings when the final forging reduction is as great as 75%; however, increasing pre-forging temperature has a deleterious effect on the final forged properties.

Evaluation of closed die forgings produced on a 3000 ton press show that ease of forging (die filling) increases in the following order: HM21XA, HK31A, EK31 (Di), commercial alloy AZ80A and ZK60A. Temperature and pressure requirements to fill the die for a given alloy increase as the draft angle (7°, 3°, 0°) decreases.

WADC TR 57-576

December 1957

ASTIA Document No. AD 142209

OTS Release

**SUBJECT:** A BASIC STUDY OF CORROSION OF MAGNESIUM  
**INVESTIGATOR:** Richard R. Addiss            Herbert Hollister  
                  Mitchell S. Cohen            Henri S. Sack  
                  Robert I. Frank            Karl Scharf

**CONTRACT:** AF 33(616)-3032

**CONTRACTOR:** Cornell University

**ABSTRACT:** This report constitutes the final report of the work done under the present contract. Attempts to obtain a perfectly clean surface of magnesium are described. Due to limitations in time no definite results on the oxidation of Mg were obtained; however some preliminary results on the patterns obtained from pure Mg in a field emission microscope, and the oxidation at low temperatures are reported.

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METALS - NONFERROUS TITANIUM

WADC TR 54-112 Pt II  
ASTIA Document No. AD 142198

December 1957

SUBJECT: INTERMEDIATE TEMPERATURE CREEP AND RUPTURE BEHAVIOR  
OF TITANIUM AND TITANIUM-BASE ALLOYS  
Part II: Influence of Microstructures on Creep-  
Rupture Properties

INVESTIGATOR: Jeremy V. Gluck  
James W. Freeman

CONTRACT: AF 33(616)-244

CONTRACTOR: The University of Michigan

ABSTRACT: An investigation was carried out to establish the relationships between types of microstructure and creep-rupture properties of titanium alloys at 600°F to 1000°F. Chemical composition and the influence of heat treatment was investigated to a limited extent for each type of alloy. The data are fairly complete for some alloys and consist of survey test data for others.

Alpha titanium as represented by commercially pure titanium had the lowest strength. Stable alpha alloys (6% Al and 6% Al - 0.5% Si) and stable beta alloys (30% Mo and 50% V) had similar strengths. These alloys had the highest strength at 800°F and 1000°F where creep was a major factor. The alpha-beta alloys (Ti 150A and Ti 155AX) and the meta-stable beta alloys had high strengths through 600°F and for short time periods at 800°F by virtue of strengthening from transition structures from the beta to alpha transformation.

Compositional differences between alloys had rather small effects in most cases. Properties of the alpha-beta and meta-stable beta alloys were markedly influenced by heat treatment except at 1000°F. The other structures were little influenced by heat treatment. Ductility varied considerably between alloys and with heat treatment and test conditions.

None of the alloys were subject to appreciable creep at 600°F and strength was governed by tensile properties for time periods up to 1000 hours. Increasing tensile strength by cold work or by heat treatment permitted application of high stresses which usually caused immediate failure at 600°F when the stress was above the yield strength. This seemed to be a delayed tensile fracture rather than true creep to rupture. Creep and structural stability were the major factors in strength at 800°F and 1000°F.

WADC TR 54-112 Pt III  
ASTIA Document No. AD 142227

December 1957

**SUBJECT:** INTERMEDIATE TEMPERATURE CREEP AND RUPTURE BEHAVIOR  
OF TITANIUM AND TITANIUM-BASE ALLOYS  
Part III: Effects of Hot Rolling, Embrittlement, and  
Interstitial Elements

**INVESTIGATOR:** Jeremy V. Gluck  
James W. Freeman

**CONTRACT:** AF 33(616)-244

**CONTRACTOR:** The University of Michigan

**ABSTRACT:** Three major factors involved in the properties of titanium  
alloys at high temperatures were investigated:

1. A limited study was made of the influence of hot-working conditions on creep-rupture properties at 600°F to 1000°F.
2. The changes in room temperature tensile test ductility as a measure of embrittlement during creep testing at 75°F to 1000°F were evaluated for a considerable number of specimens of several alloys.
3. A limited study was carried out on the influence of hydrogen content and the hardness of the sponge (interstitial alloying elements) used in making alloys. Creep-rupture properties and embrittlement during stressed exposure were evaluated for temperatures of 600°F to 1000°F.

WADC TR 54-278 Pt IV  
ASTIA Document No. AD 142147  
OTS Release

November 1957

**SUBJECT:** DEVELOPMENT OF TITANIUM-BASE ALLOYS FOR ELEVATED  
TEMPERATURE APPLICATION

**INVESTIGATOR:** Frank A. Crossley  
William F. Carew  
David W. Levinson

**CONTRACT:** AF 33(616)-2853

**CONTRACTOR:** Armour Research Foundation

**ABSTRACT:** The objectives of this research program were as follows:

- (1) To determine the effect of forging and heat treatment variables on the mechanical properties of the Ti-7Al-3Mo alloy. Evaluations were made of tensile specimens, both machined from bar stock and cut from plate fabri-

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cated in such a manner as to simulate compressor blade forging practice. The purpose of this dual evaluation was to determine what effect the forged skin had on mechanical properties.

- (2) To determine the effect of sponge quality on mechanical properties of 7Al-3Mo alloy.
- (3) To evaluate the tensile, creep-rupture and stability properties of the alloys Ti-6Al-3Nb, Ti-6Al-3Mo-2Ge and Ti-6Al-3Mo-4Ge.

The program for the evaluation of the Ti-7Al-3Mo alloy encountered considerable difficulties with the result that the work was halted before evaluation was completed. The material with which the program was started was discarded because of substandard ductility which was attributed to high oxygen content. While considerable data were collected on the replacement material, it too proved to have substandard ductility. The cause of the low ductility in the replacement material could not be attributed to off-chemistry of aluminum, molybdenum, carbon, hydrogen, nitrogen or oxygen. The sulfur content was indicated to be 0.034% by analyses conducted at the Foundation and 0.01% by analyses conducted by Mallory-Sharon. While the low ductility of this material cannot with any degree of surety be attributed to sulfur, it is a possibility.

The significant findings from the data collected on the low ductility replacement 7Al-3Mo material were the following:

- (1) In heat treating, increasing the solution treating temperature followed by water quenching increases the time required to achieve overaging.
- (2) The Widmanstätten structure has creep-rupture properties at 1000°F superior to the equiaxed structure.
- (3) The "skin" effect (as represented by comparing test specimens machined from bar stock with specimens cut from forged plate) on creep-rupture properties at 1000°F was considered to be masked and perhaps dominated by the "size" effect, i.e., ratio of surface area to volume. The forged specimens which were higher in area-to-volume ratio were found to have lower creep-rupture strength compared to machined specimens. This result was in agreement with findings of other investigators for the size effect.

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It is emphasized that all data for the Ti-7Al-3Mo alloy reported herein unless stated otherwise were determined for low ductility materials.

The 6Al-3Nb alloy contained less  $\beta$  than the 6Al-3Mo alloy and was higher in tensile strength, but lower in ductility at room temperature. The 6Al-3Mo-2Ge and 6Al-3Mo-4Ge alloys were significantly stronger at room temperature than 6Al-3Mo. The 6Al-3Mo-4Ge alloy was very low in ductility.

The alloys in order of increasing creep resistance at 800°F were: 6Al-3Nb, 6Al-3Mo, 6Al-3Mo-2Ge and 6Al-3Mo-4Ge. The alloys in order of increasing 500-hour rupture strength at 1020°F were: 6Al-3Nb, 6Al-3Mo-2Ge and 6Al-3Mo and 6Al-3Mo-4Ge. Only the 6Al-3Mo-2Ge alloy showed loss of ductility in stability tests.

Only the 6Al-3Mo-4Ge alloy was superior in creep-rupture properties to 6Al-3Mo. However, the ductility of this alloy is considered too low for practical application.

WADC TR 54-546 Pt II  
ASTIA Document No. AD 151010  
OTS Release

March 1958

SUBJECT: PILOT PRODUCTION, FABRICATION AND EVALUATION  
OF PROMISING TITANIUM ALLOYS

INVESTIGATOR: Frank A. Crossley  
Benjamin R. Rajala  
David W. Levinson

CONTRACT: AF 33(616)-2060

CONTRACTOR: Armour Research Foundation

ABSTRACT: Approximately 300 pounds of Ti-7Al-3Mo alloy were fabricated to bar stock and distributed to various jet engine manufacturers. About 70 pounds of hot-rolled, centerless-ground bar was sent to Thompson Products, Inc. They were highly successful in manufacturing 140 J-57 7th stage compressor blades from this material. The endurance limit for blades bench-tested at room temperature under completely reversed stress was 75,000 psi, 5000 psi higher than Ti-6Al-4V alloy blades of the same configuration. Tensile properties for specimens cut from blades and heat treated: 1560°F-1/2 hour-air cooled, 1020°F-24 hours, were as follows: 170,000 psi ultimate tensile strength, 167,000 psi 0.2% offset yield strength, 49% reduction in area, and 16% elongation. As a result of the promising preliminary evaluations, Pratt and Whitney Aircraft, Division of United Aircraft, is preparing to engine test these blades.

Johnston and Funk Titanium Corporation was successful in producing, on an experimental basis, wire in sizes down to 0.020 in.

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diameter from Ti-7Al-3Mo 9/32 in. diameter rod. No difficulty in supplying production quantities is anticipated.

The high strength condition produced by applying the heat treatment: 1800°F-1 hour-water quench, 1000°F-2 hours to equiaxed Ti-7Al-3Mo alloy material was found to be unstable to aging under stress at 800°F.

Tests conducted by Eaton Manufacturing Company indicated that the alloy is stress-relieved by 2 hours at 1300°F.

The Ti-7Al-3Mo alloy shows considerable promise. This is a fact implicit in the production by Cramet, Inc., of 1500 to 2000 pounds of Ti-7Al-3Mo material for sale, and the announcement by Rem-Cru Titanium, Inc., of the availability of experimental quantities of their version of the alloy containing 6-1/2% aluminum and 3-3/4% molybdenum.

WADC TR 54-616 Pt IV  
ASTIA Document No. AD 131088  
OTS Release

September 1957

SUBJECT: HYDROGEN CONTAMINATION IN TITANIUM AND  
TITANIUM ALLOYS  
Part IV: The Effect of Hydrogen on the Mechanical  
Properties and Control of Hydrogen in Titanium Alloys

INVESTIGATOR: D. N. Williams W. M. Albrecht  
F. R. Schwartzberg M. W. Mallett  
P. R. Wilson R. I. Jaffee

CONTRACT: AF 33(616)-2813  
CONTRACTOR: Battelle Memorial Institute

ABSTRACT: A comprehensive investigation of hydrogen in titanium alloys was carried out. Tests were conducted to gain an insight into the mechanism of the slow-strain embrittlement of titanium alloys by hydrogen, and a tentative theory is presented. Eighty titanium alloys were examined in the stabilized condition to determine the effect of composition on the tendency toward hydrogen embrittlement. The effect of microstructural variations on the tendency toward embrittlement was also studied. In addition to studying the effects of hydrogen on the properties of titanium, factors affecting hydrogen pickup by titanium and methods of removing hydrogen from titanium were investigated. Included were investigations of low pressure solubility, degassing methods, and the pickup of hydrogen from various atmospheres.

WADC TR 57-251 Pt I  
ASTIA Document No. AD 130847  
OTS Release

June 1957

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**SUBJECT:** HIGH TEMPERATURE BRITTLENESS IN TITANIUM ALLOYS  
**INVESTIGATOR:** Nick Makrides  
W. M. Baldwin, Jr.  
**CONTRACT:** AF 33(616)-3536  
**CONTRACTOR:** Case Institute of Technology  
**ABSTRACT:** An alpha-titanium alloy (A-110-AT) containing 5% Al and 2.5% Sn, and an alpha-beta titanium alloy (Ti-140-A) containing 2% Cr, 2% Fe, and 2% Mo were each prepared (a) free from impurities, (b) with 0.2% O, (c) 0.2% C, and (d) 0.1% N for a program designed to explore strain aging phenomena, high temperature brittleness, and stress-rupture behavior of titanium alloys.

Tensile tests at different strain rates have been made on the alpha-alloy containing 0.1% N. The tests revealed a brittle range at 680°F (360°C) at a strain rate of 0.05 in/in/min. This brittle range was moved to 1020°F (550°C) employing a strain rate of 100 in/in/min. The minima in the ductility vs. temperature curves for the two strain rates when plotted on log strain rate vs. reciprocal absolute temperature coordinates gives a straight line (assumed) having a slope which is parallel to the line defining the diffusion coefficient of nitrogen in alpha-titanium. On the other hand, tests are still to be made on the impurity-free, alpha-alloy to see whether the brittleness occurs in the absence of nitrogen.

A review was made of the various types of brittleness found at elevated temperatures in metals and alloys so that any brittleness encountered in titanium could be compared and identified.

WADC TR 57-269  
ASTIA Document No. AD 142137  
OTS Release

November 1957

**SUBJECT:** PARTITION OF SOLUBLE CARBON IN Ti-6Al-4V ALLOY  
**INVESTIGATOR:** Richard D. Seibel  
Richard L. Beck  
Leonard E. Olds  
**CONTRACT:** AF 33(616)-3349  
**CONTRACTOR:** Denver Research Institute  
**ABSTRACT:** The mechanism by which alpha-beta titanium alloys are embrittled by small amounts of interstitial-type solutes such as nitrogen, oxygen and carbon can not be fully understood until more is known of the micro-distribution of these dissolved impurities in the two-phase alloys. This report describes the results of an investigation of possible means for determining the micro-distribution of these impurities, particularly in the more complex alloys.

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A useful method was developed for measuring micro-distribution of solutes in alloys. This method was completely independent of the presence of other solute elements, whether interstitial or substitutional. In addition, some information is presented regarding the use of micro-autoradiographic techniques to study the micro-distribution of the interstitial type solutes in two-phase titanium alloys.

WADC TR 57-360  
ASTIA Document No. AD 142142  
OTS Release

November 1957

SUBJECT: RESEARCH ON THE EFFECTS OF STRESS, STRAIN, AND  
TEMPERATURE ON THE EUTECTOID DECOMPOSITION OF TITANIUM ALLOYS

INVESTIGATOR: Adolph W. Goldenstein  
Arthur G. Metcalfe  
William Rostoker

CONTRACT: AF 33(616)-3394  
CONTRACTOR: Armour Research Foundation

ABSTRACT: Three titanium-chromium alloys have been forged in the  $\alpha$ - $\beta$  range to give six systems with controlled amounts of each phase. The isothermal transformation at 400°, 500°, and 600°C of each of these systems was followed by resistivity, X-ray diffraction, elastic modulus measurements, and metallography. The reactions occurring in the transformation were identified where possible. This transformation study was repeated under a stress which produced 1% creep in 1000 hours. An acceleration of four to seven times in the rate of transformation occurred under the action of this stress. Complete tensile test data were obtained at all stages of the transformation and plotted on the T-T-T diagrams. These reveal that the formations of omega and TiCr<sub>2</sub> are the embrittling reactions.

The eutectoid temperature in the Ti-Cr system has been determined to be 670° ± 5°C.

WADC TN 57-396  
ASTIA Document No. AD 142283  
OTS Release

January 1958

SUBJECT: THE EFFECT OF HEAT TREATMENT ON THE STABILITY  
AND CREEP RESISTANCE OF A TI-AL-MO ALLOY

INVESTIGATOR: Harold L. Gegel, 1/Lt.

ABSTRACT: An investigation was undertaken to observe the influence of heat treatment on stability of a Ti-Al-Mo ternary alloy. Tests show that the instability of the alloy is innocuous, and that transformation during testing helps to maintain the material strength. The creep resistance of the alloy was improved by heat treatment, and the stress-rupture properties were not deteriorated. A mechanism of embrittlement by heat treatment is discussed.

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WADC TR 57-420  
ASTIA Document No. AD 142145  
OTS Release

November 1957

SUBJECT: STUDY OF MICRODISTRIBUTION OF INTERSTITIAL ELEMENTS  
IN TITANIUM BY INTERNAL FRICTION TECHNIQUES

INVESTIGATOR: Devendra Gupta  
Sheldon Weinig

CONTRACT: AF 33(616)-3393

CONTRACTOR: New York University

ABSTRACT: An investigation of the microdistribution of interstitial elements in Ti and Ti binary alloys and its effect on the mechanical properties was initiated. The experimental techniques which were utilized during this period were internal friction measurements and "hard" tensile testing. The overall study can be separated into three separate sections, i.e. room temperature effects, interstitial stress relaxation, and grain boundary effects. The major results of the investigation are as follows:

1. A study of the solute atom-dislocation interaction of Ti-O alloys was performed. It was found that the behavior of the phenomenon was in accord with the relationship,  $C = C_0 \exp (V/KT)$ . The interaction energy between an oxygen atom and a dislocation was calculated to be 0.015 ev.

2. The oxygen content required to saturate the dislocations was found to be approximately 1.5 atomic percent. It was also observed that the transition from ductile to brittle behavior occurred at the same composition.

3. It was suggested that the transition phenomenon was due to the adsorption of oxygen atoms at the dislocations. In view of this suggestion it was possible to predict the proper treatment to shift the onset of the embrittling effect to higher oxygen contents. This was demonstrated experimentally and it was possible to increase the ductility of Ti-O alloys.

4. It was demonstrated that an interstitial stress relaxation phenomenon could be observed in "superpurity" titanium.

5. The development of the interstitial peak in alpha titanium is shown to be directly related to the atomic size of the substitutional element in solution with the oxygen. The greater the difference in size factor between the solute and the titanium atom, the greater the enhancement of the "interstitial" peak.

6. It is suggested that the mechanism whereby the substitutional solute atoms enhance the appearance of the interstitial peak is as follows: The out-of-size substitutional atom causes a localized distortion of the lattice which effectively destroys the isotropic nature of the



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octahedral interstitial sites. Hence, one site becomes energetically favorable to another upon the application of an external stress. This causes a stress-induced interstitial relaxation phenomenon which is manifested by the appearance of an internal friction peak.

7. The effect of oxygen on the grain boundary stress relaxation phenomenon was investigated. The necessary data is now available for the adsorption analysis and microdistribution part of this overall study of interstitial effects in titanium.

WADC TR 57-470  
ASTIA Document No. AD 142196  
OTS Release

December 1957

SUBJECT: DETERMINATION OF TESTS FOR HYDROGEN EMBRITTLEMENT  
OF TITANIUM ALLOYS

INVESTIGATOR: Albert H. Fleitman

CONTRACT: AF 33(616)-3244

CONTRACTOR: Crane Co.

ABSTRACT: The mechanical properties of two commercial alloys, Ti-8 Mn and Ti-4 Al-4 Mn were investigated in the as received, helium annealed, vacuum annealed, and 150, 225 and 300 ppm hydrogen conditions. The Ti-8 Mn alloy in the form of 1/16 inch sheet and the Ti-4 Al-4 Mn alloy in the form of 1/2 inch round bar were each ordered from three vendors to represent the range of interstitial content found in present production.

Slow room temperature notched and unnotched tensile and tensile stress rupture tests up to 1000 hours were performed on both alloys. Slow room temperature notched and unnotched torsion, bend and bend stress rupture tests up to 1000 hours were made on Ti-4 Al-4 Mn alloy. None of the unnotched specimens in any of these tests failed in a brittle manner. The notched tensile stress rupture tests showed that the stress required for fracture at a given time slightly decreased with increasing hydrogen content. In contrast, the notched bend stress rupture tests showed an increase in stress required for fracture.

Longitudinal and transverse tear tests at 80, 32 and -40°F were made on the Ti-8 Mn alloy sheet in the vacuum annealed and 300 ppm hydrogen condition. No apparent relationship was found between tear test failures and hydrogen content. Pin shear tests on Ti-4 Al-4 Mn alloy specimens containing similar quantities of hydrogen failed with ductile fractures.

Room temperature unnotched slow tensile tests on prestrained and aged specimens showed good ductility in both alloys. Specimens in the vacuum annealed and 300 ppm hydrogen condition were prestrained 5% in tension at room temperature and aged at 500°F for 24 or 48 hours prior to test.

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Comparison of results in this investigation with earlier work on hydrogenized specimens of these titanium alloys indicates that brittle fractures would have been expected in both alloys when tested in stress rupture at the 300 ppm hydrogen level.

WADC TR 57-630  
ASTIA Document No. AD 151000  
OTS Release

February 1958

SUBJECT: THE DETERMINATION OF THE EFFECT OF HEAT TREATMENT ON  
THE ELEVATED TEMPERATURE STRESS-STABILITY OF TITANIUM ALLOYS  
INVESTIGATOR: G. A. Lenning H. D. Kessler  
M. L. Greenlee  
W. M. Parris  
CONTRACT: AF 33(616)-3638  
CONTRACTOR: Titanium Metals Corporation of America  
ABSTRACT: The effect of duplex solution and age type heat treatments on the properties of one commercial heat each of the Ti-140A (Ti-2Fe-2Cr-2Mo), Ti-155A (Ti-5Al-1.5Fe-1.5Cr-1.5Mo) and Ti-6Al-4V alloys were investigated. The properties studied included tensile, notch tensile, notch-bend impact and elevated temperature stress-stability.

WADC TR 57-639  
ASTIA Document No. AD 151061  
OTS Release

March 1958

SUBJECT: A STUDY OF THE FACTORS INFLUENCING THE PROPERTIES  
OF HEAT TREATABLE TITANIUM SHEET ALLOYS  
INVESTIGATOR: R. S. Richards  
D. L. Day  
H. D. Kessler  
CONTRACT: AF 33(616)-3727  
CONTRACTOR: Titanium Metals Corporation of America  
ABSTRACT: A number of factors were investigated which influence the properties of two titanium sheet alloys, Ti-4Al-3Mo-1V and Ti-2Al-6Mo-2V, in the annealed, solution treated, and aged conditions. Annealed material exhibits a narrow spread between yield and ultimate tensile strengths along with the best bendability. Solution treated sheet possesses a high degree of uniform elongation with bendability that is only slightly inferior to that of annealed material. The high-strength aged alloy sheet exhibits a lower level of ductility, and is stable under stress at temperatures to at least 800F.

Elevated temperatures lower the strength, decrease uniform elongation, but increase the bendability and local elongation at fracture. Cold work increases the strength in all three conditions of heat treatment.

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although deformation at elevated temperatures produces a smaller strengthening effect in annealed and aged material as the temperature increases. Temperatures of 400 - 600F initiate the age hardening reaction in solution treated sheet. Cold deformation of solution treated Ti-4Al-3Mo-1V results in increased aged strengths, while Ti-2Al-6Mo-2V exhibits significant losses in final aged strengths after prior cold work in the solution treated condition.

Successful dimpling operations can be performed at elevated temperatures provided proper heating and forming techniques are used. Solution treated sheet from both alloys is amenable to bending, stretch wrapping, jogging, and hydroforming operations at room temperature, as indicated by results of production forming tests. No appreciable dimensional changes occur in the material as a result of aging.

WADC TR 57-694  
ASTIA Document No. AD 151125  
OTS Release

April 1958

SUBJECT: METALLURGICAL AND MECHANICAL CHARACTERISTICS OF  
HIGH-PURITY TITANIUM-BASE ALLOYS  
INVESTIGATOR: Frank C. Holden Horace R. Ogden  
Jerry A. Houck Robert I. Jaffee  
CONTRACT: AF 33(616)-3469  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: Studies have been made to establish the relationships between thermal history, microstructure, and mechanical properties for high-purity titanium-base alloys. These have included the following alloy systems: Ti-Al-Mo, Ti-O-Mo, Ti-C-Mo, Ti-O-Al-Mo, Ti-O-Mn, Ti-O-Cu, Ti-C-Cu, and Ti-Cu-Mn. Mechanical test data reported include tensile and flow properties, impact behavior over a range of temperatures, hardness, aging, and cooling-rate test data. The basic physical metallurgical principles involved here are discussed.

WADC TR 58-20  
ASTIA Document No. AD 151029  
OTS Release

March 1958

SUBJECT: DEVELOPMENT OF IMPROVED TITANIUM ALLOYS FOR  
APPLICATION AT ELEVATED TEMPERATURES  
INVESTIGATOR: Bernard S. Lement  
CONTRACT: AF 33(616)-3986  
CONTRACTOR: Manufacturing Laboratories, Inc.  
ABSTRACT: A metallurgical investigation of the embrittling reaction that occurs in binary Ti-Al alloys was carried out in the range of 6 to 12 wt. pct. aluminum. Changes in bend ductility, hardness, precision length, electrical resistance, lattice parameters, and in microstructure that occur on solutionizing and aging were determined. It was found that the embrittl-

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ing reaction that occurs on aging in the vicinity of 1020 F (550 C) is characterized by a decrease in both specific volume and electrical resistivity as well as a marked change in the character of striations that exist within the primary grains of the alpha solid solution matrix. The observed changes in properties are best explained on the basis that these striations represent the formation of aluminum-rich segregations. Evidence was found that cracking in a bend test occurs more readily along rather than across these striations. Recommendations are given for future work on this problem.

## PACKAGING

WADC TN 55-328  
ASTIA Document No. AD 142004  
OTS Release

September 1957

**SUBJECT:** EVALUATION OF CONTAINER-GRADE PAPER-OVERLAID VENEER PANEL BOXES FOR OVERSEAS USE

**INVESTIGATOR:** Edward H. Clarke

**CONTRACT:** AF 33(600)53-4023

**CONTRACTOR:** Forest Products Laboratory

**ABSTRACT:** Laboratory tests were conducted in an effort to investigate the suitability of some commercially available paper-overlaid veneers for use in overseas-type, cleated panel boxes. Rough-handling tests and diagonal-compression tests involving some 220 overseas-type boxes provided information regarding the relative performance of various paper-overlaid veneers and two currently acceptable panel materials, V3s fiberboard and container-grade plywood.

After immersion in water for 24 hours, the cleated plywood boxes were superior to V3s fiberboard and paper-overlaid veneer in both rough handling and resistance to diagonal distortion or twisting. Only a few paper-overlaid veneer materials approached the rough-handling performance of V3s fiberboard panel boxes, except when intermediate cleats were used as required for the fiberboard. Then the paper-overlaid veneer boxes exhibited a rough-handling performance equal to that of the V3s fiberboard boxes. The paper-overlaid veneer boxes resisted compressive forces better than the cleated fiberboard boxes.

WADC TR 56-342 Sup 1  
ASTIA Document No. AD 151195  
OTS Release

May 1958

**SUBJECT:** THE THEORY AND OPERATION OF A DYNAMIC TESTER FOR EVALUATING PACKAGE CUSHIONING MATERIAL

**INVESTIGATOR:** Stewart M. Krakover, 1/Lt.

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**ABSTRACT:** The purpose of the ASTM dynamic cushion testing program is described and data from the laboratories which participated in this program are presented. On the analysis of the platform type tester together with a discussion of the significance of the elasticity of impact of containers is covered. Several redesigns of the dropping carriage are shown. The desirability of using the platform tester to gather design data is discussed. A discussion is included of the peak reading voltmeter system and a portable spring calibrator.

WADC TR 56-539  
ASTIA Document No. AD 130873  
OTS Release

July 1957

**SUBJECT:** TESTS TO EVALUATE AND COMPARE VARIOUS OPEN CRATE SPECIFICATIONS FOR AIRCRAFT SURFACES  
**INVESTIGATOR:** Thomas B. Heebink  
**CONTRACT:** PO 33(600)53-4023  
**CONTRACTOR:** Forest Products Laboratory  
**ABSTRACT:** Thirteen open crates for aircraft surfaces were tested to evaluate the construction details of various open crate specifications. The results of these tests suggested various advantageous modifications which were incorporated into a final design and tested. The result was that the three crates built according to the final modified design were superior generally in all respects including performance, tare weight, cubic displacement, and cost.

The final design was incorporated into the proposed specification, being prepared by the Forest Products Laboratory for WADC entitled "Crates, Wood (For Lightweight, Bulky Airframe Items)."

WADC TR 56-547  
ASTIA Document No. AD 131019  
OTS Release

August 1957

**SUBJECT:** INVESTIGATION OF SHOCK WAVES DEVELOPED DURING DYNAMIC TESTS OF CUSHIONING MATERIALS  
**INVESTIGATOR:** Vern N. Smiley  
**CONTRACT:** AF 33(600)53-4023  
**CONTRACTOR:** Forest Products Laboratory  
**ABSTRACT:** Certain irregularities of acceleration-time pulses were recorded during dynamic compression tests of package cushioning materials for determining their energy absorption characteristics. These irregularities were theorized to have been caused by shock wave oscillations in the material. The validity of this theory was checked by deriving mathematical expressions based upon shock wave theory and then comparing theoretically expected results with actual test results. Sufficiently close agreement was found to prove that the irregularities were caused by shock waves. A discussion of the significance of shock waves in relation to cushioning performance is also made.

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WADC TR 56-660  
ASTIA Document No. 118314  
OTS Release

May 1957

**SUBJECT:** AN EVALUATION OF POPCORN AS A CUSHIONING MATERIAL  
**INVESTIGATOR:** R. K. Stern  
**CONTRACT:** DO 33(600)53-4023  
**CONTRACTOR:** Forest Products Laboratory  
**ABSTRACT:** This investigation had the purpose of determining if popcorn could be used as a cushioning material. Conventional cushioning materials such as bound hair and cellulosic wadding were used as a basis for comparison. Stress-strain curves were established for popcorn, bound hair, and cellulosic wadding under several conditions of temperature and humidity. Repeated loading tests on these materials were also performed. The mold resistance and the effect of high humidity on treated and untreated popcorn were established. Combustibility of the materials was also investigated. The results showed that popcorn is not a good cushioning material. It is stiff in comparison to other cushioning materials and has very little ability to recover after compression. The extreme hygroscopicity of popcorn is undesirable since it shrinks appreciably in high humidity. Its only conceivable use is indicated to be as a blocking material in moisture-vaporproof packages.

WADC TR 57-682  
ASTIA Document No. AD 142282  
OTS Release

January 1958

**SUBJECT:** THE DEVELOPMENT OF A NON-ADHERING CHEMICALLY FOAMED-IN-PLACE POLYURETHANE CUSHIONING MATERIAL FOR PACKAGING PURPOSES  
**INVESTIGATOR:** Sidney Childers  
Sidney Allinikov  
**ABSTRACT:** A chemical process for the production of a non-adhering chemically foamed-in-place polyurethane foam is presented. An evaluation of several organic fatty acids as a release agent for chemical foamed polyurethane cushioning is included. Fatty acids such as stearic acid in proper concentration were found to impart excellent release properties to foam after foaming on bare metal and glass surfaces. Further benefits from the release agent are shown to be improvement of the stress-strain properties and the ability to vary density and hence cushioning properties by the use of sufficient amounts of fatty acid. The effect of the release agent on the foaming process and the finished cushion is discussed. Graphs and a comparison analysis of the dynamic and static cushion properties are presented for a polyurethane cushion with and without a release agent.

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## PLASTICS, STRUCTURAL

WADC TR 52-183 Sup 5  
ASTIA Document No. AD 142081

October 1957

**SUBJECT:** ANNUAL REPORT ON RESEARCH FOR USE IN ANC-17  
BULLETIN "PLASTICS FOR AIRCRAFT"  
**INVESTIGATOR:** Donald G. Coleman  
**CONTRACT:** AF 33(616)-56-9  
**CONTRACTOR:** Forest Products Laboratory  
**ABSTRACT:** Developments in the program of research in plastics for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1957 are summarized. The approach has been in general to derive criteria mathematically and then to check by test. Eight technical reports issued during the fiscal year are abstracted.

WADC TR 53-185 Pt 4  
ASTIA Document No. AD 155501

May 1958

**SUBJECT:** THE STUDY OF EROSION OF AIRCRAFT MATERIALS AT HIGH SPEEDS IN RAIN  
**INVESTIGATOR:** Roy R. Lapp  
Donald H. Thorpe  
Raymond H. Stutzman  
Norman E. Wahl  
**CONTRACT:** AF 33(616)-2758  
**CONTRACTOR:** Cornell Aeronautical Laboratory  
**ABSTRACT:** The results obtained on the relative rain erosion resistance, at subsonic speeds, of a large number of different types of materials, are reviewed in detail in this report.

Spray or brush-on types of coatings were evaluated for conformance to Military Specification MIL-C-7439B. Only those coatings based upon neoprene had erosion resistance meeting the requirements.

Erosion tests were conducted on a large number of white coatings based upon various pigmented polyacrylics, silicones, chlorosulfonated ethylene and neoprene elastomers. Of all these white materials tested, coatings based upon Hypalon alone and combinations of white pigmented neoprene and polyacrylic rubber appeared to merit further study.

Of the glass and ceramic materials tested, alumina bodies with over 90% alumina had the best erosion resistance.

Comparison of current epoxy and polyester glass reinforced laminates indicates that the erosion resistance of standard test specimens of epoxy-glass laminates have four to five times the erosion resistance of similar laminates made with polyester resins.

In collaboration with the National Bureau of Standards, studies on the mechanism of erosion and tests were conducted on a variety of different materials under specific conditions.

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WADC TR 53-192 Pt IX  
ASTIA Document No. AD 130909

July 1957

SUBJECT: MECHANISM OF RAIN EROSION  
PART IX - Observation of the Fragmentation of Waterdrops  
in a Zone behind an Air Shock

INVESTIGATOR: Olive G. Engel

CONTRACT: AF 33(616)53-9

CONTRACTOR: National Bureau of Standards

ABSTRACT: Observations made on the fragmentation of two waterdrop sizes after collision with air shocks that were moving at three different supersonic velocities are reported. The possible mechanism of various aspects of the fragmentation process are discussed. The experimental observations indicate that high-speed-rain-erosion damage should not be observed on spheres having a diameter as large as 4 ft and moving with a Mach Number in the range of 1.3 to 1.7 in rain having a drop diameter of 1.4 mm. Drops of this size should be reduced to mist in the zone of separation between the detached shock and the surface of the sphere according to the results that are reported. A means to extend this protection to spheres of smaller diameter or to rain of larger size is pointed out. The need for further experimental observation of the time required for the fragmentation of waterdrops using shocks moving at higher Mach Numbers is indicated to verify and extend the information.

WADC TR 53-192 Pt X  
ASTIA Document No. AD 142240

December 1957

SUBJECT: MECHANISM OF RAIN EROSION  
PART X - A Review and Evaluation of the Present State  
of the Problem

INVESTIGATOR: Olive G. Engel

CONTRACT: AF 33(616)53-9

CONTRACTOR: National Bureau of Standards

ABSTRACT: A review of the rapidly accumulating literature on the subject of erosion by waterdrop impingement has been made. The types of experimental apparatus that have been used by the investigators, and the factors that have been found to determine the extent of the erosion damage, are briefly discussed. Results of microscope and X-ray studies of eroded surfaces, and of parallel studies of damage marks produced by the impingement of steel spheres, deforming lead pellets, oil-filled gelatin capsules, and waterdrops are presented. Several theoretical estimates of the impact pressure that results from the collision of a waterdrop with a solid surface are reviewed. The result of a piezoelectric measurement of this pressure is given. Some of the theories that have been advanced in regard to the mechanism of the erosion process and of the micromechanism of failure are discussed. The important role that design can play both in mitigating the erosion and in completely bypassing the problem under some conditions is pointed out.

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WADC TR 53-192 Pt XI  
ASTIA Document No. AD 142115

November 1957

**SUBJECT:** MECHANISM OF RAIN EROSION  
PART XI - Effect of Residual Stresses and of Molding  
Variables on the Erosion Resistance of Nylon

**INVESTIGATOR:** Olive G. Engel

**CONTRACT:** AF 33(616)-53-9

**CONTRACTOR:** National Bureau of Standards

**ABSTRACT:** It is shown that residual stresses in the original plastic sheet material and the use of improper molding conditions for the fabrication of test specimens may provide an incorrect rain-erosion-resistance rating for the material in question and misleading evidence in regard to the failure mechanism of it. Test results indicate that properly molded nylon FM-10001, which was heat treated by the manufacturer to remove residual stresses in the plastic sheet, is one of the most rain-erosion resistant of the rigid plastic materials that have been evaluated at impingement velocities up to 600 mi/hr. This rigid plastic closely approaches the rain-erosion-resistance of neoprene elastomers at 600 mi/hr.

WADC TR 55-186 Pt II  
ASTIA Document No. AD 142044

October 1957

**SUBJECT:** ASBESTOS FIBER REINFORCED PLASTIC LAMINATES

**INVESTIGATOR:** Conrad C. Spatz

**CONTRACT:** AF 33(616)-2257

**CONTRACTOR:** Johns-Manville Corporation

**ABSTRACT:** The investigation to evaluate the effectiveness of medium length and short asbestos fibers in reinforcing low-pressure laminating resins was continued. This report covers the evaluation of laminates prepared from various experimental asbestos papers and phenolic resins.

A number of these laminates meet the mechanical test requirements of Military Specification MIL-P-8013 for polyester-glass mat laminates. Several of these laminates meet the flexural and edgewise compressive strength requirements of some of the glass cloth laminates in this specification. The tensile strength of these laminates is below the requirements of glass cloth laminates. The best asbestos-reinforced laminates retain 75 per cent ultimate flexural strength and up to 100 per cent flexural modulus at 500F after one-half hour at 500F.

WADC TR 55-186 Pt III  
ASTIA Document No. AD 142139

November 1957

**SUBJECT:** ASBESTOS FIBER-REINFORCED PLASTIC LAMINATES

**INVESTIGATOR:** Wayne O. Jackson

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CONTRACT: AF 33(616)-2257  
CONTRACTOR: Johns-Manville Corporation  
ABSTRACT: The investigation of medium length and short asbestos fibers as reinforcement for low-pressure laminating resins for use in structural laminates was continued. This report covers the evaluation of laminates prepared from medium length asbestos fiber paper and phenolic, epoxy, and polyester resins.

A number of these laminates meet the mechanical test requirements of Military Specification MIL-P-8013A for glass mats. Several laminates meet the flexural and edgewise compressive strength requirements of glass-cloth laminates. Phenolic, epoxy, and polyester laminates retained 100 per cent of their flexural strength and flexural modulus after three months' exposure to outdoor weathering conditions. No change was noticed after one year for the phenolic laminates while the epoxy and polyester laminates' tests are continuing for one year.

WADC TR 55-290 Pt II  
ASTIA Document No. AD 155502  
OTS Release

May 1958

SUBJECT: THE DEVELOPMENT OF FIBROUS GLASSES HAVING HIGH ELASTIC MODULI  
INVESTIGATOR: J. A. Waugh H. I. Glaser  
V. E. J. Chiochetti R. Z. Schreffler  
CONTRACT: AF 33(616)-2422  
CONTRACTOR: Owens-Corning Fiberglas Corporation  
ABSTRACT: This report describes the work done to draw fiberized glass having a high modulus of elasticity, from two kinds of glass cullet; that containing calcium aluminate and that containing beryllium oxide.

Fiberized glass was obtained from each although not in any quantity. Devitrification problems made the calcium aluminate fibers "brashy", brittle, and rough. It is not expected that any known "tip environment" control will eliminate this devitrification problem.

A small amount of fiberized glass was obtained from the cullet containing beryllium oxide. This was smooth in texture and had a good appearance. Pure powdered beryllium oxide is extremely toxic and studies were made after this substance was incorporated into the glass melt to determine if such toxicity remained a hazard when the cullet was remelted. It proved to be no hazard although in the interests of good precautionary measures, a hood for optimum fume control is recommended.

In the initial evaluation program, six (6) different bushings were used incorporating eight (8) different tip designs or

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modifications in an effort to fiberize calcium aluminate glass cullet. Results were generally unsuccessful.

In the subsequent (Supplemental Agreement) evaluation regarding beryllium oxide glass cullet, one (1) bushing incorporating two (2) different tip designs was used in the attempted fiberization. Results were generally successful.

WADC TR 56-270  
ASTIA Document No. AD 118324  
OTS Release

May 1957

**SUBJECT:** INVESTIGATION OF THE EFFECTS OF GLASS FABRIC GEOMETRY ON THE STRENGTH PROPERTIES OF LOW PRESSURE GLASS FABRIC BASE STRUCTURAL LAMINATES

**INVESTIGATOR:** W. R. Bowditch  
E. L. Johnson

**CONTRACT:** AF 33(616)-2543

**CONTRACTOR:** Taylor Fibre Co.

**ABSTRACT:** This report describes work done to develop glass fabrics which make stronger plastic laminates than are produced by the glass fabrics which are commercially available today.

In the initial evaluation program, seventeen (17) experimental fabrics and two (2) high modulus fabrics were compared to four (4) standard glass fabrics in laminate form. Five (5) experimental fabrics were selected for further evaluation after improvements were made in crimp, twist, fiber diameter and weave. Based on data obtained, further consideration and study should be made to determine the practicality of

- a. Using Final Fabric S-14619-1
- b. Using the original HM-9 fabric or the HM-9 fabric made from ECK Filaments
- c. Using the HM-18 (original) or the HM-18 fabric made from ECK filaments
- d. Using a 143 type fabric with ECG or ECK filaments in the warp direction.

WADC TR 56-534 Pt II  
ASTIA Document No. AD 130838

June 1957

**SUBJECT:** NUCLEAR RADIATION EFFECTS ON STRUCTURAL PLASTICS AND ADHESIVES  
Part II Extension of Literature Survey

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INVESTIGATOR: R. Y. Mixer  
Shirley B. Radding  
CONTRACT: AF 33(616)-3632  
CONTRACTOR: Stanford Research Institute  
ABSTRACT: A survey was made of literature on radiation chemistry available during the period 30 September 1956 to 1 February 1957. Information pertinent to the effect of radiation on the syntheses and the properties of polymers, elastomers, and adhesives was tabulated. A brief summary of the more general observations on the effects of radiation on these materials was prepared.

The literature available prior to 30 September 1956 was covered in Part I.

WADC TR 56-534 Pt III  
ASTIA Document No. AD 131029

August 1957

SUBJECT: NUCLEAR RADIATION EFFECTS ON STRUCTURAL PLASTICS  
AND ADHESIVES  
Part III Experimental Research  
INVESTIGATOR: R. Y. Mixer  
D. B. Parkinson  
CONTRACT: AF 33(616)-3632  
CONTRACTOR: Stanford Research Institute  
ABSTRACT: Nuclear radiation damage mechanisms are presented for model compounds representing several typical aircraft structural adhesives and laminates. Laminates were prepared from eight resin-curing agent systems and irradiated to  $10^9$  to  $10^{10}$  rep for determination of the threshold dose for damage. The threshold dose for one system was found to be less than  $10^8$  rep. In the case of typical amine-cured, epoxy-type adhesives, radiation appears to cleave the amine groups with volatilization of some fragments and to crosslink the polymer chain. Small amounts of chemical changes result in large changes in physical properties of the cured adhesive. Beta and gamma irradiation have produced equal effects on one vinyl-phenolic and one epoxy-type adhesive. One organic scintillator, 2, 5-diphenyloxazole, appears to be partially effective in preventing irradiation damage in an epoxy type adhesive.

WADC TR 57-200  
ASTIA Document No. AD 131041

September 1957

SUBJECT: ASBESTOS REINFORCED LAMINATES  
INVESTIGATOR: Norman E. Wahl  
Harold M. Preston

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CONTRACT: AF 33(616)-2926  
CONTRACTOR: Cornell Aeronautical Laboratory  
ABSTRACT: The use of long fibered asbestos as a reinforcement for typical polyester, epoxy, silicone and phenolic resins was investigated. Low pressure laminates were prepared and the physical properties were determined.

It was found that the molding pressure and the resin content of asbestos laminates had a considerable influence on their ultimate physical properties. Asbestos reinforced laminates molded at 15, 30 and 60 psi had considerably lower mechanical properties than glass reinforced laminates molded at the same pressure with the same resin. Molding pressures from 200-800 psi and resin contents of 28-38 percent by weight of the asbestos laminate are required to achieve physical properties that are comparable to glass cloth reinforced laminates. Of the various types of resins investigated, the phenolic resins with asbestos felts resulted in laminates with the most reproducible and the highest physical properties at room temperature. However at elevated temperatures the silicone resins with the asbestos gave the highest strengths. After exposure to temperatures of 700° and 800°F for 192 hours and tested at 700° or 800°F, these silicone asbestos laminates generally retained 50% or more of their room temperature strength. For applications where plastic laminates must be used at temperatures of 700° or 800°F, the silicone asbestos laminates merit consideration.

It was observed that the resin content of an asbestos laminate could not be lowered to any great degree by increasing the pressure during molding, as can usually be done with laminates using glass as a reinforcement. If lower resin contents are required, it is necessary to treat the asbestos by various methods such as the use of a solvent diluted resin with subsequent removal of the solvent in an oven or drying tower with the proper amount of heat.

The final properties of asbestos laminates depend on many factors such as fiber treatment, asbestos binders, resin content, flow and gel time of the resin, layup of the plies, molding temperature pressure and posture. It is apparent from this study that a great deal has to be done to develop a technique of impregnating and molding asbestos felt in order to obtain higher physical properties with various types of resins.

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WADC TR 57-246  
ASTIA Document No. AD 130881

July 1957

**SUBJECT:** ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF VIBRIN  
135 GLASS FABRIC-PLASTIC LAMINATE

**INVESTIGATOR:** Gilbert M. Gynn  
John A. Vanecho  
Ward F. Simmons

**CONTRACT:** AF 33(616)-3215

**CONTRACTOR:** Battelle Memorial Institute

**ABSTRACT:** Basic engineering data have been obtained for the benefit of airframe designers on a glass-fabric laminate containing Vibrin 135, a polyestertriallyl cyanurate (TAC) resin. These data include results of creep and creep-rupture in both tension and compression, flexural, deterioration, and short-time tensile and compression tests.

The strength properties of Vibrin 135 were found to be dependent upon both the temperature and the length of time the material was exposed to a given temperature. The test results indicate that the laminate is relatively strong and stable for exposures to 1000 hours at low temperatures (80, 300, and 400 F). At higher temperatures, however, the strength of Vibrin 135 decreased rapidly with increases in either temperature or exposure time.

The results reported are for particular and specified panels of the test material, and should not be considered as precise design data for all panels of Vibrin 135 laminate.

WADC TR 57-352  
ASTIA Document No. AD 142065

October 1957

**SUBJECT:** EVALUATION OF A VARIATION IN GLASS COMPOSITION OF  
GLASS FIBERS FOR PLASTIC LAMINATES

**INVESTIGATOR:** G. P. Peterson

**ABSTRACT:** A variation of "E" type glass, designated 1145 "E" glass was developed by Owens-Corning Fiberglas Corporation to replace the standard "E" type glass designated 621 "E" glass for use in glass fibers for glass fiber reinforced plastic laminates. The evaluation program included the fabrication and evaluation of laminates with: (1) 181 style glass cloth woven from both types of glass fiber, (2) Owens-Corning 136 and 139 finishes on each type of glass fiber, (3) typical polyester (Paraplex P-43) and heat resistant polyester (Vibrin X-1068) resins. Room temperature, elevated temperature, chemical immersion, water immersion, fatigue and electrical tests were conducted to provide a comparison of the properties of laminates made from the

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two glasses. Electrical tests were also made on bulk glass samples of the two glasses. The 1145 "E" glass fiber was satisfactory and equivalent to the standard 621 "E" type glass fiber in all respects except for dielectric constant properties. At room temperature the dielectric constant at 8.5 KMC of the 1145 "E" glass was somewhat higher than that of 621 "E" glass, which is undesirable, but within acceptable limits. However, the dielectric constant at 8.5 KMC of the 1145 "E" glass increased far more rapidly with increasing temperatures than that of 621 "E" glass, and to an unacceptable extent. Consequently the 1145 "E" glass would not be satisfactory for radomes for radar guidance control purposes. Since the proposed alternate composition is not acceptably equivalent to the 621 "E" glass, the 1145 "E" glass is not considered satisfactory for Air Force reinforced plastic applications.

WADC TR 57-531  
ASTIA Document No. AD 142136

November 1957

SUBJECT: ROOM- AND ELEVATED-TEMPERATURE STRENGTH PROPERTIES  
OF BAKELITE BV-17085 GLASS FABRIC-PLASTIC LAMINATE

INVESTIGATOR: Gilbert M. Gynn  
John A. Vanecho  
Ward F. Simmons

CONTRACT: AF 33(616)-3215

CONTRACTOR: Battelle Memorial Institute

ABSTRACT: Basic design data including creep and creep-rupture, flexural, deterioration, and short-time tensile and compression properties were obtained for Bakelite BV-17085, a phenolic resin-glass-fabric laminate. The creep and creep-rupture data were obtained at 80, 300, and 500 F with rupture times ranging from about zero to 1000 hours. The other properties were determined at 80 to 1000 F with prior exposures at the test temperatures for times ranging from 0 to 1000 hours. The creep and creep-rupture specimens were not exposed to elevated temperatures prior to testing.

The over-all strength properties of BV-17085 are relatively good with useful load-carrying ability as high as 1000 F for time periods of less than about 1 hour. The short-time tensile strength of the material seems to be largely independent of test temperature or prior exposure within limits of the test conditions. The short-time compression, creep-rupture, and flexural strength properties, on the other hand, vary to a large degree with both test temperatures and prior exposure or exposure during test.

Stability of the test material, as determined by exposure at elevated temperatures for various periods of time, is quite good at 300 and 400 F. At higher temperatures, deterioration of the laminate is directly related to the temperature and time of exposure.

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The test data discussed in this report should be considered only as property trends because of the wide degree of scatter generally found in the property values of glass-fabric laminates including BV-17085.

WADC TR 57-574  
ASTIA Document No. AD 142190

December 1957

**SUBJECT:** ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF CONOLON  
506 PLASTIC-GLASS FABRIC LAMINATE

**INVESTIGATOR:** Gilbert M. Gynn  
John A. Vanecho  
Ward F. Simmons

**CONTRACT:** AF 33(616)-3215

**CONTRACTOR:** Battelle Memorial Institute

**ABSTRACT:** Basic engineering properties for Conolon 506, a phenolic resin-glass fabric laminate, are presented in this report. The data included were obtained from creep and creep-rupture, flexural, deterioration, and short-time tension and compression tests. The tests were conducted at temperatures ranging from 80 to 1000 F with prior exposures at these temperatures ranging from about 0 to 1000 hours.

In general, the creep-rupture strength was found to be greater in tension than in compression. The minimum creep rates were found to be relatively low since very little plastic strain was measured after loading. The flexural and short-time tensile and compressive strengths, and flexural and tensile elastic moduli were deleteriously affected by increases in either temperature or exposure time. Also, under the conditions of this evaluation, the short-time tensile strength exceeded the short-time compressive strength. The results of this evaluation merely indicate property trends and should not be considered as precise design data.

WADC TR 57-638 Pt I  
ASTIA Document No. AD 142181

December 1957

**SUBJECT:** EFFECTS OF HIGH INTENSITY THERMAL RADIATION ON  
STRUCTURAL PLASTIC LAMINATES

**INVESTIGATOR:** Herbert S. Schwartz  
Ben J. Lisle

**ABSTRACT:** Polyester, silicone and phenolic glass-fabric-base plastic laminates were exposed to thermal radiant energy up to an intensity of 25 cal/cm<sup>2</sup>-second, with and without mechanical stress, to determine their resistance to mechanical and physical degradation.

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Absorptivities of thermal radiant energy from a 6000°K source were computed from spectral reflectance and transmittance data measured over the region from 0.4 to 2.0 microns.

The resistance of the laminates to thermal radiation measured by degradation of mechanical properties was found to be a function of absorptivity, heat resistance of the laminating resin, and stress applied. Based on mechanical property considerations, the optimum material for resisting the effects of high intensity radiant energy and moderate stresses was found to be the silicone laminate, and for low intensities over a wide range of stresses was found to be the phenolic laminate.

WADC TR 57-674  
ASTIA Document No. AD 150972

February 1958

**SUBJECT:** THE EFFECTS OF HYDRAULIC FLUIDS ON THE PROPERTIES OF GLASS FIBER REINFORCED PLASTIC LAMINATES  
**INVESTIGATOR:** D. F. Starks  
**ABSTRACT:** The object of this investigation was to provide a wider range of mechanical strength and physical property information on the effects of immersion on some reinforced plastic materials in chemical types of fluids being considered for future use at the projected use temperatures. Weight and thickness change determinations were conducted on the flexural specimens of the five (5) plastic materials utilized, with four (4) experimental hydraulic fluids. Flexural, tensile and compressive strength tests were conducted both before and after immersion.

The various immersion conditions utilized for the materials were as follows: 1) 24 hours at room temperature, 2) 7 days at room temperature, 3) 24 hours at 160°F for Paraplex P-43 polyester only, 4) 24 hours at 250°F for the heat resistant materials, 5) 24 hours at 400°F in OS-45 silicate, and 6) 24 hours at 500°F in F-50 silicone. The immersions at 250°F, 400°F and 500°F were conducted utilizing the heat resistant materials only.

Immersion in the high temperature fluids under the various conditions used had negligible effect on the mechanical properties of the plastic materials. The room temperature immersion in the fluids had negligible effect on the weight and thickness physical properties, however, variations in weight and thickness were determined from the elevated temperature immersions at 400 F and 500 F, particularly.

WADC TR 58-27  
ASTIA Document No. AD 151091

March 1958

**SUBJECT:** MECHANICAL PROPERTIES OF PLASTIC LAMINATES REINFORCED WITH NOL 24 - FINISHED GLASS FABRIC

**INVESTIGATOR:** Kenneth E. Kimball

**CONTRACT:** DO 33(616)56-9

**CONTRACTOR:** Forest Products Laboratory

**ABSTRACT:** This report presents the results of tests of three parallel-laminated, glass-fabric-base, plastic laminates in which the glass fibers had been treated with a universal type of chemical finish. The finish was developed by the Naval Ordnance Laboratory and is designated as NOL 24. This finish was tested with a 181 glass fabric and polyester, epoxy, or phenolic resin.

The laminates were tested in tension, compression, and flexure after normal and wet conditioning, with some tests at elevated temperatures. Data are presented both in tables and as typical curves.

Also included is a table of comparative data showing how the laminates reinforced with NOL 24-finished fabric compared with similar laminates reinforced with fabric having other finishes.

## POLYMERS AND SYNTHESIS STUDIES

WADC TR 54-613 Pt II  
ASTIA Document No. AD 130761  
OTS Release

May 1957

**SUBJECT:** ORGANOSILICON COMPOUNDS  
Part II - The Cyclopentamethylenedialkylsilanes

**INVESTIGATOR:** Dr. Harold Rosenberg  
Dr. Christ Tamborski  
Marvin D. Rausch, 1/Lt

**ABSTRACT:** An investigation into the use of cyclic tetra-substituted organo-silanes was conducted to determine their applicability as base materials for extreme-temperature lubricant and hydraulic fluid applications. A series of cyclopentamethylenedialkylsilanes was synthesized and certain of the physical properties correlated with molecular structure. In addition, one aryl derivative, cyclopentamethylenediphenylsilane, was prepared and evaluated. The cyclopentamethylenedialkylsilanes were found, similar to the unsymmetrical

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tetraalkylsilanes, to be liquid over a wide temperature range and, in the case of certain members, to be thermally stable at elevated temperatures. These compounds, on the basis of preliminary data, appear to show promise as base materials for certain wide-temperature hydraulic fluid and lubricant applications.

WADC TR 54-613 Pt III  
ASTIA Document No. AD 131066  
OTS Release

September 1957

SUBJECT: ORGANOSILANES AND RELATED COMPOUNDS AS HIGH-TEMPERATURE LUBRICANTS  
Part III. Mixed Symmetrical Tetraalkylsilanes  
INVESTIGATOR: Dr. Harold Rosenberg  
Dr. Christ Tamborski  
James D. Groves, 1/Lt

ABSTRACT: One class of mixed symmetrical tetraalkylsilanes was investigated to determine their applicability as base materials for Air Force high-temperature hydraulic fluids and lubricants. A series of di-n-dodecyldialkylsilanes was prepared and various physical properties of the fluids obtained were correlated with molecular structure. These materials were found to be thermally stable at elevated temperatures and liquid over a wide temperature range. In addition to the tetraalkyl compounds, one aralkyl derivative, di-n-dodecyldiphenylsilane, was also synthesized. On the basis of preliminary property data, this fluid and at least several mixed symmetrical tetraalkylsilanes offer considerable promise as base stocks for 700°F lubricant and hydraulic fluid applications in new advanced air weapon systems.

WADC TR 55-220 Pt III  
ASTIA Document No. AD 131044  
OTS Release

September 1957

SUBJECT: DEVELOPMENT OF FLUORO-SILICONE ELASTOMERS  
INVESTIGATOR: George W. Dyckes  
CONTRACT: AF 33(616)-3238  
CONTRACTOR: Peninsular Chemresearch, Inc.  
ABSTRACT: Some promising fluoroalkyl silane monomers and polymers have been prepared for use as high temperature and aircraft fuel resistant elastomeric materials.

Appreciable quantities of the monomers,  $\text{CF}_3\text{CH}_2\text{CH}_2\text{Si}(\text{CH}_3)\text{Cl}_2$ ,  $\text{CH}_3\text{CH}(\text{CF}_3)\text{CH}_2\text{Si}(\text{CH}_3)\text{Cl}_2$ ,  $\text{CF}_3\text{CF}_2\text{CH}_2\text{CH}_2\text{Si}(\text{CH}_3)\text{Cl}_2$  and  $\text{CF}_3 - \text{CF}_2\text{CF}_2 - \text{CH}_2\text{CH}_2\text{Si}(\text{CH}_3)\text{Cl}_2$  were synthesized for polymerization studies. Hydrolysis reactions were carried out on the monomers and the constant boiling

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products have been characterized. Polymerization of the cyclic hydrolyses products to elastic homopolymers has been realized using the active metals in Group IA of the periodic table as polymerization catalysts. Copolymers with octamethylcyclotetrasiloxane were also prepared using potassium hydroxide as a catalyst. An attempt to prepare a high molecular weight ternary polymer from the trimer,  $[\text{CF}_3\text{CH}_2\text{CH}_2\text{Si}(\text{CH}_3)\text{O}]_3$ , and the cyclic tetramers,  $[(\text{CH}_3)_2\text{SiO}]_4$  and  $[\text{CH}_2=\text{CHSi}(\text{CH}_3)\text{O}]_4$ , gave only a medium viscosity oil.

A study was made of the reaction of hydrogen fluoride with the compound,  $\text{CF}_2\text{BrCH}_2\text{CH}_2\text{Br}$  in attempt to improve the yield of the fluorinated product,  $\text{CF}_3\text{CH}_2\text{CH}_2\text{Br}$ . Improved yields were obtained; however, percent conversion still remains relatively low.

In the preparation of new fluoroalkyl silane monomers, preliminary experiments were carried out on the fluorination of the beta carbon atom on the compounds  $\text{CCl}_2=\text{CHSi}(\text{CH}_3)_3$  and  $\text{CHCl}=\text{CHSi}(\text{CH}_3)_3$ . Analytical results indicate the fluorinated compounds were obtained.

WADC TR 55-220 Pt IV  
ASTIA Document No. AD 151180

April 1958

SUBJECT: DEVELOPMENT OF FLUORO-SILICONE ELASTOMERS  
INVESTIGATOR: Byron H. Wise  
CONTRACT: AF 33(616)-3238  
CONTRACTOR: Peninsular Chemresearch, Inc.  
ABSTRACT: Some promising fluoroalkylsilane monomers and polymers have been prepared for use as high temperature and aircraft fuel resistant elastomeric materials.

In addition to the halosilanes previously synthesized, the following new silanes were prepared:

Bis(trifluoropropyl)dichlorosilane

(Chlorohexafluorocyclobutylethyl)-methylchlorosilane

(2-Trifluoromethyltrifluoropropyl)-methylchlorosilane.

Polymerization of the cyclic polymers of (pentafluorobutyl)-methylsiloxane and (heptafluoropentyl)methylsiloxane was attempted, but resulted in oils of lower viscosity than desired. Polymerization of the cyclic polymers of (trifluoropropyl)methylsiloxane yielded high viscosity oils and in one case an elastic gum was formed.

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WADC TR 55-221 Pt III  
ASTIA Document No. AD 118294  
OTS Release

May 1957

**SUBJECT:** INVESTIGATION OF CONDENSATION TYPE ELASTOMERS  
**INVESTIGATOR:** George C. Schweiker      Russell R. White  
                  Burton S. Marks             Rudolph N. Deleo  
**CONTRACT:** AF 33(616)-2421  
**CONTRACTOR:** Hooker Electrochemical Company  
**ABSTRACT:** The ultimate goal of the exploratory investigations described is the development of a rubber for special Air Force applications. High thermal stability (350°F or higher); resistance to aromatic fuels, synthetic esterbase oils and hydraulic fluids; resistance to ozone and to weathering oxidation effects; resistance to acids, bases, and salts; resistance to abrasion; and satisfactory performance at -65°F or lower are major requirements for such an elastomer.

Preliminary compounding and testing studies with an elastomer based on a fluorine-containing polyester, made from adipyl chloride and 2,2,3,3,4,4-hexafluoropentanediol, have given promising results. At the present stage of development, compounded and cross-linked specimens of hexafluoropentylene adipate retain useful mechanical properties after aging in air at 400°F (70 and 168 hour tests), and in diester oil at 350°F (70 hour test); are resistant to paraffinic and aromatic fuels; and exhibit a brittle temperature of -98°F (ASTM D-746).

This report describes the preparation, compounding, cross-linking and properties of hexafluoropentylene adipate elastomer as well as syntheses and properties of other fluorine-containing condensation polymers and difunctional starting materials. Data indicating that increasing fluorine content of the polymers decreases their solubility in common solvents without regard to position of the fluorine in the polymer are extended. On the other hand, these data show that position of the fluorine, rather than total fluorine content of the polymer is of most importance in affecting brittle temperatures.

WADC TR 55-221 Pt IV  
ASTIA Document No. AD 151009  
OTS Release

February 1958

**SUBJECT:** INVESTIGATION OF CONDENSATION TYPE ELASTOMERS  
**INVESTIGATOR:** George C. Schweiker      Elaine L. Lucas  
                  Burton S. Marks             Edward V. Gouinlock  
                  Carl J. Verbanic  
**CONTRACT:** AF33(616)-2421  
**CONTRACTOR:** Hooker Electrochemical Company

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**ABSTRACT:** The ultimate goal of the exploratory investigations described is the development of a rubber for special Air Force applications. Major requirements for such a material include good mechanical properties, high thermal stability (originally 350°F or higher), satisfactory performance at -65°F or lower, and resistance to aromatic fuels, synthetic ester-base oils, and hydraulic fluids.

To this end, fluorine-containing condensation polymers are being investigated in an effort to discover and develop suitable new elastomers.

This report describes the preparation, compounding, cross-linking, and properties of certain fluorine-containing polyester elastomers which appear to meet the goals outlined above. The report also describes the research performed on the synthesis of fluorine-containing difunctional starting materials, polyesters and nitrogen-substituted polyamides therefrom, and their characterization.

WADC TR 55-453 Pt II  
ASTIA Document No. AD 131036  
OTS Release

September 1957

**SUBJECT:** DEVELOPMENT OF INORGANIC POLYMER SYSTEMS  
**INVESTIGATOR:** Carlin F. Gibbs                      George Shkapenko  
                    Harold Tucker                      John C. Park  
**CONTRACT:** AF33(616)-2744  
**CONTRACTOR:** The B. F. Goodrich Company  
**ABSTRACT:** The purpose of this investigation was to prepare polymeric inorganic or semi-inorganic materials capable of withstanding elevated temperatures.

The investigation of the aluminum-oxygen-silicon system initiated last year has been continued through this year. Previous difficulties in formation of aluminum-oxygen-silicon bonds were traced to exchange reactions. Reaction of aluminum halides with sodium silanates has been found to lead to formation of aluminum-oxygen-silicon bonds in high yields with no evidence of exchange reactions. By this means, tris(triphenyl)siloxyaluminum and tris(trimethyl)siloxyaluminum have been prepared. Attempts to extend this principle to the preparation of polymers using difunctional aluminum halides and silanol salts have not resulted in formation of high polymers, but only polymers very low in molecular weight.

Preparation of trimethylsiloxyaluminum diisopropoxide in high yield by reaction of aluminum isopropoxide with trimethylace-

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toxysilane indicates that this type of reaction might also be a good one for the establishment of aluminum-oxygen-silicon bonds.

A rubbery tin-silicon polymer containing 8-10% tin was stable upon being heated at 300°C for three hours.

WADC TR 56-82 Pt II  
ASTIA Document No. AD 118301  
OTS Release

May 1957

SUBJECT: PRINCIPLES OF INORGANIC POLYMERIZATION  
INVESTIGATOR: Anton B. Burg Gordon L. Juvinall  
Gottfried Brendel Walter Mahler  
Aimery P. Caron Kurt Modritzner  
Peter J. Slota, Jr.

CONTRACT: AF 33(616)-2743  
CONTRACTOR: University of Southern California  
ABSTRACT: This project continues to seek and to find new patterns of chemical bonding and to consider their possible usefulness in relation to the invention of new polymers having unusual thermal stability. Some of our amino-phosphino-boron resins are stable at temperatures approaching 500°C., and the limit is not yet known; however the problem of their poor mechanical properties remains. The phosphinoborine bonding principle withstands a great increase in the electronegativity of substituents on phosphorus. The stability of P-P bonding seems much enhanced by CF<sub>3</sub> groups on P, so that the new compounds (PCF<sub>3</sub>)<sub>4</sub> and (PCF<sub>3</sub>)<sub>5</sub> are decidedly stable. The previous tentative interpretations of the by-products of the fluorocarbonphosphorus iodide reactions with mercury are reconsidered in the light of new facts. Progress toward more electronegative substituents on boron is reported, and our recently initiated work on phosphinoaluminum compounds has produced some interesting materials. Nitrilophosphoric chloride is not depolymerized by pyridine, but it reacts with triethylamine in an interesting manner.

WADC TR 56-82 Pt III  
ASTIA Document No. AD 151175

April 1958

SUBJECT: PRINCIPLES OF INORGANIC POLYMERIZATION  
INVESTIGATOR: Anton B. Burg Gordon L. Juvinall  
Gottfried Brendel Walter Mahler  
Aimery P. Caron Kurt Modritzner  
Peter J. Slota, Jr.

CONTRACT: AF33(616)-2743  
CONTRACTOR: University of Southern California

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**ABSTRACT:** The work of the past year has been devoted largely to the search for new substances which might seem promising as components of inorganic polymer systems, and to a study of their chemical bonding properties. The aminophosphine  $(\text{CH}_3)_2\text{NP}(\text{CH}_3)_2$  has been converted to  $(\text{CH}_3)_2\text{PCL}$  and to the new diphosphine  $\text{P}_2(\text{CH}_3)_4$ . The latter was used to make a  $(\text{CH}_3)_2\text{PBH}_2$ -chain polymer and a somewhat improved thermally stable resin. A new interpretation of such resins extends earlier ideas. The phosphino-aluminum system has been explored, indicating new-type compounds of some interest. A considerable further study of fluorocarbon-phosphorus compounds included a demonstration of  $\text{CF}_3\text{P}$  higher polymers, the  $(\text{CF}_3\text{PO}_2)_n$  system, and  $(\text{CF}_3\text{P})_n$ -nickel-carbonyl polymers. The synthesis of  $(\text{CF}_3)_2\text{PBH}_2$  trimer and tetramer has been studied thoroughly, with recognition of interesting by-products. The  $(\text{CH}_3)_2\text{N}-(\text{NPCL}_2)_3$  reaction has been shown to split the amine to form  $(\text{CH}_3)_2\text{N}-\text{P}$  derivatives. An acetylide derivative of  $(\text{NPCL}_2)_3$  formed a higher polymer. Work with  $(\text{NPF}_2)_3$  has begun. The interesting unstable product of the  $\text{CF}_3\text{SCL}-\text{B}_2\text{H}_6$  reaction may prove to be  $\text{CF}_3\text{BF}_2$ .

WADC TR 56-203 Pt II  
ASTIA Document No. AD 142191  
OTS Release

December 1957

**SUBJECT:** COORDINATION POLYMERS  
**INVESTIGATOR:** W. Conrad Fernelius                      David E. Goldberg  
                  Maurice Shamma                              Dean E. Martin  
                  Norman R. Garofano                         Forrest D. Thomas, III  
**CONTRACT:** AF 33(616)-2742  
**CONTRACTOR:** The Pennsylvania State University  
**ABSTRACT:** Various approaches towards the development of polymers through coordination have been investigated. Several bis( $\beta$ -diketones) have been prepared; this work will now be followed by attempts at their polymerization.

Attempts to prepare a silicon monomer by the reaction of dimethyldichlorosilane with the sodium salt of acetylacetonone were unsuccessful, the product formed being unstable. On the other hand, a series of monomeric bis(acetylacetononeate)dialkoxy titanium compounds were synthesized.

Investigations on the coordination ability of organic phosphorus radicals have shown that they do not coordinate easily. Dithiophosphates, on the other hand, coordinate well. A series of salts of dithiophosphates has been prepared, and efforts have been made to prepare an analogous bifunctional ligand. Polymeric materials have been produced from what probably is a mixed ester of a dialcohol and monoalcohol. These dithio compounds have low thermal stability.

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Investigations of similar compounds with other donor atoms led to the conclusion that compounds with various combinations of oxygen, sulfur, amido nitrogen, and substituted amido nitrogen do not coordinate well, except for the dithio compounds already mentioned. Because of little hope of producing high temperature polymers, this phase of the problem has been abandoned.

Efforts towards the formation of coordination polymers from bis(*o*-hydroxyazo)compounds did not yield promising results because of the insolubility of the products formed.

A series of *o*-hydroxyketones was prepared by means of the Fries Rearrangement preliminary to the synthesis of bis(*o*-hydroxyketones).

WADC TR 56-376  
ASTIA Document No. AD 130798  
OTS Release

June 1957

**SUBJECT:** DEVELOPMENT OF THERMALLY STABLE POLYMERIC MATERIALS  
**INVESTIGATOR:** Charles P. Haber  
**CONTRACT:** AF 33(616)-56-41  
**CONTRACTOR:** U. S. Naval Ordnance Laboratory  
**ABSTRACT:** The work described within this report has as its objective the synthesis of polymeric materials having extreme chemical and thermal stability which are suitable for use in various aircraft applications such as elastomers, plastics, fluids, dielectrics, and adhesives. In most applications the needed materials must be able to resist the action of the atmosphere at the elevated temperatures; in some, resistance to corrosive fuels and fuel combustion products is required. The task of this program is to investigate promising inorganic bonding systems for thermal and chemical resistance and to incorporate these bonding systems into high polymer molecules with the required physical and mechanical properties.

Diphenylvinylsilane has been prepared in good yields. The polymerization of this material by peroxide-initiated reactions in bulk, solution, and emulsion has led only to low molecular weight, low melting solids of no apparent practical significance. The mechanism of polymerization has been established as the addition of the elements of Si-H bond of one molecule across the vinyl unsaturation of another molecule. Catalysis of the polymerization by a platinum-on-carbon catalyst led to a different type of reaction and a dissimilar product tentatively assigned the structure  $\phi_2C_2H_3-Si-SiC_2H_3\phi_2$ .

In an attempt to prepare  $(CF_2)_2$ PN polymers, a multi-step synthesis giving an excellent overall yield of the desired starting material,  $(CF_3)_2PCl_3$ , was developed. The reaction of  $(CF_3)_2PCl_3$  with

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ammonia and ammonium chloride under a wide variety of experimental conditions led to rearrangement, disproportionation, and ammonolysis of  $CF_3$  groups. As a result of these side reactions, it has not been possible to prepare the desired nitride polymer.

WADC TR 56-520  
ASTIA Document No. AD 118247  
OTS Release

August 1957

**SUBJECT:** COPOLYMERS OF 1, 1-DIHYDROPERFLUOROBUTYL ACRYLATE WITH SOME VINYL SILANES  
**INVESTIGATOR:** Eugene C. Stump, Jr., 1/Lt  
**ABSTRACT:** The purpose of this investigation was to prepare an elastomer with high temperature resistance as well as improved resistance to the deteriorating and swelling effects of fuels and oils.

Four silane monomers; vinyltriethoxy-, vinylethyl-diethoxy-, vinyldimethylethoxy-, and vinyltrimethylsilane were copolymerized with 1,1-dihydroperfluorobutyl acrylate. The resulting polymers were tested for thermal stability by determining percent weight loss in dry air at 350°F and 400°F. In most cases weight loss of the silane containing polymers was appreciably less than that of the homopolymer poly-1,1-dihydroperfluorobutylacrylate. The enhanced thermal stability is attributed to a unique type of cross-linking, which is discussed theoretically.

Infrared spectra of the samples were recorded and discussed. Results of compounding, testing, and evaluation will be reported at a later date.

WADC TR 56-590 Pt I  
ASTIA Document No. AD 142171  
OTS Release

November 1957

**SUBJECT:** PHOTOCHEMICAL SYNTHESIS OF ORGANIC FLUORINE COMPOUNDS  
**INVESTIGATOR:** Joseph D. Park  
John R. Lacher  
**CONTRACT:** AF 33(616)-3266  
**CONTRACTOR:** University of Colorado  
**ABSTRACT:** The purpose of this research work has been to synthesize organic and organo-metallic fluorine compounds with the major objective of obtaining monomers suitable for polymerization into elastomers, plastics, fluids and related material of high thermal and chemical stability. In conjunction with these aims, this Laboratory has synthesized two halogenated pentadiene.

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Several important four-membered fluorinated ring compounds have been synthesized and their cyclic ethers. To furnish adequate proof of structure and determine the position of unsaturation, the compounds have been halogenated.

In the work on alcohols both 1,2-difluoro-2-chloroallyl alcohol and 1,1-difluoro-2-bromoallyl alcohol have been synthesized, the first reported synthesis of a fluorinated allyl alcohol obtainable directly from a Grignard reagent. However, synthesis of trifluoroallyl alcohol has not been achieved.

Attempts which have been made to synthesize monohydric and polyhydric alcohols indicate that the proposed routes are feasible. Work in this area has not been completed.

A variety of methods have been used in attempts to prepare alpha, beta-unsaturated ketones (Grignard, free-radical, preparation of alkyl lithium derivatives, Friedel-Crafts-type reactions, and preparation of an unsaturated acid) without success. Several of the ketones derived from the addition of a Grignard reagent to Kel-F Acids have been prepared. As intermediates to the preparation of ketones, two alkynes have been made.

WADC TR 56-590 Pt II  
ASTIA Document No. AD 151014  
OTS Release

March 1958

SUBJECT: PHOTOCHEMICAL SYNTHESIS OF ORGANIC FLUORINE COMPOUNDS  
INVESTIGATOR: Joseph D. Park  
John R. Lacher  
CONTRACT: AF 33(616)-3266  
CONTRACTOR: University of Colorado  
ABSTRACT: The purpose of this research work has been to synthesize organic and organometallic fluorine compounds with the major objective of obtaining monomers suitable for polymerization into elastomers, plastics, fluids and related material of high thermal and chemical stability. In conjunction with these aims, this Laboratory has synthesized a number of diols and cyclic fluorosilanes. At the present time, however, the structures assigned to several of the synthesized fluorosilanes have not been verified.

Further work in the preparations of cyclic diols involving the dimerization reaction of fluoroolefins with alkenes containing functional groups has produced intermediates with interesting fluorescent properties.

Initial investigation has been made of obtaining a monomeric unit with fluorine substitution on the aromatic nucleus.

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WADC TR 56-596 Pt II  
ASTIA Document No. AD 142212

December 1957

**SUBJECT:** RESEARCH ON HIGH TEMPERATURE LAMINATING RESINS BASED ON MALEIMIDE AND ITS DERIVATIVES  
**INVESTIGATOR:** Roger W. Amidon  
Charles H. Alexander  
**CONTRACT:** AF 33(616)-3606  
**CONTRACTOR:** Naugatuck Chemical  
**ABSTRACT:** The object of this contract was the preparation of laminating resins for service in the range of 600° to 700°F based primarily on maleimide or derivatives thereof.

Resins were evaluated by preparing from them a Fiberglas cloth laminate and exposing cured test pieces to elevated temperature. Quality was judged by retention of flexural strength of exposed samples.

A VIBRIN 135 type system which contained 25% maleimide showed superior modulus under unaged and aged conditions. Flexural strength values after 192 hours exposure at 500°F have been as high as 38,000 psi.

New alkyds have been prepared and evaluated with triallyl cyanurate monomer. None investigated showed outstanding properties when compared to VIBRIN 135.

Several N-substituted maleimide compounds were investigated with a VIBRIN 135 alkyd and triallyl cyanurate. The strength of aged pieces did not compare well with maleimide resins.

Triallyl isocyanurate, triallyloxybenzene maleimido-methyl acrylate and N-allyloxymethyl maleimide were synthesized and screened as copolymerizable monomeric materials.

Effort was made to gain some insight into the cause and minimization of interlaminar blistering. A low acid number polyester reduced blistering somewhat. Blistering was not experienced with non-nitrogenous monomers such as diallyl *o*-phthalate or styrene.

WADC TR 57-45 Pt I  
ASTIA Document No. AD 131099

September 1957

**SUBJECT:** HIGH TEMPERATURE STABLE SEMIORGANIC FLUIDS  
**INVESTIGATOR:** Daniel Grafstein  
Rita Dudak  
**CONTRACT:** AF 33(616)-3417  
Murray S. Cohen  
Joseph Green

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CONTRACTOR: Reaction Motors, Inc.  
ABSTRACT: Phenylchlorodichlorosilane and methylchlorodichlorosilane, monomeric materials suitable for the preparation of polyorganosiloxane polymers, were prepared in good yield and good conversion by the reaction of gaseous boron trifluoride with phenylchloroethoxysilane and methylchloroethoxysilane. Phenylmethylchlorodichlorosilane and phenylmethylfluoroethoxysilane were synthesized and evaluated as chain stopping agents for the organosiloxane polymers. The polymerization of these monomers were studied and the liquid polymers obtained were evaluated for potential use as high temperature base stock fluids. The relationship of physical properties to structural features of the polymeric oils was established.

WADC TR 57-45 Pt II  
ASTIA Document No. AD 142290

January 1958

SUBJECT: HIGH-TEMPERATURE STABLE SEMIORGANIC FLUIDS  
Part II. Organophosphorus Compounds  
INVESTIGATOR: Daniel Grafstein  
Murray S. Cohen  
CONTRACT: AF 33(616)-3417  
CONTRACTOR: Reaction Motors, Inc.  
ABSTRACT: Several alternate synthetic procedures were developed for the preparation of substituted aryldialkylphosphine oxides. Among the tertiary phosphine oxides prepared were diethylphenylphosphine oxide, p-chlorophenyldiethylphosphine oxide, triethylphosphine oxide, ethylmethylphenylphosphine oxide and 2,5-dimethylphenyldiethylphosphine oxide. Other intermediates of some synthetic importance were prepared including o-tolydichlorophosphine, diethylphosphonyl chloride and ethylphenylphosphonyl chloride.

The inherent thermal and oxidative stability of the tertiary phosphine oxides has been experimentally verified at 285°C with both diethylphenylphosphine oxide and p-chlorophenyldiethylphosphine oxide. Principal efforts were devoted to improving the liquid range by introducing chosen substituents in certain positions of the aromatic ring of an aryldialkylphosphine oxide.

WADC TR 57-62 Pt I  
ASTIA Document No. AD 142326  
OTS Release

January 1958

SUBJECT: METAL-CYCLOPENTADIENYL HIGH TEMPERATURE LUBRICANTS  
AND RELATED MATERIALS  
Part I. Synthesis and Properties of Alkyl, Alkaryl,  
and Metalated Derivatives of Ferrocene

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INVESTIGATOR: Marvin Rausch, 1/Lt  
Martin Vogel, 1/Lt  
Dr. Harold Rosenberg

ABSTRACT: The purpose of this program was to uncover new types of cyclopentadienyl metals, or "ferrocene-like," compounds with properties suitable for extreme temperature lubricant and hydraulic fluid applications. A series of long-chain alkylferrocenes and arylalkylferrocenes were prepared by the Friedel-Craft acylation of ferrocene and subsequent reduction. Furthermore, the metalation of ferrocene by *n*-butyl-lithium and by mercuric acetate was studied in detail, and some reactions of the metalated derivatives were investigated.

WADC TR 57-62 Pt II  
ASTIA Document No. 150979  
OTS Release

February 1958

SUBJECT: METAL-CYCLOPENTADIENYL HIGH-TEMPERATURE LUBRICANTS  
AND RELATED MATERIALS  
Part II. A. Synthesis and Properties of Aryl, Benzyl,  
and Silyl Derivatives of Ferrocene. B. Preliminary  
Studies on the Effect of Tetrahydrofuran on the Lithi-  
ation of Ferrocene

INVESTIGATOR: Marvin Rausch, 1/Lt Dana Mayo, 1/Lt  
Martin Vogel, 1/Lt Paul Shaw, 1/Lt  
Dr. Harold Rosenberg

ABSTRACT: The purpose of this program was to uncover new types of ferrocene-containing materials with properties suitable for extreme-temperature lubricant and hydraulic fluid applications. A number of new aryl- and benzylferrocenes have been prepared. A valuable modification of the lithiation of ferrocene by *n*-butyllithium has been devised using ethyl ether-tetrahydrofuran as the solvent system. This procedure has been employed for the preparation of both carboxy- and 1,1-dicarboxyferrocene in good yield, and for the synthesis of long-chain alkylsilylferrocenes.

WADC TR 57-110  
ASTIA Document No. AD 142050  
OTS Release

October 1957

SUBJECT: POLYMERIZATION STUDIES ON MONOMERS AND EVALUATION  
OF DERIVATIVE POLYMERS  
INVESTIGATOR: Faber B. Jones Palmer B. Stickney  
Cherie A. Lichtenwalter Randall G. Heiligmann  
CONTRACT: AF 33(616)-3313

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# Contracts

CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: This report describes research to determine the polymerization characteristics of a group of experimental fluorine-containing monomers and a preliminary evaluation of the potential of the resulting polymeric products as high-temperature and solvent-resistant rubbers and plastics.

The homopolymerization of a group of six fluorinated olefin oxides in mass and solution using numerous different polymerization catalysts yielded only low-molecular-weight liquid polymeric products.

Copolymerization of several fluorine-containing propenyl ketones with dienes yielded some promising elastomers. Copolymerization of some fluorinated pentadienes, butenes and pentenes was effected to yield resinous solids; homopolymerization of these monomers was unsuccessful. Some fluorine-containing cyclic olefins were homopolymerized and copolymerized.

WADC TR 57-110 Pt II  
ASTIA Document No. 151011  
OTS Release

March 1958

SUBJECT: POLYMERIZATION STUDIES ON MONOMERS AND EVALUATION OF DERIVATIVE POLYMERS  
INVESTIGATOR: Faber B. Jones Palmer B. Stickney  
R. A. Markle Randall G. Heiligmann  
CONTRACT: AF 33(616)-3313  
CONTRACTOR: Battelle Memorial Institute  
ABSTRACT: This report describes research to determine the polymerization characteristics of a group of experimental monomeric compounds and a preliminary evaluation of the potential of the resulting polymeric products as thermally stable and solvent-resistant rubbers and plastics.

The homopolymerization of a group of six fluorine-containing olefin oxides in mass and solution using numerous different polymerization catalysts yielded only low-molecular-weight liquid polymeric products.

Copolymerization of perfluoromethylpropenyl ketone with butadiene at 5 C in aqueous emulsion by free-radical initiation yielded solid rubbery polymers. Copolymerization of perfluoropropylpropenyl ketone with 1, 1, 2-trifluoro-3-chlorobutadiene; 1,1,2-trifluorobutadiene; and normal butadiene by free-radical initiation in aqueous emulsion also yielded solid polymers with varying degrees of elastomeric character. Copolymerization of perfluoropropenyl ketone with 1,1,2-trifluoro-3,4,4-

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trichlorobutadiene or 1,1,2-trifluoro-3,4-dichlorobutadiene to solid polymeric products could not be accomplished by free radical or ionic initiation. Perfluoroheptylpropenyl ketone was observed to copolymerize with 1,3-dienes under conditions similar to those used for perfluoromethyl- and perfluoropropylpropenyl ketone but appeared to be less reactive.

The copolymers of perfluoromethylpropenyl ketone with butadiene and perfluoropropylpropenyl ketone with butadiene and 1,1,2-trifluoro-3-chlorobutadiene were briefly evaluated as rubber vulcanizates and showed some promise in high-temperature and solvent resistance.

The three perfluoroalkylpropenyl ketones appeared to homopolymerize in low yield to low-molecular-weight products by  $\text{BF}_3$  catalysis at low temperatures. Generally, good yields of solid polymeric products were obtained when these perfluoroalkylpropenyl ketones were copolymerized with styrene or 1,1,1-trifluoroethyl vinyl ether by  $\text{BF}_3$  catalysis under similar conditions.

Homopolymerization of perfluoro-1, 4-pentadiene; 1,1,3, 3,5,5-hexafluoro-1, 4-pentadiene; 1,1,2,3,3,4,5,5-octafluoro-4,5-dichloro-1-pentene; and 1-ethoxyperfluorocyclobutene-1 could not be accomplished by free-radical or ionic polymerization techniques. Copolymerization of these monomers with such comonomers as vinyl acetate; acrylonitrile; butadiene; styrene; 1,1,1-trifluoroethyl vinyl ether; 1,1-dihydroperfluorobutylacrylate; and N-n-butyl-N-1, 1-dihydroperfluorobutylacrylamide yielded polymeric products ranging from oils to elastomeric and resinous solids.

Homopolymerization of 1,2-diethoxyperfluorocyclobutene-1, 1-allyloxyperfluorocyclobutene-1; and 1,1,2,3,3,4,5,5-octafluoro-2-chloro-4-pentenylmethyl ketone by free-radical and ionic polymerization methods yielded liquid and solid polymeric products. Copolymerization of these monomers with styrene and N-n-butyl-N-1, 1-dihydroperfluorobutylacrylamide yielded resinous solid polymeric products.

Attempts were made to prepare high-molecular-weight homopolyesters by polycondensation of isomers of pyridine dicarboxylic acid with ethylene glycol. The products were brittle, low-molecular-weight, highly colored solids.

Transesterification of the dimethyl esters of the pyridine dicarboxylic acids with ethylene glycol did not give products of significantly improved properties. Replacement of ethylene glycol with neopentyl glycol, however, gave products of less coloration and apparently greater thermal stability.

Reaction of the diacid chlorides of some of the pyridine



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dicarboxylic acid isomers with ethylene and neopentyl glycol gave essentially the same results as transesterification reactions of the dimethyl esters, except that the reactions were more rapid and the products had, in general, greater clarity.

Thermosetting copolyesters were prepared by copolymerizing the pyridine dicarboxylic acids, their dimethyl esters or diacid chlorides, and an unsaturated dicarboxylic acid with ethylene glycol or neopentyl glycol. The products, translucent or opaque, dark brown semi-solids or solids at room temperature, could be modified with triallyl-cyanurate and cured to give dark brown, opaque, hard, nontacky solids.

Thermosetting polyesters were obtained by reacting pyridine diacrylic acid or its dimethyl ester with ethylene or neopentyl glycol. The uncured products were clear, dark brown, low-melting solids retaining double-bond activity.

A polycarbonate of moderate molecular weight was prepared by reacting 4,4' -dioxydiphenyl-2,2-propane (Bisphenol A) with phosgene or with diphenyl carbonate. A molded film approximately 0.010 inch thick prepared from the product obtained by fusion with diphenyl carbonate was fairly stiff but could be bent double without cracking.

A low-molecular-weight water-soluble resin was prepared by the reaction of bis(hydroxymethane) phosphinic acid with dimethyl terephthalate.

WADC TR 57-126 Pt I  
ASTIA Document No. AD 142028

October 1957

SUBJECT: PHOSPHINOBORINE POLYMERS

INVESTIGATOR: Ross I. Wagner

Earl M. Evleth

Levern D. Freeman

Robert D. Stewart

Ernest Levens

Leland W. Marple

CONTRACT: AF 33(616)-3506

CONTRACTOR: American Potash & Chemical Corporation

ABSTRACT: One of the continuing objectives of this project is the accumulation of fundamental data relating the structure of phosphinoborines to their chemical and physical properties. To this end, ten new phosphinoborines have been synthesized and several of these, as well as some of the previously known derivatives, have been screened for oxidative, hydrolytic, and thermal stability. The synthesis of phosphine and borine intermediates by known methods has produced four new alkyl phosphines. Several approaches to the preparation of aryl phosphines and diphosphines have been attempted without success.

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WADC TR 57-126 Pt II  
ASTIA Document No. 151182

April 1958

SUBJECT: PHOSPHINOBORINE POLYMERS  
INVESTIGATOR: Ross I. Wagner  
Frederick F. Caserio, Jr.  
Earl M. Evleth  
Levern D. Freeman  
Ernest Levens  
Kendrick R. Eilar

CONTRACT: AF 33(616)-3506

CONTRACTOR: American Potash & Chemical Corporation

ABSTRACT: One of the continuing objectives of this project is the accumulation of fundamental data relating the structures of phosphinoborines to their chemical and physical properties. To this end six new phosphinoborines have been synthesized and several of these, as well as some of the previously known derivatives, have been screened for oxidative, hydrolytic, and thermal stability. The synthesis of phosphine intermediates by known methods has produced one new alkylphosphine and six new organophosphine derivatives. The successful preparation of a primary aromatic diphosphine has been achieved.

WADC TR 57-143  
ASTIA Document No. AD 118308  
OTS Release

May 1958

SUBJECT: DEVELOPMENT OF THERMALLY STABLE SILICON  
CONTAINING RESINS  
INVESTIGATOR: L. W. Breed  
Fred Baiocchi  
Howard W. Christie

CONTRACT: AF 33(616)-3675

CONTRACTOR: Midwest Research Institute

ABSTRACT: The purpose of this program is the development of thermally stable silicon containing resins, and the parts of that program already completed are described in full. This program includes the synthesis of silane monomers which contain two silicon atoms connected by arylene groups and have sufficient functionality to form a thermosetting polymer when hydrolyzed and cured. It also includes finding methods of hydrolyzing and polymerizing these monomers in which the maximum use is made of their functionality.

Possible methods for synthesizing the monomers have been investigated. Those methods which were found suitable for the preparation of quantities of one pound or more were used to prepare the following compounds: 1,4-bis(methyldiethoxysilyl)benzene, 4,4'-bis(methyldiethoxysilyl)phenylether, and phenyldichlorosilyl-trichlorosilylbenzene. Also, the description of a statistically designed series of experiments to study the variables effective in the formation of laminates from silicone resins and glass fiber fabric is included.

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WADC TR 57-143 Pt II  
ASTIA Document No. AD 151002  
OTS Release

February 1958

SUBJECT: DEVELOPMENT OF THERMALLY STABLE SILICON  
CONTAINING RESINS

INVESTIGATOR: L. W. Breed  
Fred Baiocchi  
Calvin C. Bolze

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

ABSTRACT: Additional work aimed at preparing thermally stable silicon containing resins is described and some information on the incorporation of these resins into glass fabric laminates is included. Other methods for synthesizing monomers were investigated, particularly the use of tetrahydrofuran as a solvent in the Grignard syntheses. The results of a statistically designed experiment to study the variables effective in the formation of laminates from silicone resins and glass fabric are given, and the results of a similar experiment to study the effect of treating finished silicone resins with catalyst solutions are also described. Use of phenyldichlorosilyl-trichlorosilyl-benzene in resin compositions is reported in considerable detail and the properties of laminates prepared from these resins are described. Laminates are also obtained from resins prepared from 1,4-bis(methyldiethoxysilyl)benzene, 4,4'-bis(methyldiethoxysilyl)phenylether, m-bis(trichlorosilyl)benzene, p-bis(trichlorosilyl)-benzene and a mixture of the last two isomers.

WADC TR 57-189  
ASTIA Document No. AD 131095  
OTS Release

September 1957

SUBJECT: REACTIONS IN ELECTRODELESS DISCHARGES BETWEEN VOLATILE  
HALIDES AND ORGANIC COMPOUNDS

INVESTIGATOR: Gosta C. Akerlof  
CONTRACT: AF 33(616)-3225  
CONTRACTOR: Aerochem Research Laboratories, Inc.

ABSTRACT: The purpose of this program was to study the formation of new types of thermally stable fluids and solids by means of high voltage electrodeless discharges through mixtures of volatile halides, particularly of silicon, and organic compounds. The effect of temperature, pressure, flow rate, current, and voltage, on the product yield and discharge behavior was investigated for typical reactant combinations.

The product properties were studied for forty different reactant systems and twenty samples were submitted to the Materials Laboratory, Wright Air Development Center, for more detailed evaluation.

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In addition, a 600 gram sample boiling above 400°C, from the reaction of methane with dimethyldichlorosilane in the electrodeless discharge, was submitted for evaluation. Fractionation of similar samples has given cuts boiling over 200°C without visible decomposition - these same fractions were liquids at -15°C. The reaction between methane and methylchlorosilanes produces substances with high boiling points which have excellent thermal stability, combined with low freezing points and relatively low viscosity over large temperature ranges.

WADC TR 57-321  
ASTIA Document No. AD 130822

June 1957

**SUBJECT:** SYNTHESIS OF POLYXYLYLYLDIMETHYLSILANE AND  
POLYXYLYLYLTETRAMETHYL SILOXANE

**INVESTIGATOR:** Glenn R. Wilson  
Gretchen M. Hutzal  
Louis B. Updegrove

**CONTRACT:** AF 33(600)-32448

**CONTRACTOR:** Ford Motor Company

**ABSTRACT:** The purpose of this program is to prepare a series of poly-p-xylylyldimethylsilanes and poly-p-xylylyldimethyl siloxanes for evaluation as high-temperature lubricants and/or hydraulic fluids. The major effort has been devoted to finding a suitable procedure for preparing one of the building blocks of the polymers, viz., bis-(trimethylsilyl)-p-xylylene and suitable intermediates for synthesizing this latter compound. Many of the approaches for synthesizing the monomeric building block were unsuccessful and resulted in the formation of undesirable side reactions. Only recently was a successful procedure developed which involved the condensation of p-dibromobenzene and chloromethyltrimethylsilane with metallic sodium. The application of this successful procedure towards synthesis of the polymeric silane and siloxane are now under investigation.

WADC TR 57-321 Pt II  
ASTIA Document No. AD 155663

June 1958

**SUBJECT:** SYNTHESIS OF POLYXYLYLYLDIMETHYLSILANE AND  
POLYXYLYLYLTETRAMETHYL SILOXANE

**INVESTIGATOR:** Glenn R. Wilson  
Gretchen M. Hutzal  
Richard C. Hetu

**CONTRACT:** AF 33(600)-32448

**CONTRACTOR:** Ford Motor Company

**ABSTRACT:** The purpose of this project is the synthesis of a series of poly-p-xylylyldimethylsilanes and poly-p-xylylyltetramethylsiloxanes for evaluation as high temperature lubricants and/or hydraulic fluids.

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# Contrails

The proposed program of syntheses was outlined and discussed previously in WADC TR 57-321 and was directed primarily at working out the details for synthesizing the basic building unit of poly-p-xylylyldimethylsilane before proceeding to the ultimate synthesis of the polymers. At that time we had obtained a very small quantity of a liquid material, whose elemental analysis agreed exactly with that calculated for bis(trimethylsilyl)-p-xylylene, from the coupling reaction of p-dibromobenzene and chloromethyltrimethylsilane with metallic sodium. Subsequent efforts to prepare larger quantities of this material by scaling up the reaction were unsuccessful and complex mixtures of difficult-to-separate materials were obtained.

Because of the multiplicity of functional groups involved in this particular coupling reaction, other routes of synthesis were investigated in an attempt to reduce the numbers of permutations and combinations of competing reactions. The results of these investigations indicate that an unequivocal route is yet to be found for preparing the desired compound.

As final efforts we are investigating a Friedel-Crafts type reaction described in the recent Russian literature<sup>1</sup> and will also attempt to prepare the meta isomer of bis(trimethylsilyl) xylylene for which there is a much better chance of success.

WADC TR 57-391  
ASTIA Document No. AD 131100  
OTS Release

September 1957

SUBJECT: POLYMERIZATION THROUGH COORDINATION  
INVESTIGATOR: John C. Bailar, Jr.  
William C. Drinkard, Jr.  
Malcolm L. Judd  
CONTRACT: AF 33(616)-3209  
CONTRACTOR: University of Illinois  
ABSTRACT: The purpose of this work is to study the preparation and properties of metal-containing polymers in which the metal ion is held in the polymer through coordinate bonds. It is expected that such polymers will have exceptional stability toward heat and chemical reagents.

The background for the formation of such polymers is presented and the principles involved in the preparation of plastic materials are discussed. Pertinent literature references are cited.

Chelate polymers containing bis-p-diketones, bis-5-hydroxyquinolines, p-hydroxyethylpolyamines, and bis-q-amino acids have been investigated, as well as polymeric phthalocyanines. The most promising results have been obtained with the polymeric phthalocyanines, which have molecular weights up to about 30,000.

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WADC TR 57-436  
ASTIA Document No. AD 142116  
OTS Release

November 1957

SUBJECT: PREPARATION OF POLYMERS FROM HALOGEN-CONTAINING OLEFINS AND DIENE MONOMERS

INVESTIGATOR: H. Iserson  
M. Hauptschein  
F. T. Lawler

CONTRACT: AF 33(616)-3137

CONTRACTOR: Pennsalt Chemicals Corporation

ABSTRACT: Synthetic work on this project included the preparation of fluoro- and fluorohalo- olefins and dienes. Among the monomers prepared were 3-chloro-1,1,2-trifluorobutadiene, 1,2-dichloro-3,4,4-trifluorobutadiene, 1,1,2-trichloro-3,4,4-trifluorobutadiene, 3-bromo-1,1,2-trifluorobutadiene, 1,1,2,3-tetrafluorobutadiene, 1,4-dichlorotetrafluorobutadiene, 1,1-difluoroisobutylene, 1-trifluorovinyl-2-chloro-2,3,3-trifluorocyclobutane, 1-vinyl-2-chloro-1,2,3,3,4,4-hexafluorocyclobutane, perfluoro-1-vinylcyclobutene-1. Polymerizations have been tried with these and with other olefins. Some of the polymers which appear of interest were prepared from: perfluoropropene and trifluoroethyl vinyl ether; 1,1,2,3-tetrafluorobutadiene; 1,1,2,3-tetrafluorobutadiene and trifluoroethyl vinyl ether; 1,1,2,3-tetrafluorobutadiene and ethylene oxide; trifluoroethylene and trifluoroethyl vinyl ether; tetrafluoroethylene and ethylene oxide; tetrafluoroethylene and trifluoroethyl vinyl ether. Terpolymers have been prepared from trifluoroethyl vinyl ether, ethylene oxide and one of the following: perfluoropropene, vinylidene fluoride, 1,4-dichlorotetrafluorobutadiene, 1-chloro-2,2-difluoroethylene, tetrafluoroethylene.

WADC TR 57-502  
ASTIA Document No. AD 142100  
OTS Release

October 1957

SUBJECT: SILICON-OXYGEN-TIN POLYMERS

INVESTIGATOR: Joseph F. O'Brien, Capt

ABSTRACT: The purpose of this work was to develop a method for preparing new types of polymeric materials having a silicon-oxygen-tin-oxygen backbone. It was found that polymers of this structure could be prepared by (1) the reaction of dialkyltin diacetates with dialkyldiethoxysilanes, and (2) the reaction of dialkyltin dialkoxides with dialkyldiacetoxysilanes. Both of these reactions are condensation reactions in which a polymer is formed upon elimination of a volatile acetate. In contrast to this condensation reaction, both dialkyltin diacetates and dialkyltin dialkoxides undergo an exchange reaction with dialkyldichlorosilanes. Dialkyltin dichlorides do not react with either dialkyldiacetoxysilanes or dialkyldialkoxysilanes.

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WADC TR 57-503  
ASTIA Document No. AD 142101  
OTS Release

October 1957

SUBJECT: THE CHEMICAL REACTIONS OF SILICON ISOCYANATES  
INVESTIGATOR: Joseph F. O'Brien, Capt.  
ABSTRACT: The purpose of this research was to examine the chemical properties of silicon isocyanates to see if these isocyanates can be used as monomers for the preparation of silicon-containing polyurethane resins. A prototype silicon isocyanate, trimethylsilylisocyanate, was reacted with amines, Grignard reagents, alcohols and glycols. It was found that amines, Grignard reagents and alcohols cleave the isocyanate group from the silicon atom to form the corresponding alkyl urea, alkyl amide and alkyl biuret respectively. The glycols do not cleave the isocyanate group from the silicon, but add to trimethylsilylisocyanate to form the corresponding bis (trimethylsilyl) carbamate.

WADC TR 57-657  
ASTIA Document No. AD 142285

January 1958

SUBJECT: CONFERENCE ON HIGH TEMPERATURE POLYMER AND FLUID SYNTHESIS  
INVESTIGATOR: Calvin H. Schmid, Capt  
ABSTRACT: This report has been prepared to make available all the technical papers presented at the conference on "High Temperature Polymer and Fluid Syntheses."

The purpose of the conference was to review the progress on a comprehensive research program sponsored by the Polymer Section, Organic Materials Branch, toward the development of new polymeric and fluid materials to meet the high performance requirements of aircraft of advanced design, and to familiarize representatives of the chemical industry with the materials problems confronting the Air Force.

WADC TR 57-764  
ASTIA Document No. 150984  
OTS Release

February 1958

SUBJECT: FLUORINE CONTAINING CHELATE COMPOUNDS  
INVESTIGATOR: Glenn R. Buell, 1/Lt  
ABSTRACT: The purpose of this work was to study the feasibility of preparing liquid chelate compounds. The thermal stability of certain chelate compounds led to their study as possible liquid base stock materials. Several fluorinated diketones were chelated with metal ions and their melting points studied to determine the effect of the fluorine on the physical properties of the molecule. Only one liquid was obtained and all compounds exhibited high vapor pressures.

WADC TR 53-373 Sup 5

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January 1958

WADC TR 58-3  
ASTIA Document No. AD 142296  
OTS Release

**SUBJECT:** SYNTHESIS AND CHARACTERIZATION OF NEW VINYL POLYMERS  
**INVESTIGATOR:** Lester E. Coleman, Jr., 1/Lt  
William S. Durrell, 1/Lt  
**ABSTRACT:** This report describes research to determine the polymerization characteristics of four classes of experimental monomers and a preliminary evaluation of the potential of the resulting polymeric products as thermally stable, solvent resistant plastics and elastomers.

The perfluoroalkyl propenyl ketones were found to copolymerize with a variety of comonomers such as vinyl acetate, styrene and butadiene to give products ranging from powders to elastomers.

The 1-alkyl-1-hydroperfluoroalkyl acrylates copolymerized readily and formed tough rubbery homopolymers which exhibited good thermal stability and low swell in diester-type lubricants.

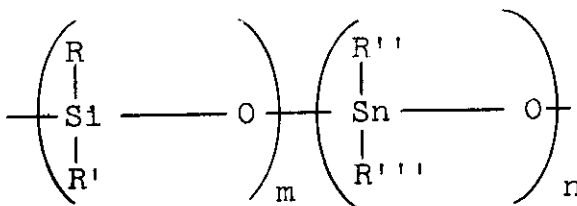
trans-Cinnamoyl ferrocene formed copolymers with a variety of comonomers but low conversions, copolymers or copolymers containing small amounts of cinnamoyl ferrocene were obtained.

Reactivity ratio determinations of two trifluoromethyl-substituted styrenes indicated that substitution of the trifluoromethyl group on styrene increased its polymerization reactivity.

WADC TR 58-44 Pt I  
ASTIA Document No. AD 151197

May 1958

**SUBJECT:** SYNTHESIS OF TIN-CONTAINING POLYMERS  
**INVESTIGATOR:** Paul E. Koenig  
James H. Hutchinson  
**CONTRACT:** AF 33(616)-3848  
**CONTRACTOR:** Ethyl Corporation  
**ABSTRACT:** This investigation has been largely directed toward the preparation of stannosiloxane polymers, whose structure may be represented as follows:

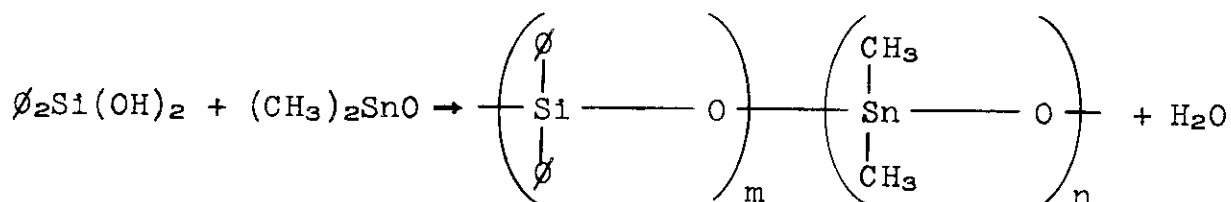


WADC TR 53-373 Sup 5



# Contrails

These compounds have been prepared by various condensation polymerizations, including a novel reaction between silanediols and organotin oxides, typified by the following example:



A second excellent synthetic method involves cohydrolysis of mixtures of dichlorosilanes and organotin dihalides; this method is well-suited to the preparation of polymers containing alkyl groups bound to silicon.

An analogous cohydrolysis reaction employing different organotin dihalides but no silicon compounds leads to formation of copolymeric stannoxanes; these are interesting in that they represent the first thermoplastic metal-oxide type polymers other than the silicones.

The products obtained are viscous fluids or transparent glasses; their hydrolytic and thermal stability is good. Difficulty has been experienced in attempting to produce high polymers over the composition range examined; the molecular weights thus far observed have been of the order of 1,-5,000, and some properties of the products are therefore correspondingly deficient. Work is in progress to obtain higher molecular weight products, which may well be exceptionally stable and highly useful materials.

WADC TR 58-51  
ASTIA Document No. 151177

April 1958

SUBJECT: HIGH POLYMERIC MATERIALS  
INVESTIGATOR: Carl S. Marvel  
Ludwig F. Audrieth  
John C. Bailar, Jr.  
CONTRACT: AF 33(616)-3772  
CONTRACTOR: University of Illinois  
ABSTRACT: The purpose of the work reported here is to provide basic information concerning new types of polymeric materials which will be more resistant to high temperatures than are known polymeric materials.

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The types of materials which have been investigated are: polymers which have the possibility of aromatization; copolymers of aromatic nuclei such as anthracene; polymers which contain metal coordination centers such as polyphthalocyanines, poly-Schiff base chelates, polybasic beryllium carboxylates; new derivatives of phosphonitrilic hydrosulfides; new polymers derived from benzene phosphonic amides; polymers containing coordination centers derived from bis-phenolic compounds.

Many new polymeric materials are described. One derived from dimethyl  $\alpha, \alpha'$ -dimethylenapimelate shows some possibilities as a high temperature resistant replacement of polymethyl methacrylate.

## RUBBER

WADC TR 52-197 Pt VII  
ASTIA Document No. AD 142027

October 1957

SUBJECT: SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS  
INVESTIGATOR: Carl A. Dahlquist  
CONTRACT: AF 33(038)-515  
CONTRACTOR: Minnesota Mining and Manufacturing Company  
ABSTRACT: Twenty pounds of the polymer of perfluoromethoxy 1,1 dihydroperfluoropropyl acrylate were prepared for Wright Air Development Center.

A copolymer of 3( $\omega$ -chloroperfluoroethoxy) 1,1 dihydroperfluoropropyl acrylate and its  $\omega$ -hydro analog has shown some promise as a heat, solvent, and low temperature resistant rubber.

Fluorine-containing silanes ( $R_fCH_2CH_2Si(CH_3)(OC_2H_5)_2$ ) were polymerized to low molecular weight oils (probably the cyclic tetramer), but attempts to polymerize the oils to high molecular weight polymers did not succeed. Rubbery copolymers of each of these silanes and dimethyl dichlorosilane have been prepared, but no useful vulcanizates have been obtained. Fluorine-containing polysiloxane oils having  $-Si(CH_3)_3$  and groups have been prepared.

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# Contrails

WADC TR 52-197 Pt VIII  
ASTIA Document No. AD 142114

November 1957

**SUBJECT:** SYNTHETIC RUBBERS FROM CARBON-FLUORINE COMPOUNDS  
**INVESTIGATOR:** Carl L. Sandberg  
**CONTRACT:** AF 33(038)-515  
**CONTRACTOR:** Minnesota Mining and Manufacturing Company  
**ABSTRACT:** Further evaluation of 3( $\omega$ -chloroperfluoroethoxy)-1,1-dihydroperfluoropropyl acrylate has shown it to be comparable to other perfluoro alkoxy acrylates in low temperature properties and resistance to aromatic solvents and hot air aging, but somewhat poorer in resistance to oxygenated solvents and to diester lubricants.

Attempts to homopolymerize 1,2-epoxyperfluoroheptane with peroxide catalysts or with a ferric chloride:propylene oxide catalyst have given mainly reaction products and little evidence of polymerization. Attempts to copolymerize 1,2-epoxyperfluoroheptane with propylene oxide, using a ferric chloride:propylene oxide catalyst, have given viscous liquid products which may be copolymers.

Exploratory copolymerizations of perfluoro-2-azapropene with vinyl-type monomers have given products containing small amounts of the azapropene.

WADC TR 55-58 Pt III  
ASTIA Document No. AD 130803

June 1957

**SUBJECT:** A STUDY OF THE EFFECTS OF NUCLEAR RADIATION ON ELASTOMERIC COMPOUNDS AND COMPOUNDING MATERIALS  
**INVESTIGATOR:** John W. Born  
Dwain E. Diller  
Eugene H. Rowe  
**CONTRACT:** AF 33(616)-2308  
**CONTRACTOR:** The B. F. Goodrich Company Research Center  
**ABSTRACT:** The effort to define, understand, and prevent radiation damage to rubber continued during 1956. Research followed four main lines: the development of Anti-Rads to prevent radiation damage; infrared and mass spectral analyses to relate radiation stability to molecular structures of elastomers; stress relaxation studies to define the mechanisms of radiation damage; and measurements of dynamic mechanical properties of representative rubber compounds after irradiation. Emphasis upon applied testing of end-item materials increased.

The best Anti-Rad extended the retention of tensile strength and ultimate elongation by natural rubber tread stock more than tenfold.

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# Contrails

The screening study is providing criteria for the selection of superior Anti-Rads. A new technique of mass spectral analysis promises to aid greatly in determining what molecular structural features impart radiation stability. Stress relaxation measurements detail the important role of oxygen in radiation damage. Dynamic test data are given for eight representative rubber compounds.

WADC TR 55-492 Pt II  
ASTIA Document No. AD 142024

October 1957

**SUBJECT:** DEVELOPMENT AND PHYSICAL TESTING OF ELASTOMERIC COMPOUNDS RESISTANT TO PETROLEUM BASE FUELS AT ELEVATED TEMPERATURES

**INVESTIGATOR:** Loren C. Smith                      Wayne G. Lajiness  
Theodore L. Eriksson                      Brock G. Peacock

**CONTRACT:** AF 33(616)-2779

**CONTRACTOR:** Wyandotte Chemicals Corporation

**ABSTRACT:** This report describes continued research directed toward the development of rubber compounds resistant to various petroleum base fuels for long exposures (500 hours) at elevated temperatures (up to 400°F.).

The test tube portion of the Type I Tester was redesigned and this tester was used to evaluate several candidate elastomers in the presence of test petroleum base fuels under simulated use conditions of elevated temperature and positive pressure.

The fabrication and construction of various parts of the multisample tester (Type II) was completed and its assembly initiated.

New selected candidate elastomers of the perfluorinated type were screened.

Compound and curing studies with the Kel-F elastomers were initiated and are continuing.

WADC TR 55-492 Pt III  
ASTIA Document No. 150978

February 1958

**SUBJECT:** DEVELOPMENT AND PHYSICAL TESTING OF ELASTOMERIC COMPOUNDS RESISTANT TO PETROLEUM BASE FUELS AT ELEVATED TEMPERATURES

**INVESTIGATOR:** Raymond G. Spain                      Eva Deck  
Wayne G. Lajiness                      Arnold L. McMaster

**CONTRACT:** AF 33(616)-2779

WADC TR 53-373 Sup 5

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# Contracts

**CONTRACTOR:** Wyandotte Chemicals Corporation  
**ABSTRACT:** This report describes continued research toward the development of elastomeric compounds resistant to petroleum base fuels at temperatures up to 400°F. for periods up to 500 hours.

Candidate elastomer compounds were screened using conventional fuel aging techniques. On the basis of these screening tests, compounds of interest were further tested under conditions of simulated use using the interim Type I Tester.

The multiple sample Type II Tester was completed during this report period.

The design and the major portion of assembly was completed for the Micro Tester which permits testing under more severe conditions than the two previous testers.

Aging studies were concentrated on compounds based on fluorinated elastomers.

WADC TR 56-155 Pt II  
ASTIA Document No. AD 131091  
OTS Release

September 1957

**SUBJECT:** HIGH TEMPERATURE RESISTANT SEALANT MATERIALS  
**INVESTIGATOR:** Leonard C. Boller      Arthur Milner  
Gene M. LeFave      John H. Emigh  
Edward O'Brien  
**CONTRACT:** AF 33(616)-2767  
**CONTRACTOR:** Coast Pro-Seal and Manufacturing Company  
**ABSTRACT:** This project was undertaken by the Coast Pro-Seal and Manufacturing Company originally to develop fuel tank sealant compounds capable of withstanding fuel vapor temperatures of 540°F and liquid fuel temperatures of 380°F for a limited period of time. These requirements have been essentially fulfilled and the results have been presented in WADC Technical Report No. 56-155.

Under an extension of the above contract, No. S1 (56-773) covered in this report, the previous temperature requirements were made inapplicable and a higher temperature (700°F) has been substituted for the 380°F and 540°F temperatures.

While the 700°F requirement was not attained, a substantial increase has been accomplished. A formula was developed for a sealant compound which was unaffected by JP-5 jet fuel in the 600°F to 625°F range.

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# Contrails

It retained flexibility and adhesion and withstood proof testing under flexing and pressure in the temperature range indicated. It is clear from the test data that operation in the presence of fuel vapor is possible in the neighborhood of 650°F.

A thorough formulation study was made of the butadiene-acrylonitrile rubber and phenolic resin system in order to develop the optimum thermal stability potential. The effect of added antioxidants, leafing pigments, plasticizers and certain other materials was determined.

Several other coating systems were briefly evaluated, such as the liquid polybutadiene modifications.

WADC TR 56-272 Pt II  
ASTIA Document No. AD 131094  
OTS Release

September 1957

SUBJECT: DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS  
INVESTIGATOR: George E. Trepus  
CONTRACT: AF 33(616)-2867  
CONTRACTOR: Boeing Airplane Company  
ABSTRACT: This study is to gain knowledge of the relationship between the physical properties of seal materials and sealing efficiency so that materials may be fully utilized in seal design. The literature survey on O-rings and seal design has been continued. Commercially available polymers have been compounded with various physical properties. Static annulus (with varying groove configuration), rotating shaft, and reciprocating shaft functional test jigs have been designed and manufactured. Seal tests, under various environmental and mechanical conditions, have been run in these jigs.

No definite relationship between physical properties and seal efficiency was found. A general relationship, however, was evident between seal life, and compression modulus and compression relaxation.

WADC TR 56-272 Pt III  
ASTIA Document No. 151181  
OTS Release

April 1958

SUBJECT: DESIGN DATA FOR O-RINGS AND SIMILAR ELASTIC SEALS  
INVESTIGATOR: Frank W. Tipton Ethel L. Schiavon  
George E. Trepus Chester J. Dezeih  
James J. Hill  
CONTRACT: AF 33(616)-2867  
CONTRACTOR: Boeing Airplane Company

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**ABSTRACT:** This is a continuation of a study to gain knowledge of design data for O-rings and similar elastomeric seals. The literature survey on O-rings and seal design has been continued and enlarged to include the effects of irradiation and extreme low temperature on seal design. Low temperature tests, relaxation and volume change tests, screening tests, and functional tests have been conducted. The functional tests include pulsed annulus tests with various groove configurations and reciprocating shaft tests with and without different types of back-ups at room temperature and at elevated temperatures.

No overall relationship between physical property values and seal efficiency has been found. It has been noted, however, that certain physical properties appear to be important to seal life for specific seal conditions.

WADC TR 56-331 Pt II  
ASTIA Document No. AD 151003  
OTS Release

February 1958

**SUBJECT:** DEVELOPMENT OF HIGH TEMPERATURE RESISTANT RUBBER COMPOUNDS  
**INVESTIGATOR:** Robert A. Hayes      Ward A. Smith  
Floyd M. Smith      Leland J. Kitchen  
**CONTRACT:** AF 33(616)-3953  
**CONTRACTOR:** The Firestone Tire & Rubber Company  
**ABSTRACT:** Elastomers of potential interest for high-temperature aircraft tires -- including butyl, silicone rubber, "heat-stable" polyurethane, and Viton A -- were studied to determine tensile, wire-cord adhesion, and air-permeability properties at temperatures ranging up to 400° or 500°F, and properties of interest in tire development, such as internal friction and running temperature. Most promising wire adhesions were obtained with silicone rubber, which retained 63% of the 75°-F adhesive strength at 450°F, compared with 9% for Hevea. Silicone rubber is the most highly permeable to air; Thiokol ST, Vyram and butyl have the lowest air permeabilities. Nitrogen appears to be the most promising of the unreactive gases to use for inflation of high-temperature tires.

Type of sulfur crosslink (mono-, di-, or tetrasulfide) had little effect on high-temperature properties of Hevea. Gamma-irradiation of vulcanized Hevea improved the high-temperature tensile strength. Balata had lower tensile strength than Hevea over the entire range of 73°F - 400°F. Vyram N-7700 was about equivalent to Vyram N-5400. Polyether-type polyurethane had 400°-F tensile strength of 60 psi vs. 515 for the "heat-stable" polyester type. Teflon resin had a tensile strength of 3825 psi at 73°F and 980 psi at 600°F.

WADC TR 53-373 Sup 5

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# Contrails

Studies on effects of a 4-hour heat-aging at 400 F<sup>o</sup> upon burst pressures of a nylon aircraft tire and of a wire tire are described. It is concluded that a 400<sup>o</sup>-F tire will require wire tirecord in place of organic fabrics used in present tires.

A prototype aircraft tire successfully was constructed from silicone rubber and steel wire tirecord. Coating the wire tirecord and the bead wire with a thin layer of partially cured silicone rubber facilitated fabrication of the beads and plies.

Experimental butadiene and acrylic polymers containing functional groups were prepared and their high-temperature properties were determined. Carboxyl as the functional group gave better results than hydroxyl or epoxy. Best properties at 400<sup>o</sup>F resulted from using a metal oxide — particularly magnesium oxide or cadmium oxide — with epoxy resin to cure the carboxy polymers, enabling 400<sup>o</sup>-F tensile strengths as high as 1400 psi to be obtained.

The advantages of the metal oxide cure were obtained with methacrylate rubber (copolymer of butadiene and methyl methacrylate) by curing with barium hydroxide octahydrate, which gave tensile strengths of 4025 psi at 73<sup>o</sup>F and 960 psi at 400<sup>o</sup>F when used in combination with bis(*o,o*-dimethylbenzyl) peroxide. In this manner the advantageous high-temperature properties of the metal oxide cure were realized with improved processability compared with the carboxy polymers.

It was found that butadiene copolymers can be vulcanized by heat alone at 500<sup>o</sup>F, a GR-S - carbon black mix giving a tensile of 2450 psi; but this crosslinking could only partially be inhibited. Salicylate esters were the most effective inhibitors.

WADC TR 57-247  
ASTIA Document No. AD 130899  
OTS Release

July 1957

SUBJECT: THE MICRO-COMPOUNDING AND EVALUATION OF RUBBER-LIKE POLYMERS  
INVESTIGATOR: E. G. Schwarz, Lt  
ABSTRACT: A micro-compounding technique has been developed to process the "research sized" samples resulting from Air Force and Industrial polymer development programs.

Scaled down equipment and testing procedures have also been developed successfully to evaluate the small gram-size samples of vulcanized elastomers. Much of the insight to compounding and vulcanizing these experimental polymers is based upon the chemistry of the system, supplemented by experience and common sense.

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# Contrails

Using the technique described in this report, experimental polymers can be evaluated and the decision made to reject or further develop the polymer system -with good assurance that a promising polymer has not been cast aside and that further effort on others is justified.

WADC TR 57-346  
ASTIA Document No. AD 142156

November 1957

SUBJECT: THE DEVELOPMENT OF A HOSE AND HOSE END FITTING FOR  
POLY FBA SYNTHETIC RUBBER  
INVESTIGATOR: John P. Thomas  
CONTRACT: AF 33(600)-29001  
CONTRACTOR: Flexonics Corporation  
ABSTRACT: Poly FBA can be processed with conventional rubber equipment modified to suit its peculiar properties in processing.

Poly FBA is limited in its use as a hose material to lip seal fittings, because of its cold flow properties even at temperatures of 300° F.

WADC TR 57-364  
ASTIA Document No. AD 142051

October 1957

SUBJECT: RESEARCH ON MATERIALS SUITABLE FOR FABRICATION INTO  
V51R DESIGN EAR PLUGS  
INVESTIGATOR: Edgar A. Verchot  
Arthur E. Raeuber  
CONTRACT: AF 33(616)-3488  
CONTRACTOR: Southern Research Institute  
ABSTRACT: This report describes research and development work directed toward finding an acceptable formulation for V51R design ear plugs and toward setting up test methods so that future lots of purchased ear plugs will equal the acceptable formulation.

A test procedure to measure the collapsibility of a medium-size ear plug has been developed. Collapsibility measurements were used to define the properties of ear plugs acceptable to those experienced in the art of using and fitting ear plugs to military personnel.

The ear plug formulations studied were also evaluated with respect to tensile strength, resistance to low temperatures, resistance to lanolin and to detergents, color, surface finish, and resistance to accelerated aging.

WADC TR 53-373 Sep 5

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# Contracts

Three ear plug formulations were selected as being the most acceptable according to the results of the tests made. The properties of these formulations have been used as the basis for suggested changes in the specifications given in Military Medical Purchase Description Number 4, dated 16 August 1954.

It is recommended that ear plugs based on these formulations be manufactured and tested in field use before further minor adjustments in formulation are attempted.

WADC TR 57-553  
ASTIA Document No. AD 142281

January 1958

**SUBJECT:** DEVELOPMENT OF REFUELING HOSE & COUPLINGS  
**INVESTIGATOR:** Stanley L. Bertholf  
Leonard Castelbaum  
Dante E. Piccoli  
**CONTRACT:** AF 33(600)-27745  
**CONTRACTOR:** United States Rubber Company  
**ABSTRACT:** Development of two basic types of 3" I.D. inflight refueling hose, wire woven type and wire braided type, along with suitable end fittings was the object of this development contract. All work pertaining to hose was performed by United States Rubber Company, at Passaic, New Jersey. Coupling design work was performed by The Weatherhead Company of Cleveland, Ohio, and the Flex-O-Tube Division of Flexonics, Inc., of Detroit, Michigan.

Three different constructions of wire braid hose were developed. The wire woven type hose was eliminated because this type of hose exhibited excessive elongation under load. A total of 35 lengths of wire braided hose assemblies coupled with swaged fittings were furnished under the contract.

WADC TR 57-651  
ASTIA Document No. AD 142205

December 1957

**SUBJECT:** DEVELOPMENT OF RUBBERLIKE MATERIALS FOR APPLICATIONS INVOLVING CONTACT WITH LIQUID ROCKET PROPELLANTS  
**INVESTIGATOR:** John H. Baldrige  
**CONTRACT:** AF 33(616)-3951  
**CONTRACTOR:** The Connecticut Hard Rubber Company  
**ABSTRACT:** A survey of available knowledge concerning the resistance of elastomers and plastics to a number of propellants has been prepared and will appear as WADC TR57-472.

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# Contrails

Elastomeric materials showing satisfactory resistance during an immersion period of seven days at 160°F. have been developed for the following propellants; unsymmetrical dimethyl hydrazine, ethylene oxide, n-propyl nitrate, and anhydrous ammonia. Compounds were tested which were resistant to 90 percent hydrogen peroxide at room temperature.

No elastomeric materials were found with satisfactory room temperature resistance to inhibited red fuming nitric acid or to JP-X fuel mixture.

WADC TR 58-18  
ASTIA Document No. AD 151196

May 1958

SUBJECT: INVESTIGATION OF OIL AGING PROCEDURES FOR ELASTOMERIC MATERIALS  
INVESTIGATOR: K. Murray  
ABSTRACT: Several oil aging procedures utilizing various venting methods, and an oven or an aluminum block heater as heat sources, were compared to determine the most suitable, reproducible test procedure for evaluating potential oil-resistant elastomers at elevated temperatures.

The "chimneyed-stoppered-tube method" as described in this report provides for better reproducible results, and a better, more reliable evaluation of potential oil-resistant elastomers at elevated temperatures.

WADC TR 58-19  
ASTIA Document No. AD 150983  
OTS Release

February 1958

SUBJECT: AN EVALUATION OF FLUORO-RUBBER 2F4  
INVESTIGATOR: Roger E. Headrick  
ABSTRACT: Fluoro-Rubber 2F4 was developed by the Minnesota Mining and Manufacturing Company under Air Force Contract AF 33(038)515 to overcome the low temperature limitations of its precursor Fluoro-Rubber 1F4 (poly FBA).

This elastomer has a 30° to 40°F. improvement in low temperature properties (-35°F. Brittle Point) over Fluoro-Rubber 1F4 and has excellent resistance to jet fuels, lubricants and solvents and has a fair retention of physical properties after aging at elevated temperatures.

WADC TR 53-373 Sup 5

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SANDWICH CONSTRUCTION

WADC TR 52-184 Sup 5  
ASTIA Document No. AD 142102

October 1957

**SUBJECT:** SUMMARY OF RESEARCH BY FOREST PRODUCTS LABORATORY ON SANDWICH CONSTRUCTIONS FOR AIRCRAFT  
**INVESTIGATOR:** Donald G. Coleman  
**CONTRACT:** AF 33(616)-56-9  
**CONTRACTOR:** Forest Products Laboratory  
**ABSTRACT:** Developments in the program of research in sandwich construction for aircraft conducted by the U. S. Forest Products Laboratory during fiscal year 1957 are summarized. The approach has been in general to derive design criteria mathematically and then to check by test. Seven technical reports issued during the fiscal year are abstracted.

WADC TR 55-417 Pt II  
ASTIA Document No. AD 142052

October 1957

**SUBJECT:** PERFORMANCE OF STAINLESS STEEL SANDWICH CONSTRUCTION AT HIGH TEMPERATURES  
**INVESTIGATOR:** E. W. Kuenzi  
W. E. Jahnke  
**CONTRACT:** DO 33(616)56-9  
**CONTRACTOR:** Forest Products Laboratory  
**ABSTRACT:** This report presents the results of test to determine structural performance of sandwich comprised of facings of stainless steel or titanium bonded to stainless steel honeycomb cores with heat-resistant, epoxy-phenol adhesive. Performance was determined at 75°, 300°, and 600°F. and after exposure at 300°F. for 100 and 500 hours. Included are results of a small-scale investigation of the effect of two methods of preparing facings for bonding and the effect of bonding with two adhesive layers instead of the usual single layer.

WADC TR 57-609  
ASTIA Document No. AD 155579  
OTS Release

May 1958

**SUBJECT:** STRENGTH AND ELECTRICAL TRANSMISSION OF THREE TYPES OF REPAIRS TO SANDWICH CONSTRUCTION FOR RADOMES  
**INVESTIGATOR:** Bruce G. Heebink  
V. P. Miniutti

WADC TR 53-373 Sup 5

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# Contrails

**CONTRACT:** DO 33(616)56-9  
**CONTRACTOR:** Forest Products Laboratory  
**ABSTRACT:** The purpose of this technical report was to evaluate three methods for repairing damage to a typical radome sandwich construction. Six similarly damaged panels were chosen, and WADC sent three to Tinker Air Force Base and three to the Forest Products Laboratory for repair. The repair procedures at Tinker Air Force Base were similar to those used at the Laboratory.

The repaired panels were tested for electrical transmission by WADC and for strength at the Laboratory.

Tests were made to determine edgewise tensile, edgewise compressive, and flatwise tensile strength of the repaired and undamaged sections of the panels. One panel in each set could not be tested because it blistered while the resin was being oven cured.

Results showed, except for edgewise compressive strength of scarf-jointed repairs, that repair efficiency was highest when only one facing was repaired, and that no one method was superior for both one- and two-facing repairs. However, because (a) results from the limited number of strength tests varied considerably, (b) the two repairing facilities used different repair resins, and (c) only one of the three repair methods was duplicated, valid conclusions regarding the repair efficiency of the three methods and a comparison of the repair efficiencies obtained by the two facilities cannot be made. The results of electrical transmission tests on two of the repair methods show that the methods are electrically suitable for general radome repair.

WADC TR 58-76  
ASTIA Document No. AD 151085

March 1958

**SUBJECT:** FLEXURE AND TORSION OF COMPOSITE CYLINDERS  
**INVESTIGATOR:** W. S. Ericksen  
**CONTRACTOR:** Air Force Institute of Technology  
**ABSTRACT:** A solution to the problem of determining the components of stress and displacement in composite cylinders supported as cantilever beams and subjected to flexural and torsional loads is given in this report. The type of cylinder considered is that composed of three circular, coaxial, layers of different materials that are bonded at their junctures. Two main cases are considered; one in which the material in the center layer is cylindrically anisotropic, the other in which it is isotropic. The material in the inner and outer layers is assumed to be isotropic. By taking the thickness of one of the layers equal to zero, the results are applicable to a two-layer cylinder and, by making the inner radius zero, they are applicable to a solid cylinder.

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## TESTS, NONDESTRUCTIVE

WADC TR 56-607

June 1957

ASTIA Document No. AD 130848

**SUBJECT:** SURVEY TO DETERMINE THE AIR FORCE RESEARCH AND DEVELOPMENT NEEDS IN THE FIELD OF NONDESTRUCTIVE TESTING

**INVESTIGATOR:** S. A. Wenk  
A. J. Schwarber, Jr.  
R. R. Showalter

**CONTRACT:** AF 33(616)-3475

**CONTRACTOR:** Battelle Memorial Institute

**ABSTRACT:** The increasing complexity of air weapons systems has resulted in increased dependence on nondestructive testing as a means of assuring the conformance of aircraft materials and components to design criteria. This survey reviews the problems inherent in existing nondestructive testing methods, as well as those inspection problems for which no present nondestructive test is adequate. In addition, the shortage of skilled personnel, and the necessity of establishing training programs, is cited.

Specific problems which require a research and development effort are enumerated and, where applicable, recommendations for action have been made, based on Air Force need and chances for technological success.

WADC TR 57-268

October 1957

ASTIA Document No. AD 142034

OTS Release

**SUBJECT:** RESEARCH AND DEVELOPMENT LEADING TO THE ESTABLISHMENT OF ULTRASONIC TEST STANDARDS FOR AIRCRAFT MATERIALS

**INVESTIGATOR:** A. D. Guinn J. C. Folz  
R. D. McKown W. C. Hitt

**CONTRACT:** AF33(616)-3363

**CONTRACTOR:** Ultrasonic Testing & Research Laboratory

**ABSTRACT:** Very little information has been available for use by engineering personnel interested in setting up ultrasonic inspection and testing reference standards for use in determining the acceptability of aircraft materials. Since the ultrasonic method of inspection and testing of aircraft materials is now widely employed, a very definite need exists for test standards for use in determining the acceptability of aircraft materials undergoing this method of inspection and testing. While this research program was somewhat limited in its scope, it is believed that the data collected will provide the basis for the establishment of ultrasonic test standards for the aircraft materials studied.

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WADC TR 58-35  
ASTIA Document No. AD 155558  
OTS Release

May 1958

SUBJECT: EARLY DETECTION OF FATIGUE IN ALUMINUM ALLOYS BY  
ULTRASONICS  
INVESTIGATOR: Karl Sittel  
Marvin Herman  
Robert C. Good, Jr.  
CONTRACT: AF 33(616)-3930  
CONTRACTOR: The Franklin Institute  
ABSTRACT: The literature concerning ultrasonic wave propagation and fatigue-induced damage in aluminum has been surveyed to determine the possible interactions. The causes of wave attenuation have been compared with the fatigue effects in metals predicted by various theories to determine the experimental conditions that would maximize the measurements of those effects. Particular attention is paid to the dislocations present in metals because they form and move throughout the fatigue life of a sample. The problem at hand is the detection of incipient fatigue which implies early detection and measurement.

The experimental work has included the design and construction of a resonant type fatiguing machine and an electro-acoustic driving system for ultrasonic waves. Enough tests were made to show the operability of the individual parts of the system, but insufficient time was available after the literature search to record full-scale tests. The specimen shape to be recommended for further tests is a torsion bar with the ultrasonic waves propagated along its axis.

## TEXTILES

WADC TR 56-576  
ASTIA Document No. AD 131055  
OTS Release

September 1957

SUBJECT: DEVELOPMENT OF DESIGN DATA ON THE MECHANICS OF AIR  
FLOW THROUGH PARACHUTE FABRICS  
INVESTIGATOR: William G. Klein  
Charles A. Lermond  
Milton M. Platt  
CONTRACT: AF 33(616)-2977  
CONTRACTOR: Fabric Research Laboratories, Inc.

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**ABSTRACT:** The purposes of this report are two: 1) A determination of the factors involved in parachute fabric permeability and the quantitative prediction of their influence with a view to rational engineering design of such materials; and, 2) An assay of a representative selection of commercially produced parachute fabrics (Type I and Type II) to determine the degree to which they meet current permeability specifications and, where such requirements are not met, the reasons therefore.

The theoretical investigation of the mechanics of airflow is prosecuted as follows:

- A) A classical fluid flow relation is restated in terms of fabric parameters.
- B) A number of experimental and theoretical relationships are formulated.
- C) The various relationships are combined to give the necessary information for checking the validity of the theoretical approach and assumption made.

The results of the various analyses support to a large extent the approach and mechanism of solution, but the work to date has been analytic only. The problem of synthesis can be rationally attacked only after the compilation of more information on the biaxial deformation of fabric structures.

The commercially produced fabrics showed a wide variation in permeability. Only 60% of the Type I and 47% of the Type II fabrics met the permeability specification of MIL-C-7020 A under which they were manufactured. In all cases the reasons for the deviations were explainable on the basis of yarn diameters and picks and ends.

WADC TR 57-157  
ASTIA Document No. AD 142261  
OTS Release

December 1957

**SUBJECT:** AERODYNAMIC HEATING OF PARACHUTES  
**INVESTIGATOR:** A. L. Ruoff  
S. W. Liu  
F. Frank  
**CONTRACT:** AF 33(616)-3572  
**CONTRACTOR:** Cornell University  
**ABSTRACT:** Heating rates for a parachute employed at varying Mach numbers (2 - 5) and altitudes (sea level to 100,000 feet) are obtained. Various methods of protecting the nylon parachute are discussed. Although

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requiring further engineering development the following methods are theoretically feasible: Coating with a silicone foam which reduces the heat transfer rate to the nylon is possible; use of a sublimating coating, e.g., hexachlorethane is feasible; and the use of water absorbed in a polyurethane foam coating on the nylon is possible.

Plots are given for the heat fluxes at various points. Since the actual total heat flux in a given case depends on the deceleration rate of the specific parachute only specific cases could be considered. However, the method is described, whereby, using the data of this report the amount of evaporating material required to keep the nylon at a safe temperature can be very readily calculated when a specific time-velocity profile is given.

WADC TR 57-416  
ASTIA Document No. AD 142094  
OTS Release

November 1957

SUBJECT: THE DEVELOPMENT OF A COATING FORMULATION AND METHOD OF APPLICATION FOR USE IN NYLON DOUBLE FABRIC  
INVESTIGATOR: Ernest H. Pagliaro  
CONTRACT: AF 33(616)-3901  
CONTRACTOR: The Connecticut Hard Rubber Company  
ABSTRACT: A two ply modified plain weave nylon fabric has been successfully coated on both sides using conventional knife over roll rubber spreading equipment. Blocking between the plies was prevented by closely controlling the solids concentration and the amount of compound deposited per pass. Butyl rubber was selected over the other elastomers evaluated because it was the most flexible when tested at  $-65^{\circ}\text{F}$  according to requirements of the contract. Specimens coated with Butyl rubber have also met the other necessary requirements including adhesion, weight, and air retention.

WADC TR 57-443  
ASTIA Document No. AD 142208  
OTS Release

December 1957

SUBJECT: DESIGN DATA ON BIAxIAL FORCES DEVELOPED IN PARACHUTE FABRICS  
INVESTIGATOR: Jan G. Krizik                      Joseph F. Cheatham  
                  Ebrahim Victory                      Stanley Backer  
CONTRACT: AF 33(616)-3253  
CONTRACTOR: Massachusetts Institute of Technology  
ABSTRACT: A high pressure permeometer has been constructed for use with parachute cloths over a differential pressure range of 0.5 to 1000 inches of water. The unit consists of a compressor, test duct with special biaxial stress jaws, and a steam ejector in series. Operation with variable

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air densities is possible to simulate high altitude parachute operation. A range of standard and experimental parachute materials has been tested on the permeometer and extensive data are available relating air flow, biaxial stress and strain and area increase to pressure differentials across the fabric. Data showing the effect of sample prestressing, cyclic testing and average air density are included.

WADC TR 57-538  
ASTIA Document No. AD 151090  
OTS Release

March 1958

**SUBJECT:** DEVELOPMENT AND EVALUATION OF WEBBING MADE FROM NYLON "6"  
**INVESTIGATOR:** Russell J. Neff  
**CONTRACT:** AF 33(600)-33484  
**CONTRACTOR:** Phoenix Trimming Company  
**ABSTRACT:** The purpose of the work herein reported was the development and evaluation of a series of nylon webbings utilizing Type "6" nylon.

A group of webbings were manufactured. Ten were made using 210 denier yarn and four were made using modified 840 denier yarn. The webbings were in natural and OD color, untreated and treated, made in accordance with Specification MIL-W-4088C and Specification MIL-W-5625. It should be noted that a solid weave was used in lieu of a tubular under Specification MIL-W-5625 and that the number of ends and yarn ply of the 1/2", 9/16" and 5/8" webbings were in accordance with the constructions used in WADC Technical Report 55-494. A series of tests were conducted to determine if the use of Type "6" nylon was feasible in Air Force webbings.

This investigation showed that the webbings manufactured from regular 210 denier Type "6" nylon have slightly lower breaking strength and were susceptible to heat degradation at lower temperature than webbings presently being used by the Air Force which utilize the Type "66" nylon. The webbings manufactured using the Type "6" modified 840 denier yarn appeared to be equal in strength and superior in their resistance to heat degradation up to a temperature of 300° F.

WADC TR 57-765  
ASTIA Document No. AD 155511  
OTS Release

May 1958

**SUBJECT:** DEVELOPMENT OF HIGH TENACITY-HEAT STABLE DACRON PARACHUTE ITEMS  
**INVESTIGATOR:** Chauncey C. Chu  
Ernest R. Kaswell  
Donald J. Doull  
**CONTRACT:** AF 33(616)-3593

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# Contracts

CONTRACTOR: Fabric Research Laboratories, Inc.

ABSTRACT: A Pilot machine has been designed, constructed, and used for the production of "Hot Stretched-Heat Relaxed" (HS-HR) Dacron yarns and threads. Yarns so produced were converted, where necessary, into selected end items, namely webbings, braid, and tape.

The HS-HR process consists of hot stretching the yarns over heated surfaces at 375 ° to 400 °F., and then relaxing the yarns at a surface temperature in approximately the same range. Considerable difficulty was encountered in producing in proper quality, the relatively large quantities of yarns needed for the selected end items, and much experience was gained in this ultimate accomplishment.

A previous investigation, WADC TR 55-135, reports on the properties of counterpart items prepared from "Heat Relaxed" (HR) Dacron yarns. The HR process consists of merely freely relaxing the yarn at 375 ° to 400 °F.

Comparison and evaluation of the HS-HR and the HR processes with particular reference to their use for Dacron parachute component are the subject of this current work. Unprocessed producer's type 5100 Dacron (U) has high tenacity, low elongation and high heat shrinkage. Heat Relaxed (HR) Dacron has low tenacity, high elongation, and essentially no shrinkage. Hot Stretched-Heat Relaxed (HS-HR) Dacron has high tenacity, intermediate elongation, and essentially no shrinkage.

HS-HR Dacron meets essentially all of the end item specifications originally delineated for nylon. In addition it has better heat degradation resistance than nylon, better heat stability than producer's Dacron, and higher strength per unit weight than Heat Relaxed (HR) Dacron.

In a Dacron arrestation parachute the relationships among deformation upon deployment, subsequent recovery, subsequent heat stability, and utility for repeated deployment, are not completely known. The HS-HR process appears to have some advantages over the HR process, but whether it is worth the additional effort and cost, can only be decided after additional study.

HS-HR end items have been delivered to WADC for comparative field evaluation with previously delivered HR end items.

WADC TR 58-65  
ASTIA Document No. AD 155517  
OTS Release

May 1958

**SUBJECT:** RESEARCH PROGRAM FOR THE DEVELOPMENT OF A DESIGN  
PROCEDURE TO ENGINEER PARACHUTE FABRICS

**INVESTIGATOR:** William G. Klein  
Charles A. Lermond  
Milton M. Platt

**CONTRACT:** AF 33(616)-3845

**CONTRACTOR:** Fabric Research Laboratories, Inc.

**ABSTRACT:** This report presents an analysis which it is felt comes closer to defining the mechanisms of air flow through parachute materials than previous attempts have done. The significant difference from prior work is that the fabric is treated as a three rather than two-dimensional structure, thus permitting a more accurate characterization of the actual flow region. The fabrics considered are those conforming to the physical specifications of MIL-C-7020, Types I and II, but with permeabilities ranging from well below to well above current specifications.

While very satisfactory results are obtained in explaining permeability on the basis of fabric structure, it is also shown that some of the variables controlling permeability must, for specified permeability range, be held closer than is possible by present commercial practice.

## TRANSPARENT MATERIALS

WADC TR 54-619 Pt 2  
ASTIA Document No. AD 142084

**SUBJECT:** THE INVESTIGATION OF MULTIAXIALLY STRETCHED ACRYLIC  
PLASTIC  
Part 2. Biaxially Stretched Plastic

**INVESTIGATOR:** Alfred Batzdorff  
Julius J. Gouza  
Dee A. Hurst

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CONTRACT: AF 33(616)-489  
CONTRACTOR: Rohm & Haas Company  
ABSTRACT: The purpose of this investigation was to further define the effects of stretching procedures and of simulated warm forming treatments on the material properties of acrylic plastic. It also expanded the scope of previous work by including studies on stretched poly(methyl alpha chloroacrylate). A new phase of these investigations was the gunfire tests on stretched materials.

Conditions were determined for stretching poly(methyl alpha chloroacrylate), or Gafite sheet, and physical properties of the stretched sheet were obtained. Stress-solvent crazing resistance increased with increase in percent stretch, and the Gafite was significantly more stress-solvent craze resistant than the MIL-P-5425 and MIL-P-8184 materials. All three materials behaved similarly in showing much less stress-solvent crazing resistance after six months outdoor aging.

Both Gafite and MIL-P-8184 materials with moderate to high degrees of stretch were highly resistant to stress-only crazing after six months outdoor aging, and were better than stretched MIL-P-5425 material in this respect.

The effect of varying the conditions of stretching, especially the rate of stretching, on the properties of three types of acrylic plastic with several different percents of stretch was evaluated by means of  $dW/dA$  tests,  $50^{\circ}C$  crazing tests, and gunfire resistance. The results indicated definite effects of stretching conditions on the gunfire resistance and crazing properties, but no significant effects on the  $dW/dA$  results on the stretched materials.

The effect of a simulated warm forming cycle on the properties of stretched materials was of no great magnitude. There was some reduction in stress-solvent crazing resistance, but the properties were still much improved as compared to unstretched material.

Conditions of temperature, speed of testing, and moisture content of the specimen affected the  $dW/dA$  test results quite significantly. Limited investigation was made of these phenomena. The effect of specimen size on the  $dW/dA$  results was investigated.

Some of the sources of variation of results on  $dW/dA$  tests were analyzed. There was considerable variation with percent stretch and the 60% stretch region on MIL-P-8184 material represented a wide range of  $dW/dA$  values which were heterogeneous in their source. It would be wise to avoid this degree of stretch in order to reduce variability in crack propagation resistance.

# Contrails

WADC TR 56-352 Pt II  
ASTIA Document No. AD 142047

October 1957

**SUBJECT:** DEVELOPMENT OF A RIGID TRANSPARENT PLASTIC MATERIAL SUITABLE FOR AIRCRAFT GLAZING AT ELEVATED TEMPERATURE

**INVESTIGATOR:** George Helvey  
Harold A. Clark

**CONTRACT:** AF 33(616)-3065

**CONTRACTOR:** Dow Corning Corporation

**ABSTRACT:** Additional research work was conducted to obtain a rigid, transparent plastic having properties at elevated temperatures superior to those of the presently available transparent materials.

Evaluation of straight silicone resins, designated as Resin Type BB, was continued but no significant improvement in properties was realized. Although this resin type has excellent optical properties (luminous transmittance of over 90 percent and haze less than 1 percent) which are not visibly affected by heat aging (thermally stable after prolonged exposure at 200 degrees C) the physical properties are, in general, well below those of other commercially available materials. The Heat Distortion Temperature of Resin Type BB is 40°C to 66°C. This material will not be satisfactory for glazing applications requiring rigidity at elevated temperatures but may be adapted for other uses.

Flake glass reinforcement of the resin was attempted but was not successful because of poor orientation.

Large sheet castings were successfully fabricated by processing the silicone resins using special techniques.

WADC TR 57-24  
ASTIA Document No. AD 130914

July 1957

**SUBJECT:** DEVELOPMENT OF A RIGID FLUORINATED TRANSPARENT PLASTIC MATERIAL SUITABLE FOR AIRCRAFT GLAZING AT ELEVATED TEMPERATURES

**INVESTIGATOR:** Harry D. Anspen  
Joseph J. Baron, Jr.

**CONTRACT:** AF 33(616)-3240

**CONTRACTOR:** General Aniline and Film Corporation

**ABSTRACT:** The field of fluoroacrylate polymers was studied as a starting point on investigation of new polymeric materials for use as high temperature resistant, transparent, glazing materials.

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A literature survey of this field showed that, although several fluoroacrylate polymers had been prepared previously, almost no information was available on the polymers and their properties.

The fluorine analogs (methyl  $\alpha$ -(trifluoromethyl)acrylate  $\text{CH}_2=\text{C}(\text{CF}_3)\text{COOCH}_3$ ; and methyl  $\alpha$ -fluoroacrylate,  $\text{CH}_2=\text{CFCOOCH}_3$ ) of the two, well-known, readily-polymerizable monomers, methyl methacrylate,  $\text{CH}_2=\text{C}(\text{CH}_3)\text{COOCH}_3$  and methyl  $\alpha$ -chloroacrylate,  $\text{CH}_2=\text{CClCOOCH}_3$ , were prepared by methods described in the literature.

Methyl  $\alpha$ -(trifluoromethyl)acrylate did not polymerize under conventional methods nor under special conditions recommended in patents by a previous investigator. It was polymerized to a transparent solid by extended gamma radiation. The characteristics of the polymer obtained by gamma radiation remain to be determined.

Methyl  $\alpha$ -fluoroacrylate polymerized readily under light irradiation to a tough, transparent, solid polymer with a heat distortion temperature of  $110^\circ\text{C}$ . ( $230^\circ\text{F}$ .) with excellent solvent craze resistance, and with a heat stability of about 2 hours at  $204^\circ\text{C}$ . ( $400^\circ\text{F}$ .) in an air oven. Its heat distortion temperature was lower than  $135^\circ\text{C}$ . ( $275^\circ\text{F}$ .), the heat distortion temperature of the corresponding chloro compound, methyl  $\alpha$ -chloroacrylate; but its toughness, solvent resistance, and heat stability were superior to those of methyl  $\alpha$ -chloroacrylate polymer ("GAFITE"). Additional quantities of methyl  $\alpha$ -fluoroacrylate are in preparation to provide samples for more extensive testing of this polymer.

Exploratory work on syntheses of methyl trifluoroacrylate,  $\text{CF}_2=\text{CFCOOCH}_3$ ; and methyl  $\alpha$ -chloro- $\beta,\beta$ -difluoroacrylate,  $\text{CF}_2=\text{CClCOOCH}_3$  was initiated.

WADC TR 57-24 Pt II  
ASTIA Document No. 150966

February 1958

SUBJECT: DEVELOPMENT OF A RIGID FLUORINATED TRANSPARENT PLASTIC MATERIAL SUITABLE FOR AIRCRAFT GLAZING AT ELEVATED TEMPERATURES

INVESTIGATOR: Harry D. Anspen  
Joseph J. Baron, Jr.

CONTRACT: AF 33(616)-3240

CONTRACTOR: General Aniline and Film Corporation

ABSTRACT: Fluoroacrylate esters were investigated to determine whether their polymers possessed properties suiting them to use as high temperature resistant, transparent, glazing materials.

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Two sheets (12" x 12" x 1/8") of methyl  $\alpha$ -fluoroacrylate were cast. The properties of these sheets were determined by tests run at Wright Air Development Center and by General Aniline and Film Corp. This polymer possesses good forming properties, excellent craze resistance, good stability toward light and heat [Polymethyl  $\alpha$ -fluoroacrylate is at least 100 times more stable than polymethyl  $\alpha$ -chloroacrylate at 191°C. (375°F.)] and is tougher than the chloro-analog. However, its heat distortion temperature (108°C.) is lower than that of polymethyl  $\alpha$ -chloroacrylate (135°C.).

Polymers and copolymers of methyl  $\alpha$ -fluoroacrylate, methyl  $\alpha$ -(trifluoromethyl) acrylate, methyl  $\alpha$ -chloroacrylate, methyl methacrylate, and glycidyl methacrylate were prepared and examined. Only cross-linked glycidyl methacrylate possessed a higher heat distortion temperature than methyl  $\alpha$ -chloroacrylate polymer. The glycidyl group seems to confer some heat stability to the acrylate over that possessed by a methyl ester.

Exploratory synthesis of methyl  $\alpha$ -chloro- $\beta,\beta$ -difluoroacrylate,  $\text{CF}_2=\text{CClCOOCH}_3$ , and methyl  $\alpha,\beta,\beta$ -trifluoroacrylate,  $\text{CF}_2=\text{CFCOOCH}_3$  were carried out. It is probable that the former compound has been isolated, but the last steps of the synthesis of the latter compound remain to be completed.

WADC TR 57-213  
ASTIA Document No. AD 130887

July 1957

**SUBJECT:** SILICONE INTERLAYER MATERIAL PROGRAM  
**INVESTIGATOR:** Keith E. Palmanteer  
Francis J. Campbell  
Thomas L. Laur  
**CONTRACT:** AF 33(600)-27183  
**CONTRACTOR:** Dow Corning Corporation  
**ABSTRACT:** Improvement of thermally stable silicone interlayer materials, Types K, L and M, was the object of this research. A method was developed to completely confine interlayer samples during heat aging. Falling ball impact and peel strength test methods for elevated temperature testing were developed. Work on Type L interlayer was ended due to its poor shelf life. Improvement in Type M interlayer material was achieved. Introduction of capillaries in Type M interlayer made it possible to prepare 12 x 12 inch laminates which withstood 400°F for 100 hours without forming defects.

Tensile strength, peel strength, bullet impact and falling ball impact properties of Type K and Type M interlayer were measured over a broad temperature range. Falling ball impact results



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showed PVB laminates to be better than Type K laminates over the temperature range of 30° to 97°F. Type K laminates had slightly higher impact strengths than Type M laminates over the temperature range of -65° to +400°F. Impact resistance was shown to be directly related to adhesion.

WADC TR 57-421  
ASTIA Document No. AD 142021

October 1957

**SUBJECT:** TRANSPARENT MATERIALS FOR AIRCRAFT ENCLOSURES WADC-  
UNIVERSITY OF DAYTON JOINT CONFERENCE

**INVESTIGATOR:** Robert E. Wittman

**ABSTRACT:** The purpose of this report is to make available all technical papers presented at the recent Seventh Conference on "Transparent Materials for Aircraft Enclosures." This conference was held for the exchange of knowledge on new developments and design concepts concerned with crew enclosures and to make known the state of the art with respect to transparent plastics, interlayer materials, and glass.

The papers contained herein have been reproduced directly from the original manuscripts.

WADC TR 57-447  
ASTIA Document No. AD 142045

October 1957

**SUBJECT:** ELEVATED- AND ROOM-TEMPERATURE PROPERTIES OF  
TRANSPARENT PRESTRETCHED PLEXIGLAS 55

**INVESTIGATOR:** G. M. Gynn  
J. A. Vanecho  
W. F. Simmons

**CONTRACT:** AF 33(616)-3215

**CONTRACTOR:** Battelle Memorial Institute

**ABSTRACT:** Creep and creep-rupture, short-time tensile, and deterioration data were obtained for prestretched Plexiglas 55, a transparent acrylic, sheet material. Data were obtained at 80, 160, 200, and 250 F. A comparison of properties is made with unstretched 5105XP, an experimental Plexiglas material.

The stress-rupture strength properties of the prestretched material were found to be superior to the unstretched 5105XP at 80, 160, and 200F. The prestretched Plexiglas deformed more than the unstretched material did, initially resulting in inferior creep-strength properties even though minimum creep rates were essentially the same. The short-time tensile strength of the prestretched Plexiglas is superior to the unstretched material at temperatures to about 170 F. At temperatures above 170F, the strength of the stretched material fell below that of the unstretched 5105XP Plexiglas.

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WADC TR 57-580  
ASTIA Document No. AD 142180

December 1957

**SUBJECT:** EVALUATION OF SELECTRON 400 TRANSPARENT PLASTIC MATERIAL  
**INVESTIGATOR:** Donald L. Schmidt  
**ABSTRACT:** Evaluation test data are presented on the optical, mechanical and thermal properties of a new experimental transparent plastic material, designated Selectron 400.

Selectron 400 has outstanding physical properties with respect to strength at high temperatures, heat distortion temperature and resistance to crazing. The material in its present stage of development has several undesirable characteristics such as brittleness, notch sensitivity and low impact strength.

Selectron 400 is suitable for use as monolithic canopy material in nonpressurized aircraft, and as surface sheets of laminated glazing assemblies on pressurized aircraft. However, Selectron 400 laminates with a silicone heat resistant interlayer have not yet been successfully fabricated and evaluated.

WADC TR 57-640  
ASTIA Document No. AD 142182

December 1957

**SUBJECT:** EVALUATION OF CAST UNFILLED TRIALLYL CYANURATE-POLYESTER LAMINATING RESINS  
**INVESTIGATOR:** D. F. Starks  
**ABSTRACT:** An exploratory evaluation of the properties of three cast unfilled triallyl cyanurate-polyester laminating resins was conducted to determine if this chemical type of resin would provide a useful material for further development as a heat resistant transparent glazing material for use in aircraft. The castings were made and all tests were conducted by the Plastic Products Section of the Organic Materials Branch, Materials Laboratory, Directorate of Laboratories, Wright Air Development Center. The tests were selected as those that would best present a basic representation of the merits of the resins. These included specific gravity, water immersion, heat distortion, tensile strength, izod impact and room and elevated temperature flexural tests. Optical quality castings with good light transmission were not obtained; however, it was considered that if the other properties were promising, the optical characteristics could be included in later developmental studies.

The triallyl cyanurate-polyester laminating resins are not 2-stage materials in the form evaluated but are cast and cured to the

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final form. They were compared to Selectron 400 which is a 2-stage material with satisfactory optical properties. Since most of the properties of the triallyl cyanurate-polyester laminating resins investigated were inferior to Selectron 400, development work as described in WADC TR 57-580 will be continued on Selectron 400.

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