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

TECHNICAL REPORT No. WADC-TR-53-373, SUPPL 10



5. JUNE 1964

3. b. AF MATERIALS LABORATORY
c. RESEARCH AND TECHNOLOGY DIVISION
d. AIR FORCE SYSTEMS COMMAND
a. WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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Statement A
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FOREWORD

This report was prepared by Materials Information Branch, Air Force Materials Laboratory, Research and Technology Division Wright-Patterson AFB, Ohio.

Technical reports published by the Air Force Materials Laboratory during the period 1 July 1962 - 30 June 1963 are abstracted herein. Reports on research conducted by the Air Force Materials Laboratory personnel as well as that conducted on contract are included.

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ABSTRACT

These reports cover basic and applied research in the materials area being conducted by the Metals & Ceramics Division, Non-Metallic Materials Division, Materials Physics Division, Manufacturing Technology Division, and Materials Applications Division of the Air Force Materials Laboratory.

This report has been reviewed and is approved.

A handwritten signature in cursive script, appearing to read 'D. A. Shinn', written in dark ink.

D. A. SHINN
CHIEF, MATERIALS INFORMATION BRANCH
MATERIALS APPLICATIONS DIVISION
AF MATERIALS LABORATORY

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I. Technical Reports and Technical Notes 1 July 1962 - 30 June 1963

BERYLLIUM

ASD TDR 62-509, Vol I

October 1962

SUBJECT: BERYLLIUM RESEARCH AND DEVELOPMENT PROGRAM
INVESTIGATOR: S. H. Gelles
CONTRACT: AF33(616)-7065, Nuclear Metals, Inc.
ABSTRACT: Summary of work conducted for the period April 1, 1960 through September 30, 1961, aimed at making Be more useful as an Air Force structural material. Report describes work in the field of purification, joining and flow and fracture. Volume I describes the preparation of high purity Be by the decomposition of BeI_2 , the joining of Be by ultrasonic welding and the resistance spot welding of Be. Also, a study of the distribution of BeO and voids in Be by replication electron microscopy, an investigation of the brittle behavior of Be by transmission electron microscopy, and a study of surface damage in Be are described.

ASD TDR 62-509, Vol II

April 1963

SUBJECT: BERYLLIUM RESEARCH AND DEVELOPMENT PROGRAM
INVESTIGATOR: S. H. Gelles
CONTRACT: AF33(616)-7065, Nuclear Metals, Inc.
ABSTRACT: The report summarizes the work conducted on the Beryllium Research and Development Program for the period April 1, 1960 through September 30, 1961. The aim of this program is to make beryllium more useful as an Air Force structural material.

The program was divided into eleven major efforts, eight of which were subcontracted and three carried out at the site of the prime contractor, Nuclear Metals, Inc. Detailed abstracts of each program are presented in their respective sections. In addition, work contemplated for a future program is described in a separate section.

Manuscript released October 1963 as a WADC Technical Report.

WADC TR 53-373 Sup 10

COATINGS - REFRACTORY METAL

ASD TDR 62-205

June 1962

SUBJECT: THE ANALYSIS OF THE BASIC FACTORS INVOLVED IN
THE PROTECTION OF TUNGSTEN AGAINST OXIDATION
INVESTIGATOR: M. G. Nicholas, et al
CONTRACT: AF33(616)-8175, General Telephone & Electronics
Laboratories, Inc.
ABSTRACT: The objective of this first phase of the contract was to decide on the areas in which lack of basic information would be likely to hamper the development of oxidation-resistant coatings for tungsten at temperatures above 1850°C. It was concluded from a preliminary review that five phenomena, in addition to refractoriness, would determine the usefulness of a coating; the rate of vaporization, breakaway, diffusion paths in ternary oxygen-metal-metal systems, coating-substrate reactions, and the rate of diffusion-controlled film growth. A literature review on each of these phenomena and trial calculations of the protectiveness of a number of coating systems enabled a number of conclusions to be drawn as to the areas in which additional basic information was most needed.

ASD TDR 63-212

March 1963

SUBJECT: THE EFFECT OF ADDITIVES ON ELECTRODEPOSITION
AND ELECTRODISSOLUTION OF METALS
INVESTIGATOR: T. C. Franklin, et al
CONTRACT: AF33(657)-7485, Baylor University
ABSTRACT: A preliminary investigation was made of the effect of additives on the electrodeposition of cadmium and on the electro-dissolution of nickel. The electrodeposition study was primarily limited to a study of techniques. These techniques included constant current pulses, cyclic voltammetry, microvolumetric measurement of hydrogen evolved, and hydrogen embrittlement. It was found that the current efficiency for the deposition of cadmium varied with concentration of additive in a regular pattern. Using different plating times, different base metals, different current densities, and in stirred and unstirred solutions the additive effects on current efficiency were similar. The electrodi dissolution study indicated that under some conditions an oxidizing additive acted on the metal in a first order corrosion reaction. This reaction was accelerated by the application of the anodic potential.

ASD TDR 62-432

July 1962

SUBJECT: INVESTIGATION OF DIFFUSION BARRIERS FOR REFRACTORY METALS

INVESTIGATOR: E. M. Passmore, J. E. Boyd, B. S. Lement

CONTRACT: AF33(616)-6354, ManLabs, Inc.

ABSTRACT: Thirty-three base barrier combinations involving the four refractory metals W, Mo, Ta, and Cb and 12 potential barrier metals having melting points over 1700°C were evaluated for relative interdiffusion behavior at 1700°C and 1800°C. The relative extent of interdiffusion decreases with increasing base-barrier solidus temperature, although wide variations occur for melting points up to 2100°C. Interdiffusion in the Mo-Cr combination was found to be substantially reduced by the presence of a Re barrier.

Re, Ru, and Ir barriers appear optimum for W base metal; W, Re, Ru, and Ir appear optimum for Ta; and W, Re, Os, and Zr appear optimum for Cb.

The thickness (X) of the total interdiffusion zone as well as of the intermediate phase layers in the W-Re, Mo-Re, Ta-Re, and Cb-Re combinations was found to increase with time (t) in accordance with the relationship $X^n = kt$, with values of n in the range 1.4 to 4.9.

ASD TDR 62-499

September 1962

SUBJECT: ALL-PURPOSE PLATING APPARATUS FOR RESEARCH ON VAPOR PHASE COATINGS

INVESTIGATOR: I. Marinow, C. A. Gellar, R. Bakish

CONTRACT: AF33(616)-8337, Alloyd Electronics Corp.

ABSTRACT: An apparatus has been developed to perform experimental vapor phase coating research which is capable of plating many of the metals and ceramic materials now deposited by the vapor phase process, and more importantly, it is capable of producing alloys and composite materials. The apparatus obtains the closest possible physical control of the deposition conditions, while emphasizing versatility and ease and simplicity of operation, so as to become an excellent vehicle for the evaluation of both plating compounds and deposited coatings. A variety of coating tasks was also undertaken to demonstrate the flexibility of this apparatus. The necessary laboratory facilities and support equipment to operate, maintain, and service the apparatus, and also to conduct pre-deposition bench testing of potential plating compounds, were also included in the program.

ASD TDR 62-888

November 1962

SUBJECT: WEATHER EXPOSURE DATA FOR HIGH TEMPERATURE COATINGS
ON CHROMIUM HOT-WORK TOOL STEELS

INVESTIGATOR: G. W. Lawless

ABSTRACT: Results are reported of a 4 1/2 year continuous exposure of coated Thermold J steel (SAE H-11) at Kure Beach, North Carolina. Eleven coatings were tested in various modifications, thicknesses, and pre-exposure thermal treatments. A nickel-zinc coating provided the best protection from corrosion during this type of exposure. A summary is included of the oxidation, mechanical, and short term corrosion tests conducted on these same coatings by Metcut Research Associates in 1957, during which investigation of the coated steel specimens were placed on exposure at Kure Beach.

ASD TDR 62-907

December 1962

SUBJECT: VAPOR PLATING OF ALUMINUM ON STEEL

INVESTIGATOR: J. J. Crosby

ABSTRACT: Adherent coatings of aluminum were deposited on low carbon steel specimens by vapor (gas) plating using the metal-organic compound tri-isobutyl aluminum. In the process, liquid tri-isobutyl aluminum was mixed with isobutylene and argon, vaporized, then thermally decomposed at a heated steel specimen into metallic aluminum plus volatile decomposition effluents. As a result of this reaction, the aluminum was deposited on the steel substrate and the volatile decomposition products were swept out the exhaust. Uniform, ductile coatings, 2 mils thick, were obtained. The capability of obtaining thicker deposits was indicated.

Significantly, the deposition of aluminum by vapor plating was not limited to mild steel or metal substrates in general. Other materials including copper, magnesium, titanium, stainless steel, glass, and graphite were also plated by this process.

ASD TDR 62-934

November 1962

SUBJECT: ADVANCEMENT OF HIGH TEMPERATURE PROTECTIVE
COATINGS FOR COLUMBIUM ALLOYS

INVESTIGATOR: J. D. Gadd, R. A. Jefferys

CONTRACT: AF33(657)-7396, Thompson Ramo Wooldridge, Inc.

ABSTRACT: The objective of this program was to improve and simplify the vacuum pack deposition process for forming the protective Cr-Ti-Si diffusion alloy coating on columbium alloys. Halide activation of the process resulted in significant reductions in the time,

ASD TDR 62-934 (Continued)

temperature and vacuum requirements for the coating process. The Cr-Ti coating can be formed on columbium alloys in 8 hours as low as 2200°F at system pressures up to 1.0-1.5 mm and alloyed with silicon in 4 hours as low as 2000°F at pressures up to 150 mm. Process scale up potential was demonstrated by deposition of the Cr-Ti-Si coating at several system pressures up to 150 mm; pressures which are easily attainable in large scale facilities. Conversion of the pack process to a completely vapor deposition process was investigated by closed retort and gas flow techniques. Cr-Ti-Si coatings were produced which were potentially comparable in protective properties to those deposited in the vacuum pack deposition process. At the conclusion of the coating development program, an evaluation of the Cr-Ti-Si coating was conducted on D-14, D-31, D-36, X-110 and Cb-752 alloys and more extensively on B-66 alloy. Reliable protection was provided by the Cr-Ti-Si coating for 150 hours at 1200-2500°F on the five columbium alloys and 1200-2600°F on B-66 alloy. Oxidation tests in air to 3000°F, thermal shock-erosion oxidation tests, cyclic oxidation + bend tests and stress oxidation + tensile tests were conducted on Cr-Ti-Si coated B-66 alloy. A detailed analysis of the work of the program and conclusions are presented.

ASD TDR 63-160

February 1963

SUBJECT: OXIDATION RESISTANT COATINGS FOR TANTALUM ALLOYS
AND OTHER METALS

INVESTIGATOR: L. Sama

CONTRACT: AF33(657)-7339, General Telephone & Electronics
Laboratories, Inc.

ABSTRACT: The effect of coating composition on structure and oxidation behavior was studied using a Sn-Al spray-slurry system with Ta alloys, Cb, W and Mo. At 3000°F, increased oxidation protection was related to the thickness of the tin-rich surface phase. Refractory metal powder additives served best to increase the thickness of this phase. Coating composition was optimized and an extensive evaluation was carried out with Ta-10W, Ta-30Cb-7.5V and Cb-5Zr. In oxidation tests at 1100 to over 3000°F protective life was significantly affected by substrate composition, coating thickness and thermal cycling. Tensile and stress rupture data on coated samples at 2000 to 3000°F correlated well with data in the literature for uncoated material. Preliminary studies were made of coating stability at air pressures in the micron to millimeter range. Oxidation effects were explored by electron probe. Oxidation data were obtained for several Ta alloys coated with Ti-Si.

April 1963

SUBJECT: COATINGS FOR TANTALUM-BASE ALLOYS
INVESTIGATOR: J. B. Hallowell, D. J. Maykuth, H. R. Ogden
CONTRACT: AF33(657)-7909, Battelle Memorial Institute
ABSTRACT: Silicide coatings, applied by pack cementation, were evaluated on a variety of tantalum alloys including Ta-10W, Ta-30Cb-7.5V, and Ta-8W-2Hf. Modification of the silicide, by diffusion with the underlying substrates, resulted in significant differences in oxidation behavior. Similar effects were obtained by incorporating aluminum, boron, and manganese additions in the silicide coating. Of the modifiers studied, vanadium was found to be most beneficial in improving the protective life of silicide coatings at low as well as high temperatures. Straight silicide coating of Ta-30Cb-7.5V alloy had no detrimental effect on room-temperature properties and provided useful lives in dynamic as well as static oxidation testing at temperatures to 3000°F. Modifications of the silicide with boron and vanadium show further improvements in static oxidation performance at temperatures from at least 1800 through 2900°F.

CORROSION

WADD TR 61-123, Part II

June 1962

SUBJECT: PROPERTIES OF YTTRIUM AND THE RARE EARTH METALS,
EFFECT OF MINOR ADDITIONS TO NIOBIUM-BASE ALLOYS
ON ELEVATED TEMPERATURE OXIDATION RESISTANCE

INVESTIGATOR: C. G. Kirkpatrick

CONTRACT: AF33(616)-6829, Research Chemicals Corp.

ABSTRACT: Approximately 180 compositions in 19 niobium based alloy types were prepared for study. Atmospheric corrosion tests were completed for 9 series of alloys. Improved corrosion resistance is indicated for a niobium alloy containing 7% titanium, 20% tungsten, and 3% molybdenum when rare earth metals are added. Possible improvement, on the addition of rare earths, is also suggested for a niobium alloy containing 11.3% titanium and 7.9% molybdenum. Metallurgical investigations of two alloy series failed to indicate correlation between metal interface movement or micro-hardness values with corrosion resistance as established by weight gain measurements.

ASD TR 61-713

May 1962

SUBJECT: RESEARCH ON THE BASIC NATURE OF STRESS CORROSION
FOR VARIOUS STRUCTURAL ALLOYS AT ROOM AND
ELEVATED TEMPERATURE

INVESTIGATOR: F. A. Crossley

CONTRACT: AF33(616)-7612, Armour Research Foundation

ABSTRACT: The relationship between quantity of ASTM sea salt, varying from 0.0002 to 0.02 g/sq in., and thickness of anodized film--0, 2, and 8 micro-inches--in elevated temperature stress-corrosion cracking of the titanium alloys Ti-6Al-4V and B-12OVCA was investigated.

Exposure conditions were 800°F - 25,000 psi - 190 hr. for the former alloy and 600°F - 100,000 psi - 190 hr. for the latter. Damage was progressively greater with increasing quantity of salt. The anodized films appeared to be of no benefit to the Ti-6Al-4V alloy; however, it appeared that limited protection was afforded B-12OVCA.

The alloys: 2024-T86, 7075-T6, ZK-60A-T5, 17-7PH RH 950, and B-12OVCA in two conditions of grain size and two conditions of surface treatment were tested as follows: tensile test in air at room temperature, and in distilled water and ASTM sea water at 32°, 75°, and 212°F; and statically loaded at 90% of the yield strength in media of air, water, and ASTM sea water at room temperature.

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ASD TR 61-713, Part II

February 1963

SUBJECT: RESEARCH ON THE BASIC NATURE OF STRESS CORROSION
FOR VARIOUS STRUCTURAL ALLOYS AT ROOM AND
ELEVATED TEMPERATURE

INVESTIGATOR: F. A. Crossley

CONTRACT: AF33(616)-7612, Armour Research Foundation

ABSTRACT: Program objectives were: to study effect of micro-
structure on susceptibility to stress-corrosion cracking in the short-
transverse direction of 7075-T6 aluminum alloy; and to study the
kinetics of stress-corrosion cracking at high temperature of candidate
materials for structural applications in the tri-sonic transport in the
presence of sea salt. It was hypothesized that the poor resistance to
stress-corrosion of 7075-T6 was due to a layered type microstructure.
Short life was associated with layered type of grain structure, and
long life with irregular or equiaxed grain structures. The experimental
results gave good support to the hypothesis.

Preliminary trials for determining stress-corrosion
kinetics by means of precision measurement of resistance show promise.

ASD TDR 62-965

March 1963

SUBJECT: EFFECTS OF LIQUID AND VAPOR CESIUM ON CONTAINER
METALS

INVESTIGATOR: W. T. Chandler, N. J. Hoffman

CONTRACT: AF33(616)-8435, Rocketdyne

ABSTRACT: The results from a program to investigate the
compatibility of cesium with 310 stainless steel, Inconel-X, zirconium,
hafnium, columbium, columbium - 1 percent zirconium alloy, molybdenum,
tantalum, and tungsten are discussed. Static capsule tests at 1600°F
and reflux capsule tests at 1800°F and 2500°F were carried out for
periods up to 720 hours. No significant corrosion was found after the
1600°F tests. Molybdenum dissolved significantly in the refluxing
tests. The columbium - 1 percent zirconium alloy was the best material
in the 2500°F reflux test, but was attacked inter-granularly at the
lower temperature. The latter attack was accompanied by the selective
leaching out of zirconium, possibly due to fluxing of ZrO by Cs₂O.

CREEP

ASD TR 61-203, Part II

March 1963

SUBJECT: THE MECHANICAL PROPERTIES OF TANTALUM WITH
SPECIAL REFERENCE TO THE DUCTILE-BRITTLE TRANSITION
INVESTIGATOR: G. T. Murray, R. A. Burn
CONTRACT: AF33(616)-7173, Materials Research Corp.
ABSTRACT: The lower yield stress-grain size relationship
was studied in tantalum of two compositions; one consisting of a total
interstitial content of 77 ppm with the major constituent being oxygen
and the other containing carbon (116 ppm) in which part of the carbon
was in the form of finely dispersed carbide precipitate. The carbon
containing material showed the lowest stresses. It was concluded that
oxygen is much more effective than carbon in restricting dislocation
movement. The effect of the carbide precipitate was to increase the
work hardening rate. The flow stresses even in the presence of the
carbide phase were lower than those observed for commercial tantalum.

k_y and σ_i values were computed.

ASD TDR 62-710

August 1962

SUBJECT: STRAIN DISTRIBUTION ON NOTCHED PLATE SPECIMENS
AT CREEP TEMPERATURES
INVESTIGATOR: H. R. Voorhees, J. W. Freeman
CONTRACT: AF33(616)-7416, University of Michigan
ABSTRACT: Resistance strain gages and grids of vapor-
deposited metal were evaluated for measuring strains at loading and
during creep in plates with edge notches. Foil strain gages mounted
with thermal-setting cements remained usable to 0.5% or higher strain
at 500°F, but indicated creep was first less than, and later more than,
optical-extensometer readings. Indications from gages mounted with
ceramic cement drifted 10 μ in/in/hr at 1200° after several hundred
hours at temperature, but agreed with the extensometer to 1% strain.
Qualitative measurements were made during leading (K_t 1.8 and 3.6)
and for creep (K_t 1.8) of M-470 alloy at 500°F. Plastic strains were
too small to determine by the grid lines used.

ASD TDR 62-930

March 1963

SUBJECT: THE INVESTIGATION OF THE EFFECTS OF LOADING RATE
AND STRESS CONCENTRATION FACTORS ON THE NOTCH
PROPERTIES OF THREE SHEET ALLOYS AT SUBZERO
TEMPERATURES

WADC TR 53-373 Sup 10

ASD TDR 62-930 (Continued)

INVESTIGATOR: A. W. Brisbane
ABSTRACT: The effect of theoretical stress concentration factors and loading rates at 26°, -78°, and -196°C are presented for 301XH stainless steel, Rene-41, and Vasco Jet-1000 sheet materials.

The unnotched specimens, tested at room temperature, were not appreciably affected by loading rate; however, some effects were observed for the specimens tested at sub-zero temperatures. The loading rate had some effect on the notched tensile specimens at all test temperatures. The temperature effect was more pronounced on the notched specimen at the fastest loading rate. In most instances, the tensile strength increased as the temperature decreased. The notch to unnotched strength ratio decreased as the stress concentration factor increased. The 301XH stainless steel was the least notch sensitive.

The amount of martensite transformed in the notched 301XH stainless steel specimens varied with the notch geometry and loading rate, with the exception of the specimen with a K_t of 12.8 at -196°C.

ASD TDR 62-968

November 1962

SUBJECT: STRAIN AGING AND DELAYED FAILURE IN HIGH-STRENGTH STEELS
INVESTIGATOR: E. A. Steigerwald, G. L. Hanna
CONTRACT: AF33(657)-7512, Thompson Ramo Wooldridge, Inc.
ABSTRACT: Spontaneous strain aging which occurs during tensile testing was examined for several high-strength steels. The results of smooth and notch tensile tests indicated that significant strain aging effects occurred in most high-strength steels in the 300°F. to 800°F. temperature range and this behavior was analogous to "blue brittleness" in mild steels.

Constant load, stress rupture tests were conducted on the steels to determine the possible relationship between strain-aging embrittlement and delayed failure. Only the 300 M steel tested at 400°F exhibited an appreciable degree of delayed failure. This embrittlement was extremely sensitive to test environment and was eliminated when tests were conducted in argon. Although strain aging was not a sufficient condition to initiate delayed failure, it appeared to increase the severity of the environmental effects in the particular range where sufficient interstitial mobility existed.

May 1963

SUBJECT: RECOVERY BEHAVIOR OF COLD-WORKED METALS
INVESTIGATOR: T. E. Tietz, et al
CONTRACT: AF33(616)-8346, Lockheed Missiles & Space Co.
ABSTRACT: Recovery of tensile flow stress of four binary aluminum alloys and of the high-purity base aluminum was studied under the no-load conditions at temperatures of 80°, 120°, 160°, and 200°C, and under conditions of creep strain at 160°C for recovery times up to 1000 hours. At the three higher recovery temperatures, 120°, 160°, and 200°C, and for the longer recovery times, the alloys Al-Mg, Al-Zn, and Al-Cu all experienced a greater degree of recovery than the high-purity base aluminum. Of these three alloys, the Al-Cu alloy clearly exhibited the greater degree of recovery. The Al-Ag alloy was excluded from the comparison because of an apparent strengthening process which occurred during recovery. All the alloys showed a strong increase in flow-stress recovery during creep strain over that experienced during no-load recovery; however, the relative order for recovery for the different alloys did not change. A 10% prestrain shifted the ductile-brittle transition temperature, as indicated by tensile elongation, from 10°C to about -22°C. A 100-hr recovery anneal at 801°C was shown to shift the transition temperature to about -5°C, the major shift occurring during the first hour of recovery.

FATIGUE

ASD TDR 62-186

February 1963

SUBJECT: ULTRASONIC METHODS IN THE STUDY OF FATIGUE AND DEFORMATION IN SINGLE CRYSTALS OF ALUMINUM AND SODIUM CHLORIDE

INVESTIGATOR: B. Chick, et al

CONTRACT: AF33(616)-6945, Brown university

ABSTRACT: The use of ultrasonic methods for studying defect formation and its consequences in connection with stress cycling and deformation in aluminum and sodium chloride single crystals is the subject of this report. The observed ultrasonic changes appeared closely associated with changes in dislocation behavior. To establish this deformation, experiments were used in such a way that results could be related to the behavior of the slip systems and to their orientation. Aluminum single crystals were used for these experiments. Simultaneous measurements of attenuation and velocity changes were made continuously during tensile deformation. In the very early stages of deformation, for all orientations, an increase in attenuation is observed before microscopic yield. Easy glide is observed by the attenuation and velocity changes. Measurements in tension have been made for the purpose of comparing the dislocation damping and pinning effects in an ionic crystal with those in a metal. Important equipment improvements have been made for automatic recording of attenuation and velocity measurements.

ASD TDR 62-186, Part II

February 1963

SUBJECT: ULTRASONIC METHODS IN THE STUDY OF FATIGUE AND DEFORMATION IN SINGLE CRYSTALS

INVESTIGATOR: B. Chick, et al

CONTRACT: AF33(657)-8324, Brown University

ABSTRACT: Ultrasonic methods for studying defect formation and motion in connection with deformation and stress cycling experiments in aluminum and sodium chloride single crystals is the subject of this report. Large single crystals of sodium chloride, deformed in tension, were measured similarly to aluminum for the purpose of comparing the dislocation damping and recovery effects in an ionic crystal with those in a metal. Also, there was significant information from electrical conductivity measurements made concurrently with attenuation and velocity measurements. Comparison of Al single crystals at 195°K with room temperature resulted in an interesting "threshold effect". Al single crystals were also stress cycled in tension and compression at levels much higher relative to yield stress

ASD TDR 62-186, Part II (Continued)

and breaking stress than polycrystalline samples, and at the same time the stress level can be raised in steps during cycling to 1/2-2 times the stress required to break the single crystal sample. The trial model automatic recording time echo (or velocity) measurement unit test gave no change in center frequency or bandwidth over a 40db range of gain control nor an artificial change of 40db in signal level to produce a detectable change in measured time.

ASD TDR 62-223

April 1962

SUBJECT: INITIAL YIELDING AND FRACTURE IN NOTCHED SHEET
MOLYBDENUM
INVESTIGATOR: R. T. Ault, J. W. Spretnak
ABSTRACT: Initial yielding and fracture initiation behavior of wrought stress-relieved and recrystallized molybdenum, and the effect of notch severity and grain size on the nature of plastic flow were investigated. Analyses of unnotched tensile properties according to Griffith-Orowan and Cottrell theories showed that the effective surface energies for crack propagation and crack initiation are the same order of magnitude. Experimentally, no microcracks were found. Initial yielding at the notch root occurred at the same stress level independent of the magnitude of the stress concentration factor. Limited dislocation density measurements revealed that a quantitative measurement of strain at the root of a sharp notch was difficult to obtain by etch pit techniques.

ASD TDR 63-267

March 1963

SUBJECT: ASPECTS OF RELIABILITY UNDER CONDITIONS OF
ELEVATED TEMPERATURE CREEP AND FATIGUE
INVESTIGATOR: A. M. Freudenthal
CONTRACT: AF 33(616)-6288, Ohio State University
ABSTRACT: The solution of the problem of attaining adequate safety and reliability in supersonic aircraft structures operating under conditions under which the damaging effects of cycle sensitivity (fatigue) and time-sensitivity (creep) of the structural material combined in gradually reducing the resistance of the structure requires the development of simplified procedures for the evaluation of the combined damage accumulation, which embody both the physical and probabilistic aspects of design.

The present report attempts to develop the basis for an approach to the solution of this problem, for which at present

ASD TDR 63-267 (Continued)

no adequate experimental information exists, and one of its purposes is to provide the guidelines for the planning of tests and experiments, the results of which would be relevant for structural design.

ASD TDR 62-351

August 1962

SUBJECT: INVESTIGATION OF NOTCH FATIGUE BEHAVIOR OF CERTAIN ALLOYS IN THE TEMPERATURE RANGE OF ROOM TEMPERATURE TO -423°F
INVESTIGATOR: D. N. Gideon, R. J. Favor, A. Koppenhafer
CONTRACT: AF33(616)-6888, Battelle Memorial Institute
ABSTRACT: The notched fatigue behavior of 13 alloys has been investigated in the temperature range room temperature to -423°F and in the lifetime range 10^4 to 10^6 cycles. The alloys studied were chosen on the basis of actual or potential application in missile and spacecraft cryogenic systems.

The materials were in sheet form and were fatigued in fully reversed bending experiments. V-notches in the edges of the specimens had theoretical stress concentration factors of about 3.1 and 6.4. The results are presented as S-N plots and comparisons with unnotched fatigue strengths are made in tables of notch sensitivity values. S-N plots of the unnotched fatigue data obtained in the previous year are included for convenience.

ASD TDR 62-354, Part II

January 1963

SUBJECT: WORK HARDENING MECHANISMS IN BODY-CENTERED-CUBIC METALS
INVESTIGATOR: D. P. Gregory
CONTRACT: AF33(616)-7855, Pratt & Whitney Aircraft Corp.
ABSTRACT: This report presents results of a continued study of work hardening mechanisms in columbium and tungsten at low temperatures, preliminary studies of their alloys at elevated temperatures, and a literature survey of the deformation of body-centered-cubic metals.

ASD TDR 62-785

August 1962

SUBJECT: THE EFFECT OF SIZE AND STRESS HISTORY ON FATIGUE CRACK INITIATION AND PROPAGATION
INVESTIGATOR: W. Weibull
CONTRACT: AF61(052)-522, Bockamollan, Brosarps Station, Sweden

ASD TDR 62-785 (Continued)

ABSTRACT: This investigation deals with the effect of size and pre-loading on the duration N_i of the crack initiation period, as well as the crack propagation period. Geometrically similar sheet specimens of two aluminum alloys were used. Each size exhibited its individual S- N_i curve. A static preload increased the N_i from 11 to 205 kc.

Equations relating crack length to number of cycles are derived for constant stress cycle and constant load cycle. The formulas are verified by tests for various combinations of material, size, and stress amplitude. For constant stress, the rate of crack growth was found, independently of crack length, to be constant after a certain transition period which is dependent on the duration of the preceding initiation period. For constant load cycle, the propagation period starts with a transition period followed by one or more stable propagation periods, their number depending on load magnitude. Failure analysis showed these periods corresponding to different fatigue mechanisms. It is concluded that total fatigue life cannot be predicted without considering separately the parts of which it is composed.

ASD TDR 63-116

May 1963

SUBJECT: MECHANICAL PROPERTIES OF AM-350, POTOMAC A, POTOMAC M, AND VASCO JET-1000 STEEL ALLOYS IN THE ANNEALED CONDITION

INVESTIGATOR: R. G. Henning, A. W. Brisbane

ABSTRACT: Mechanical properties of three hot-worked steels and one precipitation-hardening stainless steel were obtained. Properties obtained were tensile, compression, sheet single shear, bearing, and 105-degree-angle bend tests.

Tests were conducted at temperatures of 80°, 400°, 600°, 800°, 1000°, and 1200°F. Stressed and non-stressed exposure tests were conducted only at 600°, 800°, and 1000°F. All properties were obtained from the longitudinal direction of the material except three tensile specimens from each material in the transverse direction, which were tested only at 80°F. Data obtained are presented graphically. Metallurgical histories and chemical analyses are also included.

April 1963

SUBJECT: FATIGUE OF COPPER-ZINC ALLOYS AT 100°K
INVESTIGATOR: J. A. Roberson, J. C. Grosskreutz
ABSTRACT: Fatigue tests were conducted to determine the S-N diagrams for a series of copper-zinc alloys at 100°K. An attempt was made to relate the endurance limit behavior to both stacking fault energy and yield strength, but no simple relationship was found. It is suggested that high stacking fault energy increases the cyclic work hardening rate by increasing the probability of dislocation intersection and jog formation. The increase in work hardening rate is reflected in a decrease in plastic strain amplitude, and a subsequent increase in fatigue life. These arguments are bounded on one side by considerations of the yield strength of the alloys, and on the other side by consideration of the dominant mechanisms operative in short and long life fatigue.

HIGH STRENGTH METALLIC MATERIALS

ASD TDR 62-396

May 1962

SUBJECT: MATERIALS CENTRAL-ASM METALLURGICAL EDUCATIONAL LECTURES
INVESTIGATOR: M. G. Fontana, et al
ABSTRACT: This report consists of a series of lectures on failure analysis. Causes, cures, and preventive measures of corrosion are discussed. The general types of elevated-temperature service failures are described and the influence of static and dynamic loading, thermal cycling corrosion, and inert environments are discussed herein. The sensitivity of present high strength structural alloys to brittle failure under various environmental and state of stress conditions is included. Present Air Force research efforts on the characteristic behavior of high strength materials are reviewed. This includes the relationship between mechanical wear and lubrication. A description of the interaction between two contacting solids causing wear and the basic principles of lubrication and lubrication techniques are given.

ASD TDR 62-520

November 1962

SUBJECT: THE EFFECT OF ADDITION ELEMENTS ON THE RATE OF BETA GRAIN GROWTH IN ALPHA-BETA AND BETA TITANIUM ALLOYS
INVESTIGATOR: F. A. Crossley
CONTRACT: AF33(616)-7687, Armour Research Foundation
ABSTRACT: Minor alloying additions have been evaluated for inhibiting beta grain growth in unalloyed titanium, B-120VCA, and Ti-8Al-1Mo-1V. Additions were: 0.11B, 0.05S, 0.45Ag-0.74Hf, 0.57Ba, 0.36Sr, and a multiple addition 0.03S-0.12Ba-0.15Hf-0.07Sr-0.01B. The boron and sulfur additions produced particulate phases in the alloys; and only these additions were effective in titanium and Ti-8Al-1Mo-1V. The barium addition, which apparently was soluble, was only slightly less effective than additions of boron or sulfur in B-120VCA. Room temperature tensile properties gave no indications of detriment to mechanical properties due to any of the additions. Strontium appeared to be beneficial to ductility through a scavenging effect.

ASD TDR 62-534

October 1962

SUBJECT: INVESTIGATION OF FACTORS RELATED TO AN UNKNOWN PHASE IN B120 VCA TITANIUM ALLOY
INVESTIGATOR: D. W. Levinson

ASD TDR 62-534 (Continued)

CONTRACT: AF33(616)-8149, Armour Research Foundation
ABSTRACT: A study was made of the needle-like precipitate which had been observed to occur in B120 VCA as a result of welding or of solution treating at temperatures above 1100°C. The phase was found to be the alpha phase, stabilized by oxygen. Under conditions of strict oxygen exclusion, the alpha needles do not appear.

The embrittlement of material aged after either welding or overheating during solution treatment was found to be unaffected by the alpha needles. Embrittlement occurred whether or not needles were present in the microstructure. Some evidence is presented which implies that omega phase precipitation may produce the embrittlement.

ASD TDR 62-561

October 1962

SUBJECT: DIFFUSION IN TITANIUM AND TITANIUM ALLOYS
INVESTIGATOR: R. P. Elliot
CONTRACT: AF33(616)-7656, Armour Research Foundation
ABSTRACT: The self-diffusion of Ti and the interdiffusion of Al, Zr, Mo, V, and O in Ti have been investigated in the temperature range 600° - 1300°C. Diffusion couples were prepared by roll-bonding or press-bonding. Electron microprobe methods were used to determine the penetration of the substitutionally dissolved penetration of interstitially dissolved oxygen.

The electron microprobe analysis could not be used to determine the penetration curves of Al in Ti because of the very high absorption of characteristic Al x-radiation by Ti.

The self-diffusion of Ti was investigated by studying penetration of Ti⁴⁴, formed by bombarding scandium with protons. Diffusion couples were formed by dissolving Ti⁴⁴-enriched TiO₂ into the Ti.

The diffusion equations for Mo and V in -Ti, and for the self-diffusion of Ti must be considered preliminary.

ASD TDR 62-692

February 1963

SUBJECT: RESEARCH IN THE MECHANISM OF STRENGTHENING IN
AUSFORMED STEEL
INVESTIGATOR: F. Martin, et al
CONTRACT: AF33(616)-8116, Aeronutronic Div., Ford Motor Co.
ABSTRACT: This investigation was conducted to define the

ASD TDR 62-692 (Continued)

mechanism responsible for the strengthening increase imparted by deformation of metastable austenite. The relationships of mechanical properties to extent and temperature of deformation were developed for Type H-11 hot work die steel. The influence of nonmartensitic decomposition products on ausformed properties were determined. The microstructures were studied by thin-film electron transmission methods for observations of martensite morphology and substructures and precipitates. Measurements of subcell sizes and distribution of precipitates were made for comparison with observed mechanical properties through application of dislocation theory. The observed dislocation density and carbide dispersion indicate that the strengthening mechanism active in both ausformed and conventionally heat treated martensitic steels is some combined effect of dislocation-precipitate and dislocation-dislocation interaction.

ASD TDR 62-734

November 1962

SUBJECT: INVESTIGATION OF THE STRENGTHENING MECHANISMS OF DISPERSION-STRENGTHENED ALLOYS
INVESTIGATOR: R. J. Murphy, V. Sadagopan, N. J. Grant
CONTRACT: AF33(616)-8085, Massachusetts Institute of Technology
ABSTRACT: In an effort to produce dispersion-strengthened alloys with higher strength properties than those reported for oxide dispersion-strengthened systems, the systems Ni-TiB₂ and Ni-TiC were selected. Dilute binary alloys of carbon, boron, and titanium in nickel were prepared both in bar and powder form. Diffusion couples between dilute alloys of Ni-C and Ni-Ti as well as between Ni-B and Ni-Ti were examined in a preliminary way. Lack of appreciable diffusion across the boundary motivated the preparation of alloys by means of powder interactions. Positive interactions were observed but measurements of intermetallic particle size titanium alloy shows more extensive diffusion interaction, suggesting use of less dilute alloys.

Transmission electron microscopy is being used to observe strain fields associated with dispersed particles, comparing observations in aged alloys, oxide dispersion-strengthened alloys, and intermetallic dispersion-strengthened alloys.

ASD TDR 62-869

April 1963

SUBJECT: DEVELOPMENT OF A NICKEL BASE ALLOY SHEET FOR HIGH TEMPERATURE APPLICATION
INVESTIGATOR: H. Greenewald, Jr., T. J. Riley
CONTRACT: AF33(616)-7999, Chance Vought Corp.

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ASD TDR 62-869 (Continued)

ABSTRACT: The objective of this contract was to develop 15 to 30 mil nickel alloy sheet having 50,000 psi tensile strength at 1900°F, having good corrosion (oxidation) resistance, and good ductility. This objective was essentially attained by developing a new process of directly rolling thin cast slabs of nickel base alloy into sheet on a specially designed rigid rolling mill. Two pre-existing nickel base casting alloys and a series of experimental compositions obtained by modifying the two starting alloys were initially investigated in the as cast condition in this program. The two starting alloys were Inco 713c and NASA's TaZ8 alloy. Of the new experimental compositions, alloy No. 429 (a Ta-W-C-Cr modified Inco 713c) has 1900°F tensile strength exceeding 50,000 psi in the as cast condition, as does the TaZ8 alloy. Inco 713c has a tensile strength of about 40,000 psi in the as cast condition. Hot rolled Inco 713c indicated good room temperature ductility. The oxidation resistance of TaZ8 alloy is adequate for limited times at 1900°F; that of No. 429 alloy is substantially better; and that of Inco 713c is best of the three.

ASD TDR 62-1034, Part I

April 1963

SUBJECT: RESEARCH AND APPLICATION ENGINEERING TO DETERMINE THE EFFECT OF PROCESSING VARIABLES ON CRACK PROPAGATION OF HIGH-STRENGTH STEELS AND TITANIUM

INVESTIGATOR: B. R. Banerjee, J. J. Hauser

CONTRACT: AF33(616)-8156, Crucible Steel Co. of America

ABSTRACT: This report describes in detail the fine-structural characterizing of several high-strength steels (AISI 4340, Type H-11 "Crucible 218", Type 422 stainless, work-hardened Type 301 stainless, PH15-7Mo semiaustenitic stainless, and B-120VCA beta titanium alloy) and gives the relationship of these structures to smooth and notch strength properties. The fine structures of these materials were characterized and correlated with strength and fracture properties to an extent never before achieved.

ASD TDR 62-1064

April 1963

SUBJECT: THE RELATIONSHIP OF MICROSTRUCTURE TO STRENGTH AND TOUGHNESS IN HIGH STRENGTH STEEL

INVESTIGATOR: Y. H. Liu, et al

CONTRACT: AF33(616)-7780, Ohio State University

ABSTRACT: This research deals with the relationship between the microstructural features and the susceptibility to brittle fracture of six selected commercially produced ultra-high strength steels. The

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parameters selected for the susceptibility to brittle fracture were the susceptibility to plastic instability, indicative of the susceptibility to ductile fracture initiation, and controlled total available energy bend test to determine the conditions for acceleration of crack propagation and the energy absorbed in rapid crack propagation. The microstructures were studied by standard techniques of optical microscopy, electron microscopy, and electron diffraction.

REFRACTORY METAL APPLIED RESEARCH

WADC TR 59-575, Part III

July 1962

SUBJECT: OXIDATION OF TUNGSTEN AND TUNGSTEN BASED ALLOYS
INVESTIGATOR: E. A. Gulbransen, et al
CONTRACT: AF33(616)-7888, Westinghouse Electric Corp.
ABSTRACT: This paper describes new experimental work on the oxidation of tungsten and a 50 w/o tantalum-tungsten alloy. The rates of oxidation of tungsten were determined from 1150° to 1615°C in oxygen pressures of 2 to 100 Torr. Very high rates of oxidation or surface recession rates were found above 1200°C. An exponential temperature behavior for the rate of oxidation was found for all the pressures studied. A heat of activation of 14,300 calories per mole was estimated from the data. A study of the effect of pressure on the rate of oxidation showed the following empirical equation could explain the weight loss data, $dw/dt = Kp^{1.22}$. Theoretical analysis of the data using the absolute reaction rate theory suggested that the rate of oxidation of tungsten was limited by a mobile adsorption process of oxygen onto a tungsten surface already covered by a surface layer of oxide. Oxidation of a 50 w/o tantalum-tungsten alloy was studied over the temperature range of 1068°C to 1458°C at 152 Torr oxygen pressure. The results showed that a protective scale was formed on this alloy. A special 250 KV electron diffraction camera was developed for the study of the tungsten-gas reacting interface.

WADD TR 60-37, Part III

November 1962

SUBJECT: PHYSICAL METALLURGY OF TUNGSTEN AND TUNGSTEN BASE ALLOYS
INVESTIGATOR: N. J. Bloomfield, H. G. Sell, et al
CONTRACT: AF33(616)-6933, Westinghouse Lamp Division
ABSTRACT: The tensile properties of polycrystalline tungsten were investigated. Ductility decreased between 2000°F and 4000°F, and it was found that annealing temperature has a greater effect on the D. -B. transition temperature than does grain size. Single crystals with a (100) orientation were grown by electron beam floating zone melting. The tensile deformation behavior of these crystals were studied from -196°C to 800°C. Internal friction measurements were made on single crystals between 20°C and 600°C and it was attempted to correlate damping peaks found at about 300°C and 475°C with interstitial effects, carbon dosing experiments, and yield point phenomena. Single crystals were worked by rolling and their recrystallization response was investigated as a function of cold work and working temperature. Twin morphology was thoroughly studied. Binary solid solution

WADD TR 60-37, Part III (Continued)

alloy single crystals of W-Ta were produced by electron beam floating zone melting from compacted ingots. Alloy single crystals containing .25% and 4% tantalum were worked and the recrystallization response was determined to be 400°C to 600°C higher than that of worked pure tungsten single crystals. The tensile strength of sintered and swaged W-TaC alloys was determined at 3000°F and 3500°F as a function of alloy composition (0.1-1%). Also measured at 3000°F and 3500°F were the tensile strengths of the systems W-2%ThO₂ and W-.5%HfO₂.

WADD TR 60-132, Part II

September 1962

SUBJECT: REFRACTORY METAL CONSTITUTION DIAGRAMS
INVESTIGATOR: E. J. Rapperport, et al
CONTRACT: AF33(616)-7157, Nuclear Metals, Inc.
ABSTRACT: Data on six binary constitution diagrams and two ternary constitution diagrams of some of the refractory metals are presented. The binary diagrams include Mo-Os, Ta-Ir, Ta--Rh, Ta-Zr, W-Ir, and W-Rh; the ternaries are Mo-Hf-Re- and Ta-W-Zr.

Care was taken to obtain reliable diagrams. In particular, the purity of the constituents (99.9 percent plus) was protected at all times, and the temperatures were measured to an accuracy of $\pm 20^\circ\text{C}$.

WADD TR 61-134, Part II

April 1963

SUBJECT: RESEARCH ON WORKABLE REFRACTORY ALLOYS OF TUNGSTEN, TANTALUM, MOLYBDENUM, AND COLUMBIUM
INVESTIGATOR: R. C. Westgren, et al
CONTRACT: AF33(616)-8135, Crucible Steel Co. of America
ABSTRACT: Under a previous contract, the W-Ta-Mo-Cb alloy system was investigated, and several tungsten- and tantalum-rich alloys were developed and evaluated in the form of extruded bars. Many of these alloys exhibited very high strengths at 3000°F; in fact, the tensile strengths of some alloys were in excess of 60,000 psi. The present work was a continuation of this effort and was aimed at producing and evaluating alloys from the W-Ta-Mo-Cb system in the form of sheet (Phase I) and increasing the high-temperature strength of alloys of this type by the formation of dispersed carbides (Phase II). For Phase I, small cylindrical ingots of six selected alloys and unalloyed tungsten were consumably vacuum arc-melted by a multiple electrode technique and successfully extruded to sheet bars. Two alloys and unalloyed tungsten were rolled to sheet and recrystallization temperatures

WADD TR 61-134, Part II (Continued)

bend transition temperatures, and high-temperature tensile properties were determined. The other four alloys could not be rolled to sheet by the techniques attempted in this program. Experiments under Phase II resulted in the development of a successful technique for consumably vacuum arc melting 88W-12Cb alloys that contain small amounts of vanadium, zirconium, and carbon. In the as-extruded condition, the alloys showed 3500°F tensile strengths of 49,000 to 57,000 psi - this constituted a two-fold increase in strength above that of the base solid solution composition.

WADD TR 61-181

October 1962

SUBJECT: SUBSTRUCTURE AND MECHANICAL PROPERTIES OF REFRACTORY METALS
INVESTIGATOR: B. S. Lement, et al
CONTRACT: AF33(616)-6838, Manufacturing Laboratories, Inc.
ABSTRACT: Coordinated program on substructural characteristics of W, Mo, Ta and Cb were carried out by ManLabs, M.I.T., Rutgers, U. Liverpool (England) and U. Cambridge (England) by means of advanced microscopic, x-ray diffraction, and mechanical testing techniques. Initial increase in hardness during recovery of heavily deformed W wire is ascribed to segregation of interstitials to dislocations and to micro-twinning. Sharp drop in ductility on 1100°C anneal is attributed to rise in transition temperature to above room temperature due to increase in fiber width. Competing processes of polygonization and strain induced fiber boundary migration occur up to 1400°C. In Ta single crystals, the maximum principal strain changes from positive to negative on deforming below and above the upper yield point respectively. Binding energy between dislocations and interstitials in Ta is about 0.53 e.v. For low interstitial contents, k_y increases as the dislocation density is decreased by higher annealing above recrystallization; for high interstitial contents, k_y is not affected. The activation energy for annealing-out of dislocation loops in Ta and Cb is about 3 e.v.

ASD TR 61-383

February 1962

SUBJECT: GRAIN BOUNDARY SLIDING IN ALUMINUM AND ITS BINARY ALLOYS
INVESTIGATOR: A. Mullendore, Y. Ishida, N. J. Grant
CONTRACT: AF33(616)-5926, Massachusetts Inst. of Technology
ABSTRACT: A study was made of grain boundary sliding in aluminum-magnesium bicrystals to test a mechanism for sliding proposed in previous work. The mechanism relates sliding to slip crossing grain

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boundaries. An investigation was also made of the effect of a second phase existing in various distributions on grain boundary sliding.

Al-3% Cu was used for this study. It was found that for tests at similar strain rates, very large differences in grain boundary sliding rate resulted from differences in heat treatment. The nature of stresses at the triple points of sliding grain boundaries was also investigated using tricrystals of aluminum and aluminum-magnesium.

ASD TR 61-474

January 1962

SUBJECT: NOTCH SENSITIVITY OF REFRACTORY METALS
INVESTIGATOR: A. G. Ingram, M. W. Mallett, B. G. Koehl, E. S. Bartlett, H. C. Ogden
CONTRACT: AF33(616)-7604, Battelle Memorial Institute
ABSTRACT: The effects of interstitial oxygen and hydrogen on the tensile and notch tensile properties of tantalum and columbium were investigated. The tensile and notch tensile properties of Ta-10W and F-48 columbium alloy were determined also. Oxygen and hydrogen additions resulted in notch-sensitive behavior at higher temperatures than for pure Ta and Cb, and, similarly, transition temperatures are increased by the interstitial additions. The F-48 alloy also shows notch-sensitive behavior at higher temperatures and higher transition temperatures than columbium. The Ta-10W alloy was not notch sensitive, and retained excellent ductility at -420°F.

In addition, reaction kinetics in the tantalum-hydrogen system were studied.

ASD TDR 62-7

April 1962

SUBJECT: RESEARCH TO DETERMINE THE COMPOSITION OF DISPERSED PHASES IN REFRACTORY METAL ALLOYS
INVESTIGATOR: F. B. Cuff, Jr.
CONTRACT: AF33(616)-7671, Advanced Metals Research Corp.
ABSTRACT: The composition, structures and lattice parameters of the precipitates in two Mo-base and two Nb-base alloys have been determined using the electron microbeam probe and x-ray and electron diffraction techniques.

In general, the precipitates were found to be complex carbides which, in some cases, could be explained on the basis of known phase relationships and thermodynamic data. The deviations of lattice parameters from those of pure carbides could be explained on the basis of atom substitution.

ASD TDR 62-211

April 1962

SUBJECT: A STUDY OF THE INFLUENCE OF HEAT TREATMENT ON MICRO-STRUCTURE AND PROPERTIES OF REFRACTORY ALLOYS

INVESTIGATOR: W. H. Chang

CONTRACT: AF33(616)-7125, General Electric Co.

ABSTRACT: The effect of solutioning and aging treatments on hardness, microstructure, and strength properties of several Mo-base and Cb-base alloys has been investigated. The simultaneous presence of C and Group IV-A elements in Mo-TZC, Cb-F-48, and Cb-F-50 rendered these alloys precipitation-hardenable with TiC, (Zr, Cb) C, and (Zr, Cb, Ti) C as the respective hardening phases.

Alloys Mo-1 Ti-0.1Zr-0.01C and Mo-0.5Ti-0.035C did not exhibit precipitation hardening due either to low C or to restricted carbide solutioning.

The Cb-1Zr alloy containing 350 ppm interstitials exhibited unusual discontinuous-flow behavior. By establishing the various precipitation processes, a basis has been provided in rationalizing the influence of processing on strength properties, in terms of the effects of temperature, time, and plastic deformation on the solutioning and aging reactions.

ASD TDR 62-211, Part II

February 1963

SUBJECT: A STUDY OF THE INFLUENCE OF HEAT TREATMENT ON MICROSTRUCTURE AND PROPERTIES OF REFRACTORY ALLOYS

INVESTIGATOR: W. H. Chang

CONTRACT: AF33(616)-7125, General Electric Co.

ABSTRACT: A study has been made of the inter-effects of chemistry, processing, and heat treatment on the microstructure and properties of five Mo-TC and Mo-TZC type alloys. The solution and ageing reactions have been established and their effect on tensile properties of several worked and non-worked conditions determined between 78 and 3500°F.

The nature and stability range of carbides, ageing kinetics and carbide morphology are shown to depend strongly on the (Ti+Zr)/C ratio, with Zr being much more influential and Ti. Increase in this ratio stabilizes the Ti- or Zr-rich monocarbide, eventually to the exclusion of the Mo₂C phase. The fineness of carbide dispersion is promoted by moderate increase in (Ti+Zr)/C ratio but is adversely affected when the ratio becomes excessive.

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The influence of processing on properties has been clearly demonstrated and its dependency on the solution and ageing reactions discussed. For the alloys with moderate (Ti+Zr)/C ratios, high-temperature extrusion followed by swaging results in high strength and generally low ductility. The ductility can be greatly improved at sacrifice in strength by ageing the extrusions or raising the swaging temperature. In contrast, the tensile properties of the high-Ti(5-6%) alloys are much less attractive and are insensitive to the processing variations.

ASD TDR 62-327, Part I

June 1962

SUBJECT: GAS-METAL REACTIONS IN ROCKET NOZZLES
INVESTIGATOR: J. D. Batchelor, S. W. McCormick, L. Eugene
CONTRACT: AF33(616)-7744, Atlantic Research Corp.
ABSTRACT: Reactions between W, Ta, & a 90%-Ta-10%-W alloy and the combustion products of solid propellants are described. Laboratory studies of hot filament tests where electrically heated wires were exposed to a burning propellant strand were used to determine the important reactions.

Three typical propellants and one with a fluoro-carbon binder (as a source of HF), were used. Experimental results and theoretical thermodynamic predictions were compared. Metallographic examination, micro-hardness measurements, X-ray analysis, and weight changes were used to define the reactions. Oxidation by CO₂ and H₂O was the most serious mode of attack. Other gases were much less damaging. Tungsten was least reactive and tantalum most reactive in each environment; the alloy behaved similar to but was less reactive than tantalum.

ASD TDR 62-506

September 1962

SUBJECT: COMPARISON OF HIGH ENERGY RATE (DYNAPAK) AND CONVENTIONAL EXTRUSION OF REFRACTORY METALS
INVESTIGATOR: D. G. Rabenold
CONTRACT: AF33(616)-7842, Westinghouse Electric Corp.
ABSTRACT: A comparison was made of the surface quality dimensions, chemistry, hardness, tensile properties, and recrystallization behavior of extrusions produced by high-velocity and conventional techniques. Temperatures were established for the development of hot-worked, cold-worked, and duplex metallurgical structures for the Mo-25W-0.1Zr and W-0.6Cb alloys which were then extruded by both methods at a constant 4:1 reduction ratio. The data indicate that equally good

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surface conditions can be obtained from the two processes if proper lubrication and tooling are used; lower hot working temperatures can be used for high-velocity extrusions; and lower recrystallization temperatures are obtained in material cold worked on a high-velocity machine. The latter fact indicates that high-velocity-extruded metals retain a higher degree of internal stress than do conventional-extruded metals.

ASD TDR 62-508

June 1962

SUBJECT: DEVELOPMENT OF WORKABLE MOLYBDENUM- AND TUNGSTEN-BASE ALLOYS

INVESTIGATOR: M. Semchyshen, R. Q. Barr, G. G. Chesmar

CONTRACT: AF33(616)-7930, Climax Molybdenum of Michigan

ABSTRACT: A number of complex Mo-, Mo-W-, and W-base alloys have been consolidated by the vacuum-arc-casting technique. Difficulty was experienced in obtaining sound castings of alloys based on varying Mo/W ratios and containing nominally 1.25% Ti and 0.3% Zr. No difficulties were experienced when the Zr content was reduced to 0.1%. Carbon synergism was studied in the alloys Mo + 3% Ti + 0.5% Zr and Mo + 25% W + 1.25% Ti + 0.1% Zr. In the case of the former alloys, the optimum carbon content from the standpoint of elevated temperature strength appeared to be lower than that predicted on the basis of earlier work with Mo-Ti and Mo-Zr binary alloys. Conversely, the optimum carbon content of the latter alloy agreed well with the extrapolated forecast. In the case of both alloys, the carbon contents yielding the highest elevated temperature strengths also resulted in the highest transition temperatures. An alloy based on Mo-50% W, and containing 0.8% Ti and 0.3% Zr, displayed exceptionally high strengths at elevated temperatures. The tensile transition temperature of this material was, however, above room temperature.

ASD TDR 62-594

October 1962

SUBJECT: INVESTIGATION OF TANTALUM AND ITS ALLOYS

INVESTIGATOR: F. F. Schmidt, et al

CONTRACT: AF33(616)-7688, Battelle Memorial Institute

ABSTRACT: Fabrication results, bend ductility, tensile properties at low and high temperature, high-temperature stress rupture data, welding studies, metallurgical studies (both recrystallization behavior and heat treatment), and oxidation behavior of tantalum and tantalum-base alloys, are presented. Several tantalum-base alloys were found to have outstanding strength properties at temperatures to

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1925°C (3500°F), combined in many cases with excellent room-temperature ductility. Particularly outstanding in this respect are alloys in the system Ta-Mo-W.

ASD TDR 62-670

October 1962

SUBJECT: THE EXTRUSION, FORGING, ROLLING, AND EVALUATION OF REFRACTORY ALLOYS
INVESTIGATOR: D. R. Carnahan, J. A. Visconti
CONTRACT: AF33(616)-8325, Westinghouse Electric Corp.
ABSTRACT: During the present contract year, metallurgical programs were conducted on the extrusion, forging, and rolling of arc cast Mo+ 25%W+0.1%Zr, Mo+0.5%Ti+0.08%Zr+0.02%C and W+0.57%Cb alloys. Each of the three alloys has been worked successfully by extrusion and subsequent reworking. The extrusion process, as established in previous work, has remained primarily the same. However, extrusion temperatures exceeding 4200°F have been reached in the working of a group of tungsten base alloys. The maximum temperature attained to date has been 4320°F. In addition, the extrusion process has been applied to a variety of refractory alloys in order to supply wrought material for other contractors and governmental agencies. The extrusion data are contained as a portion of the work, but the metallurgical evaluation is not included, as it is accomplished by the particular agency for which the work was performed.

ASD TDR 62-756

October 1962

SUBJECT: DEVELOPMENT AND TESTING OF TUNGSTEN EMITTERS FOR ION PROPULSION SYSTEMS
INVESTIGATOR: J. M. Gerken, et al
CONTRACT: AF33(616)-8297, TAPCO, Ramo Wooldridge, Inc.
ABSTRACT: A process is presented for the production of porous tungsten emitter discs with a pore spectrum of 0.7 to 0.9 microns and a mean pore diameter of 0.8 microns. Studies of joining the emitter discs to refractory metal plenum chamber by electron beam welding were conducted. Work was performed on both tungsten and columbium plenum chambers and in both cases, embrittlement was a serious problem. Although difficulties were encountered in successfully welding round emitters into the plenum chamber, sufficient assemblies were prepared for testing. Assemblies successfully withstood 100 hour exposure at 2500°F in a cesium atmosphere. An initial ion efficiency of 98.4% was obtained but due to poisoning, the ion efficiency decreased with increasing time at temperature.

ASD TDR 62-781

October 1962

SUBJECT: VACUUM ARC MELTING OF TUNGSTEN ALLOY (TUNGSTEN-MOLYBDENUM-COLUMBIUM)

INVESTIGATOR: G. A. Reimann

CONTRACT: AF33(616)-7459, Westinghouse Electric Corp.

ABSTRACT: A satisfactory melting technique was established for the production of fine-grained, homogeneous, high quality, tungsten base alloy ingots. The 88W-6Mo-6Cb alloy originally selected for the program was discarded because of ingot cracking and replaced by a 92W-6Mo-2Cb alloy. The reduction of columbium from 6 to 2 percent permitted ingots, 6 to 9 inches in length. Macroexamination revealed a mixture of fine, equiaxed grains and short, thin columnar grains. Seven ingots of the 92-6-2 composition were extruded successfully at 4000°F, using ratios of 4.4:1, 5.7:1, and 7.5:1. The extruded material was forged and rolled to determine workability. A time, temperature, recrystallization study was conducted on forged specimens of the 92-6-2 alloy.

ASD TDR 62-858

November 1962

SUBJECT: A COMPUTER PROGRAM FOR THE MATANO ANALYSIS OF BINARY DIFFUSION DATA

INVESTIGATOR: C. S. Hartley, K. Hubbard

ABSTRACT: A computer program for the Boltzmann-Matano solution to Fick's second law in semi-infinite binary diffusion couples is presented. The program is written in Fortran for the IBM 7090 digital computer. An error function curve fit is used to interpolate between and smooth experimental data, and the calculations performed on the regenerated data.

Examples are given of application of the program to incremental couples with no intermediate phases and to pure metal couples with and without intermediate phases. Phase boundaries in couples with intermediate phases can also be obtained with a high degree of accuracy. By substituting lattice spacings for ordinary distance units, a correction for molal volume change due to composition differences can be programmed.

ASD TDR 62-859

November 1962

SUBJECT: DISLOCATION PINNING EFFECTS IN UNALLOYED MOLYBDENUM

INVESTIGATOR: C. S. Hartley, R. J. Wilson

ABSTRACT: The rate of yield point return and modulus defect

ASD TDR 62-859 (Continued)

recovery is studied in powder metallurgical and arc-cast molybdenum, respectively. The activation energy for yield point return is found to be $25,100 \pm 2700$ cal/mol, and is believed to be due to pinning of deformation produced free dislocations by interstitial oxygen or nitrogen atoms. A calculation of the number of impurity atoms per unit length of dislocation line necessary to cause yield point return suggests that this pinning is effected by the formation of micro-precipitates. Possible mechanisms consistent with this idea which exhibit kinetics similar to the Cottrell-Bilby "atmosphere" theory are discussed.

Recovery of the modulus defect is shown to be controlled by the diffusion of the slowest pinning defect. The activation energy for diffusion of the slowest defect calculated from a treatment based on the Granato-Hikata-Lucke theory of point defect pinning is $44,300 \pm 3000$ cal/mol. It is suggested that this defect is carbon.

ASD TDR 63-233

May 1963

SUBJECT: RESEARCH AND EVALUATION OF MATERIALS FOR THERMO-
COUPLE APPLICATION SUITABLE FOR TEMPERATURE
MEASUREMENTS UP TO 4500°F ON THE SURFACE GLIDE
RE-ENTRY VEHICLES

INVESTIGATOR: W. C. Kuhlman

CONTRACT: AF33(657)-8472, General Electric Co.

ABSTRACT: The thermoelectric output versus temperature of
a W/W - 26Re thermocouple was measured to 5800°F. Thermoelectric
sensitivity was relatively high over the entire temperature range.

Thermoelectric output on new thermocouple systems versus temperature was also measured with respect to W - 26Re commercial wire for Re, Re - 6W, Re - 9W, Re - 12W, Re - 15W, Re - 10Ru, Re - 5Ru, Re - 10Rh, Re - 20Rh, Re - 10Pt, Re - 30Ir, Re - 5Mo, Re - 10 Os, Re - 20 Os, Mo, Mo - 10W, Mo - 20W, Mo - 10Re, Ir - 20Re alloy materials. Of the alloys studied, Re - 10Ru and Re - 12W show the greatest potential for high-temperature thermocouple application.

The oxides, BeO, ThO₂, and HfO₂, are compared as electrical insulators. Beryllia is best, but with proper geometry ThO₂ and HfO₂ would also be satisfactory. Boron nitride was found to react with W and W - 26Re materials above 3700°F in helium. The melting points of many rare earth oxides, zirconates, and hafnates.

ASD TDR 63-233 (Continued)

Weight loss of Ir, Ir - 10Re, Os, Ir - 15 Os, and Ir - 20 Os at 0.1 atmosphere pressure at approximately 4400°F was measured. The test shows that Ir - 20 Os alloy, with a measured melting point of 4600°F should make satisfactory sheathing material for short time service.

ASD TDR 63-296

April 1963

SUBJECT: VACUUM ARC MELTING OF TUNGSTEN + 0.6 COLUMBIUM
INVESTIGATOR: G. A. Reimann
CONTRACT: AF33(616)-7459, Westinghouse Electric Corp.
ABSTRACT: Vacuum arc melting techniques were developed to produce sound 3 1/2 inch and 4 inch diameter ingots of tungsten base alloys. The effect of melting rate on ingot characteristics was determined and the effects of carbon, oxygen, zirconium, and titanium on melting characteristics, ingot structure, extrudability, recrystallization, and high temperature tensile properties of the W+0.6Cb alloy were studied also.

Refinement of ingot structure was produced by 0.06 and 0.12% zirconium additions and by increasing the melting rate. Coarsening of ingot structure was produced by 0.04% titanium and 500 to 1000 ppm oxygen additions. W+0.6Cb ingots containing more than 50 ppm carbon could not be extruded, and correlation between extrudability and carbon content below 50 ppm was not found.

Adding 0.12% zirconium to the W+0.6Cb alloy significantly improved the 3000°F tensile properties.

ASD TDR 63-324

April 1963

SUBJECT: SYMPOSIUM ON THE ROLE OF SUBSTRUCTURE IN THE MECHANICAL BEHAVIOR OF METALS
ABSTRACT: The papers contained in this technical documentary report were presented at a symposium which dealt with the subject of substructure and its effect on the mechanical properties of metals. The symposium was held in Orlando, Florida on 5-7 December 1962. Both review papers and reports of current research are included. The following general subject areas are covered: experimental techniques for observing and measuring substructure; development of substructure by deformation, recovery, and recrystallization; the role of substructure in yielding, twinning, and fracture; and the role of substructure in transformation and precipitation.

ASD TDR 63-360

SUBJECT: INVESTIGATIONS OF THE RELATIONSHIP OF TWINNING
TO BRITTLE STRUCTURE OF REFRACTORY METALS
INVESTIGATOR: C. N. Reid, A. Gilbert, G. T. Hahn
CONTRACT: AF33(657)-7376, Battelle Memorial Institute
ABSTRACT: The first year's work on this program is reported. It is concluded that there is good evidence for brittle fracture induced by mechanical twinning in single crystals and polycrystalline Mo-C with grain sizes larger than 0.40 mm. However, there is no evidence for such failures in Mo-C of grain size smaller than 0.25 mm. or in high-purity Cb. The effect of recrystallization texture on twinning is demonstrated, and the orientation dependence of twinning in Cb crystals is reported; a most unusual plastic instability is described in crystals of one orientation. It is shown that twinning in Mo-35Re occurs in bursts, which form in less than 2 μ sec; nucleation of twins occurs only at small strains but lateral growth takes place up to at least 12 per cent strain.

ASD TDR 63-420

April 1963

SUBJECT: FLOW AND FRACTURE OF TUNGSTEN AND ITS ALLOYS:
WROUGHT, RECRYSTALLIZED AND WELDED CONDITIONS
INVESTIGATOR: J. W. Clark
CONTRACT: AF33(616)-7484, General Electric Co.
ABSTRACT: Studies of the effects of composition, heat treatment, and microstructure on the bend ductility of vacuum-melted W-27Re and dilute W-Ru, W-Hf, W-Hf-C, and W-B alloys are presented. It is shown that W-27Re exhibits ductility at -100°F in fine-grained structures. The flow and fracture of alloys containing C or B is particularly responsive to heat treatment. At equivalent grain sizes, the ductile-brittle transition temperature has been decreased by as much as 300°F by treatments which promote matrix or grain-boundary precipitation. Effects of annealing commercial powder metallurgy W in an organic-free, sputter-ion vacuum system (5×10^{-8} torr) are explored. The carbon content is markedly reduced and the transition temperature of subsequently worked and recrystallized strip shows a significant decrease.

SOLID PHASE & INTERPHASE PHENOMENA IN CRYSTALLINE SUBSTANCES

WADD TR 61-58, Part II

April 1961

SUBJECT: THE EFFECT OF SURFACE-ACTIVE AGENTS ON THE MECHANICAL PROPERTIES OF METALS. PART II. The Effect of Surface-Active Agents on the Mechanical Behavior of Aluminum Single Crystals

INVESTIGATOR: I. R. Kramer

CONTRACT: AF33(616)-6220, The Martin Co.

ABSTRACT: Single crystals of aluminum were pulled in tension in a solution of paraffin oil and stearic acid. The critical resolved shear stress did not change with the concentration of the stearic acid solution; however, the extent and slopes of Stages I and II were affected greatly. The observations lend evidence that the weakening effect of surface-active agents is controlled by the rate of desorption of the metal soap formed by the reaction of the surface-active agent and the metal surface.

WADD TR 61-145, Part II

April 1962

SUBJECT: DISLOCATIONS AND PLASTIC BEHAVIOR OF IRON SINGLE CRYSTALS

INVESTIGATOR: K. E. Kimball

CONTRACT: AF33(616)-6348, Manufacturing Laboratories, Inc.

ABSTRACT: The purpose of this investigation was to determine the behavior of imperfections during plastic deformation of high purity highly perfect single crystals of iron. To carry out this investigation, it was necessary to develop the equipment and techniques necessary for growing large single crystals and to obtain accurate, quantitative measurements of the behavior of imperfections during plastic straining.

There were four main accomplishments of the present program; vis., the growth of highly perfect iron single crystals, the measurement of crystal perfection as a function of plastic strain by rocking curve measurements, the measurement of the stress-strain relationship for highly perfect iron single crystals, and the comparison of the stress-strain relationship with the etch pit and rocking curve measurements. Possible dislocation interpretations of the observed data are presented.

ASD TDR 62-99

August 1962

SUBJECT: EFFECTS OF SURFACE ENVIRONMENTS ON THE MECHANICAL BEHAVIOR OF METALS
INVESTIGATOR: I. R. Kramer
CONTRACT: AF33(616)-7976, The Martin Co.
ABSTRACT: A study of the mechanical behavior of copper single crystals in solutions containing surface-active agents shows that the weakening effect is associated with the formation and solution of metallic soaps. A large change in the stress-strain characteristics of polycrystalline aluminum alloys was found when the surface of the specimens were removed during plastic deformation. Studies of the effect of size of the specimen on the changes of slopes of Stages I and II by surface removal showed that the change of the slope of Stage I was independent of size with respect to the polishing rate; however, the change in the slope of Stage II with polishing rate increased directly in proportion to surface area or cross section. A surface removal treatment on single crystals eliminated the yield point, whereas the same type of treatment enhanced the yield point drop on aluminum alloys. Measurements of slipband spacing indicate that surface treatment affects the egress of dislocations.

ASD TDR 62-329

May 1962

SUBJECT: A STUDY OF QUENCH HARDENING IN PLATINUM AND GOLD
INVESTIGATOR: H. L. Gegel
ABSTRACT: The results of this program have shown that vacancy complexes form during the quenching period when the average quenching speed is less than 10^{50}C/second . The binding energy for divacancies in gold was estimated to be approximately 0.28 ev. The influence of increased quenching speeds is to increase the temperature recovery range for isochronal recovery. Similarly, fast quenching rates caused an incubation period in the isothermal aging experiments for gold. Two distinct hardening mechanisms exist for platinum and gold when the values of the activation energy for recovery are taken into account. The hardening mechanism for gold is thought to be due to the interaction of dislocations with extended sessile dislocations, and the hardening mechanism for platinum is thought to be due to the interaction of dislocations with dislocation loops. This model has been extended to other F.C.C. metals.

ASD TDR 62-331

June 1962

SUBJECT: X-RAY DIFFRACTION INVESTIGATIONS ON IRON WHISKERS
INVESTIGATOR: W. R. Rambauske, R. R. Gruenzel
CONTRACT: AF33(616)-6607, University of Dayton

ASD TDR 62-331 (Continued)

ABSTRACT: Iron whiskers have been investigated by x-ray diffraction. The Laue method is employed to determine the orientation of the lattice planes. For a series of hexagonal type whiskers it was found that the lateral face, which according to earlier reports belonging to a (110) plane orientation, belongs to a (211) plane orientation. In another series of whiskers which were grown in one boat condition, a twist of the lattice indices of direction over the whisker length was found. This twist is not visible optically. Necessary exposure times and degree of information obtainable from Laue-diffraction are determined. Higher resolution over the whisker length is necessary to find variations in the parameters, which determine the microstructure of the whiskers. To achieve this resolution, experimental ways are proposed and in progress. Digital computation for analysis of data is reported.

ASD TDR 62-356

June 1962

SUBJECT: RESEARCHES ON HYDROGEN OVERVOLTAGE ON METALLIC SINGLE CRYSTALS: GOLD
INVESTIGATOR: L. P. Bicelli, M. Graziano
CONTRACT: AF61(052)-1144, Chimica Fisica Metallurgia del Politecnico di Milano, Milano, Italy
ABSTRACT: Hydrogen overvoltage on gold polycrystalline and single crystal-cathodes, oriented following the (100), (110), and (111) planes, has been investigated in 0, 1 M perchloric acid solutions, in a c.d. range up to 200 A/m².

The Tafel law holds true, but two different slopes are observed, that in the lower c.d. range having the smallest value.

The parameters are different for the different electrodes.

ASD TDR 62-357

May 1962

SUBJECT: RESEARCHES ON HYDROGEN OVER-VOLTAGE ON METALLIC SINGLE CRYSTALS
INVESTIGATOR: R. Piontelli, L. P. Bicelli, B. Rivolta, A. LaVecchia, M. Graziano, A. Pappagallo
CONTRACT: AF61(052)-1144, Chimica Fisica Metallurgia del Politecnico di Milano, Milano, Italy
ABSTRACT: The purpose of the research work sponsored by

ASD TDR 62-357 (Continued)

ARDC, USAF, Contract No. AF61(052)-144, and here summarized was the determination of the hydrogen-overvoltage for metallic single crystal electrodes (SCE), whose working surface was oriented following different lattice planes and for polycrystalline ones, in various conditions as far as current density (c.d.), temperature and other conditions are concerned.

ASD TDR 62-388

May 1962

SUBJECT: CALCULATION OF STACKING FAULT ENERGIES FOR FACE-CENTERED CUBIC METALS

INVESTIGATOR: H. L. Gegel

ABSTRACT: A theoretical investigation was undertaken to determine the stacking fault energy for face-centered cubic metals. An expression was derived which relates the Debye characteristic temperature of the metal and the absolute temperature to the stacking fault energy. An Einstein oscillator was used as a model for the derivation.

ASD TDR 62-593

August 1962

SUBJECT: IONIC THINNING OF SPECIMENS FOR ELECTRON MICROSCOPY

INVESTIGATOR: R. Castaing

CONTRACT: AF61(052)-338, University of Paris

ABSTRACT: The effect of very short ion bombardments on thin evaporated crystals of gold has been investigated by means of the electron microscope. The main effect is the production of a large number of very small loops and the disappearance of the pre-existing dislocations. Annealing eliminates the small loops and dislocations appear again. This phenomenon is responsible for the roughness observed on samples of heavy metals thinned down by ionic polishing. The production of the loops is interpreted in terms of vacancy and divacancy movements inside the bombarded layer. Heating the specimen during the bombardment seems to be the only way for eliminating the production of the defects.

ASD TDR 62-1013

March 1963

SUBJECT: INVESTIGATION OF THE GENERAL STRESS DISTRIBUTION IN IMPELLER WHEELS

INVESTIGATOR: W. Dettmering, W. Soetebeer

ASD TDR 62-1013 (Continued)

CONTRACT: AF61(052)-520, Technische Hochschule, Aachen, Germany
ABSTRACT: This report contains a detailed illustration of new calculation data for impeller wheels. They are designed to furnish comparative figures for experimental investigations with systematically varied impeller wheels. Furthermore, initial experimental results are presented on the distribution of stresses in a model of an impeller wheel.

ASD TDR 62-1040

March 1963

SUBJECT: PREPARATION OF HIGH PURITY ANHYDROUS FERROUS CHLORIDE
INVESTIGATOR: H. W. Chandler, J. Malgiolio
CONTRACT: AF33(616)-8187, Isomet Corp.
ABSTRACT: Several methods for preparing high purity anhydrous ferrous chloride were investigated. The technique finally developed involved the high temperature chlorination of iron sponge with anhydrous hydrogen chloride and collection of the sublimed ferrous chloride in a condenser held at such a temperature that ferric chloride did not condense. Ferric iron was undetectable in the final product and other impurities were well below the specified limits. Over 3 kilograms of high purity anhydrous ferrous chloride were prepared by this technique.

ASD TDR 62-1067

March 1963

SUBJECT: STUDIES OF HYDROGEN AND OXYGEN EVOLUTION AND THE INFLUENCE OF ADSORBED SUBSTANCES
INVESTIGATOR: L. Kandler, et al
CONTRACT: AF61(052)-305, Technische Hochschule, Munich, Germany
ABSTRACT: In this report, A.C. measurements of the interface impedance prove to be suitable for studying adsorption phenomena on porous electrodes like platinated platinum and sintered nickel material. The impedance on platinum electrodes was evaluated for 8N H_2SO_4 and 1N KOH solutions. Further investigations dealt with inhibition problems on highly activated porous nickel electrodes. Several alcohols were used as additives to 1N KOH.

The applicability of the potentiostatic method of applying triangular voltages was examined on bright nickel electrodes. The electrolytes 8N H_2SO_4 in 1N KOH in general were stirred with purified nitrogen. The applicability of the potentiostatic method of

ASD TDR 62-1067 (Continued)

triangular voltages to show the influence of inhibitors by applying the principle of Wagner-Traut was examined.

ASD TDR 63-361

May 1963

SUBJECT: INVESTIGATION OF THE DISTRIBUTION OF TENSION IN
NOTCHED CONSTRUCTION ELEMENTS

INVESTIGATOR: H. Neuber

CONTRACT: AF61(052)-200, Technische Hochschule, Munchen,
Germany

ABSTRACT: The theoretical part of the research work concerns a new treatment of stress- and strain concentrations for a special non-linear deformation law for shear. A relation is obtained by which the real notch stress can be evaluated from the nominal stress and the elastic stress concentration factor. The notch-angle is exactly taken into account.

The experimental part contains static and fatigue tests with variously notched specimens. The strain-measurements were performed by electrical strain gages; additional measurements were made by means of the photoelastic stress-coat method. The aim of the static tests was the investigation of the notch-angle influence on the elastic stress concentration factor for tension and bending, the determination of the stress- and strain distribution in the elastic and plastic range, respectively, and also the dependence of the stress concentration factor on the nominal stress for the materials used (Steel St.00.12, St.00.21, Aluminum alloy "Velodur"). The result of the fatigue tests was the establishment of Wohler-curves for two different specimen shapes (bars with central holes) and their comparison with the Wohler-curve of the corresponding unnotched specimen.

SPRINGS

ASD TR 61-566

May 1962

SUBJECT: WIDE-RANGE TEMPERATURE SPRINGS
INVESTIGATOR: H. Gascoigne, et al
CONTRACT: AF33(616)-6284, University of Michigan
ABSTRACT: Bimetallic spring systems of 40% nickel-60% iron alloy combined with Inconel X and Type 304 stainless steel exhibit characteristics suitable for applications from -65° to 600°F. In this range, the spring constant is held within $\pm 0.25\%$.

The general theory of bimetallic, temperature independent helical coil and Belleville type springs is presented. The bimetallic systems tested show that a temperature independent spring is feasible although working stresses must be maintained at relatively low levels.

STATISTICS

ASD TDR 62-379

April 1962

SUBJECT: STATISTICAL RESPONSE OF A BAR IN TENSION
INVESTIGATOR: D. A. Smith, T. I. Smits, R. F. Lambert
CONTRACT: AF33(657)-7453, University of Minnesota
ABSTRACT: Theoretical and experimental statistical analysis of the random response of a continuous bar in tension is presented. Particular attention has been paid to the probability distribution of the strain response which, for a linear second-order system under gaussian excitation, follows a Rayleigh distribution. However, when the excitation level of the clamped-clamped continuous bar is sufficiently high so that the tensile strain becomes comparable with the bending strain, then the strain crest distribution no longer follows the Rayleigh prediction. At high strain levels, the distribution of positive crests as well as maxima is greater than the Rayleigh prediction and the distribution of negative crests as well as minima is less. The distribution of positive maxima falls below the positive crest distribution as the Q of the system decreases. Similarly, the distribution of negative minima falls below the negative crest distribution as the Q decreases.

ASD TDR 63-63

January 1963

SUBJECT: OUTLINE OF AN ALGEBRA OF STOCHASTIC QUANTITIES
INVESTIGATOR: W. Weibull
CONTRACT: AF61(052)-522, European Office, Office of Aerospace Research
ABSTRACT: A method is developed for solving random equations (equations involving variates). A set of elements, called stochastic quantities, was constructed to constitute a field, which implies that there exists for every element of it an inverse element relative to the additive and multiplicative laws of composition. This makes it possible to compute with stochastic quantities as usually is done with rational numbers in the four fundamental operations $+$, $-$, $.$, $:/$. Problems leading to random equations are presented and general properties of variate and multiplex stochastic quantities are indicated. It is demonstrated how simplified methods for composition and inversion of variates can be developed. Finally, classification and some solutions of random equations are given.

STRESS ANALYSIS

WADD TR 60-869, Part II

June 1962

SUBJECT: EFFECT OF STATE OF STRESS ON THE FAILURE OF METALS AT VARIOUS TEMPERATURES
INVESTIGATOR: R. M. Haythornthwaite, D. R. Jenkins
CONTRACT: AF33(616)-6041, University of Michigan
ABSTRACT: Experimental observations of initial yielding, strain hardening, and fracture of Zamak-3 tubes for various states of combined stress are presented. Testing temperatures of 32°F and 78°F were employed. These observations are compared with predictions of Mises, Tresca, and maximum reduced stress theories of initial yielding and with isotropic and kinematic theories of strain hardening. Fracture data are compared with the Griffith theory of rupture for brittle materials. It is concluded that Zamak-3 behaves as an essentially isotropic material in which yielding is independent of mean stress. Multiple loading path test results agree rather well with the predictions of kinematic hardening theory in conjunction with the Tresca yield criterion. Fracture results conform to a maximum normal stress theory which coincides with the Griffith theory for the stress combinations investigated.

WADD TR 61-42, Part II

January 1963

SUBJECT: STUDY OF ULTRASONIC METHODS FOR NONDESTRUCTIVE MEASUREMENT OF RESIDUAL STRESS
INVESTIGATOR: F. R. Rollins, Jr., D. R. Kobett, J. L. Jones
CONTRACT: AF33(616)-7058, Midwest Research Institute
ABSTRACT: The study of stress-induced birefringence has been continued in both polycrystalline and single crystal experiments. The effect is explained on the basis of nonlinear elasticity theory. Experiments indicate that dislocation activity does not strongly affect results in polycrystalline specimens, however, a pronounced influence may be observed in single crystals.

Nonlinear elasticity theory has been used to investigate the interaction of two intersecting, plane, elastic waves in a homogeneous, isotropic medium. A criterion for the occurrence of a strong scattered wave has been derived. The criterion is formulated as a relationship between the second order elastic constants of the material, the angle between the intersecting wave vectors, and the ratio of primary wave frequencies. The amplitude of the scattered wave is found to be proportional to the volume of interaction and dependent on the third order elastic constants of the material. Preliminary efforts to experimentally verify the theoretical predictions are described.

WADC TR 53-373 Sup 10

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WADD TR 61-42, Part III

May 1963

SUBJECT: STUDY OF ULTRASONIC TECHNIQUES FOR THE NON-DESTRUCTIVE MEASUREMENT OF RESIDUAL STRESS
INVESTIGATOR: F. R. Rollins, P. Waldow
CONTRACT: AF33(616)-7058, Midwest Research Institute
ABSTRACT: The theoretical and experimental investigation of ultrasonic beam interaction in solid materials has been continued. Interaction of pulsed beams (3-15 mc/s) under "resonant" conditions reveals that interaction does occur in many materials. A theoretically predicted third beam is generated at the "point" of intersection and has been experimentally observed in samples of fused silica, polycrystalline aluminum, and polycrystalline magnesium. A potential method of three-dimensional stress analysis is discussed. An optical system for studying beam interaction in transparent solids is also described.

WADD TR 61-91, Part II

February 1963

SUBJECT: ULTRASONIC METHODS FOR NONDESTRUCTIVE EVALUATION OF CERAMIC COATINGS
INVESTIGATOR: K. E. Feith, W. E. Lawrie
CONTRACT: AF33(616)-6396, Armour Research Foundation
ABSTRACT: This report describes investigations into the use of ultrasonic techniques to determine the strength and integrity of ceramic-metal bonds. An acoustic image converter system was used successfully to obtain a television type display of 1/32 in. diameter laminar defects in a zirconium oxide-inconel bond. The measured defect thickness varied between 300 and 500 micro-inches or about a factor of three greater than the average large grain in the coating material. Schlieren optical and acoustic lens techniques were used to visually investigate detailed properties of ultrasonic fields and the interaction of ultrasonic energy with a solid. The theory of Rayleigh waves was reviewed to provide a framework for experimental integrity determinations of ceramic coatings.

WADD TR 61-91, Part III

March 1963

SUBJECT: ULTRASONIC METHODS FOR NONDESTRUCTIVE EVALUATION OF CERAMIC COATINGS
INVESTIGATOR: A. M. Baumanis, W. E. Lawrie
CONTRACT: AF33(657), 8938, Armour Research Foundation
ABSTRACT: This report describes investigations into techniques to determine the strength and integrity of ceramic-metal bonds. Primary emphasis was placed on techniques for examining diffusion coatings although work continued on flame-sprayed coatings. Extensive

WADD TR 61-91, Part III (Continued)

investigations of Rayleigh wave techniques have been made with promising results. Further work was done using the Schlieren optical method, and thermal effects were investigated in relation to a Schlieren result. Investigations of an interferometric method for measurements of elastic constants have shown the method to have low accuracy. Neither destructive nor nondestructive methods have been found to successfully measure bond strength of diffusion coatings. Preliminary investigations have been made of thermal, electrical, and nuclear methods for examining flame-sprayed coatings.

ASD TR 61-436, Part I

February 1963

SUBJECT: NONDESTRUCTIVE ANALYSIS OF THE BRITTLE FRACTURE BEHAVIOR OF CERAMIC MATERIALS
INVESTIGATOR: J. H. Lauchner, T. F. Torries, J. L. Pentecost
CONTRACT: AF33(616)-7347, Mississippi State University
ABSTRACT: A literature survey of theoretical and experimental approaches to brittle fracture was directed toward a nondestructive evaluation point of view. Surface decorating techniques were investigated.

A technique for calculating maximum stress in an elastic loop was developed and applied to the measurement of the strength of glass fibers from three to seven mils in diameter. The strength values were related to surface condition by surface decoration techniques. *Surface flaw decoration of cleaned and polished surfaces was performed by condensation of water on the surface. Minute flaws estimated to be less than 1000Å deep were easily revealed.

Capacitance behaviors of polycrystalline materials were studied. Residual stress effects were observed under statically loaded conditions.

ASD TR 61-725

May 1962

SUBJECT: THE EFFECT OF STRESS GRADIENT AND STRESS BI-AXIALITY ON THE BEHAVIOR OF MATERIALS
INVESTIGATOR: V. Weiss, J. Sessler, K. Grewal
CONTRACT: AF33(616)-7609, Syracuse University Research Inst.
ABSTRACT: The effects of stress gradient and stress bi-axiality on the notch strength of brittle, semi-brittle and semi-ductile materials were investigated. The experimental results obtained on a nearly ideally brittle titanium alloy, are in agreement with the

predictions of a fracture concept based on Neuber's theory of notch stresses, which was developed at Syracuse University. Accordingly, the stress concentration factor is the dominant parameter which affects the notch strength. The magnitude of the stress gradient and stress biaxiality at the notch root has a secondary effect in that the notch strength of brittle materials is reduced with decreasing gradient and increasing biaxiality. Notch strength behavior of brittle ceramics has to be interpreted in terms of inhomogeneity of these materials. Weibull's statistical theory of fracture was applied to this problem. Semi-brittle and semi-ductile materials exhibit a behavior similar in trend to that predicted for brittle materials with respect to stress concentration factor, stress gradient and stress biaxiality. However, for low K_t values, these effects are altered by phenomena due to plastic deformation at and near the notch root which significantly modify the stress and strain distribution and cause notch strengthening, the amount of which, in itself, appears related to the stress gradient and the stress biaxiality.

ASD TDR 62-323

February 1963

SUBJECT: ANELASTIC BEHAVIOR OF TANTALUM AND COLUMBIUM
 INVESTIGATOR: R. J. Sneed, E. L. Fink, M. C. Abrams
 CONTRACT: AF33(616)-7235, General Dynamics
 ABSTRACT: A theoretical model is developed based on dislocation-interstitial interaction during stress application, which describes the yield delay behavior in bcc metals. Reorientation of interstitial impurities apparently controls both the pre-yield micro-strain rate and the time to yield. The model is supported for Ta and Cb by the results of yield delay experiments over a temperature range from -97°F to 400°F . Activation energies of the yield delay process indicate that hydrogen has a controlling effect, with other interstitials contributing.

The model is further substantiated by x-ray diffractometry which demonstrates the occurrence of anelastic lattice strains during load application and their consequent recovery.

A brief resume' is given on the initial efforts of a high temperature study on the anelasticity in tantalum and columbium.

ASD TDR 62-385

January 1963

SUBJECT: APPLIED RESEARCH TO ESTABLISH INFRARED DETECTION
METHODS FOR NONDESTRUCTIVE ANALYSIS OF METALLIC
AND CERAMIC STRUCTURES

INVESTIGATOR: D. R. Maley

CONTRACT: AF33(616)-7725, Automation Industries

ABSTRACT: This program was a study to determine the feasibility of thermal nondestructive evaluation of materials. A thermal testing system was conceived and developed with which certain fabricated material inconsistencies have been detected in std test samples. The system operation is basically a programmed heating and subsequent temperature measurement over one surface of the test sample. Heat flow from the heated surface into the material is examined through the surface temperature measurement. Temperature is measured with an infrared detection system. A display of the temperature pattern over the surface is then interpreted with respect to internal material inconsistencies. A section of the report entitled "Test Results" summarizes the tests, showing the detection of inconsistencies in and std material samples. Inconsistencies detected to date include voids, delamination (areas of unbonding), and metallic inclusion.

ASD TDR 62-829

November 1962

SUBJECT: AGING STUDY OF 2024 ALUMINUM BY DYNAMIC MODULUS
TECHNIQUES

INVESTIGATOR: G. W. King

ABSTRACT: Dynamic modulus measurements are used to study precipitation rates in 2024 aluminum alloy. The results are discussed in terms of the effect of structural changes on the elastic modulus as well as the kinetics of precipitation at various stages of the age-hardening process. Activation energies were determined for aging in the temperature ranges from room (77°F) to 130°F, and 375°F to 425°F. A value of 20.7K Cal/gm mole was obtained for the lower temperature range, and 23K Cal/gm mole for the higher temperatures. The data indicate that modulus changes are primarily influenced by the structure of the second phase, however, some evidence of strain energy effects are indicated.

UNIQUE METALLIC MATERIALS AND TECHNIQUES

WADD TR 60-893, Part II

August 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON HIGH-PRESSURE-HIGH-TEMPERATURE METALLURGY

INVESTIGATOR: S. A. Kulin, et al

CONTRACT: AF33(616)-6837, ManLabs, Inc.

ABSTRACT: The kinetics of recrystallization of polycrystalline copper (99.999% purity) cold-rolled to 98% reduction have been determined by means of an x-ray diffraction technique for the temperature range 80° - 170°C at atmospheric pressure and at 42 kilobars. High pressure is found to retard both the initiation and rate of recrystallization. The effect of high pressure on the Hall voltage of cerium has been determined. A study of the pressure-temperature characteristics of the transformation of the hexagonal phase, $\text{MoCo}_{0.67}$ to the cubic phase, $\text{MoCo}_{0.67+x}$ has been completed. The role of carbon deficiency in the transformation was investigated. The results of a series of experiments designed to investigate pressure-quenching in several different iron-nickel alloys are reported. Thermodynamic data obtained at one atmosphere is used to correlate the high-pressure transitions in thallium and tin. Several iron-carbon alloys and plain carbon steels ranging from 0.08 to 1.23 wt percent carbon content have been subjected to various heat treatments at a pressure of 42 kilobars. A series of experiments in which pressure is used to enhance the mechanical properties of selected steels is described together with the apparatus employed.

WADD TR 60-893, Part III

May 1963

SUBJECT: RESEARCH AND DEVELOPMENT ON HIGH-PRESSURE-HIGH-TEMPERATURE METALLURGY

INVESTIGATOR: S. A. Kulin, et al

CONTRACT: AF33(616)-6837, ManLabs, Inc.

ABSTRACT: Two separate high-pressure systems have been designed, constructed and tested. Their successful use to pressures of 60 kilobars and temperatures up to 1500°C has been demonstrated. It is anticipated that both of these systems will be utilized for high-pressure heat treatment of large specimens in the current high pressure program.

ASD TDR 62-181

June 1962

SUBJECT: INVESTIGATION OF THE EFFECT OF ULTRA-RAPID
QUENCHING ON METALLIC SYSTEMS, INCLUDING BERYLLIUM
ALLOYS

INVESTIGATOR: C. Jordan

CONTRACT: AF33(616)-8011, Electro-Optical Systems, Inc.

ABSTRACT: The effect of ultra-rapid quenching on eight binary
alloy systems, namely, Be-H, Be-O, Be-Al, Be-Si, Be-Sc, Be-Ni, Be-Cu,
and Be-Zn, has been investigated. In four of the systems, namely,
Be-H, Be-O, Be-Sc, and Be-Zn, attempts to carry out the necessary ex-
periments were unsuccessful because of difficulties as to material or
technique which could not be overcome within the scope of the contract.
In the other four systems, the best experimental results obtainable
indicate that no significant change of structure is produced by rapid
quenching.

ASD TDR 62-354

September 1962

SUBJECT: RESEARCH FOR A FUNDAMENTAL STUDY ON THE WORK
HARDENING OF BODY-CENTERED CUBIC METALS

INVESTIGATOR: D. P. Gregory, G. H. Rowe, A. N. Stroh

CONTRACT: AF33(616)-7855, Pratt & Whitney Aircraft Corp.

ABSTRACT: The first portion of this report contains a
literature survey and a critical analysis of the development of work
hardening theory. The second portion, experimental, describes work
hardening mechanisms in columbium deduced from results of tensile
studies at various strain rates and temperatures, strain rate and
temperature cycling tests, and transmission electron microscopy.
Work hardening in the fine grain Cb polycrystals at 500 appears to re-
sult from a decrease in both the number of mobile dislocations and
the activation volume with increasing strain. The particular mechanism
responsible for work hardening appears to be the movement of jogs in
screw dislocations which results in the formation of lattice vacancies.
Polycrystals were found to yield by a Luders mechanism while the single
crystals yield homogeneously. The number of mobile dislocations in
the polycrystals decreases with increasing strain while the number of
mobile dislocations in the single crystals increases continuously with
strain.

ASD TDR 62-442

August 1962

SUBJECT: EFFECT OF SHOCK-INDUCED HIGH DYNAMIC PRESSURES
ON IRON-BASE ALLOYS

INVESTIGATOR: S. M. Silverman, L. Godfrey, et al

CONTRACT: AF33(616)-8190, Pratt & Whitney Aircraft Division

ASD TDR 62-442 (Continued)

ABSTRACT: The object of this program was to investigate the effect of high dynamic pressures generated by strong shock waves on the metallurgical properties of selected iron based alloys. This investigation included a study of the effects of shock wave duration, repeated shocks on a single test specimen, increasing shock wave intensity, and post shock treatment on the yield and tensile strengths of H-11 tool steel and 25 percent nickel steel. As a result of explosive shock hardening, H-11 steel increased in yield strength from 235 ksi in the pre-shocked condition to 340 psi in the as-shocked (360 kilobars) condition, while the 25 percent nickel steel showed yield strength increases from 235 ksi in the austenitized-plus-aged condition to 255 psi in the shocked-plus-aged condition. These increases in yield strength were brought about without any significant macroscopic plastic deformation.

ASD TDR 62-535

February 1963

SUBJECT: INVESTIGATE THE EFFECT OF HIGH DYNAMIC PRESSURES
UPON THE METALLURGICAL PROPERTIES OF IRON AND
TITANIUM BASE ALLOYS

INVESTIGATOR: A. W. Hall, et al

CONTRACT: AF33(616)-8191, Aerojet-General Corp.

ABSTRACT: Effects of high dynamic pressures upon the metallurgical properties of iron and titanium base alloys are given. Pressure levels ranging from 86 kilobars to 390 kilobars were used to demonstrate refinement of structure with indications of ausforming in H-11 steel shocked at elevated temperatures, a reduction in retained austenite for heat-treated and tempered UHB-46 steel, and an increase in susceptibility to aging for Bl20VCA titanium. Metallurgical examinations of shocked specimens were conducted optically with X-ray diffraction and with electron microscopy. Changes in hardness, structure and dimensions were recorded and correlated with the pressures used. Hypotheses to explain the observed effects are presented. Details are presented pertinent to the explosive techniques developed for generating the dynamic shock, the recovery system employed to recover the specimens after shocking, and the problem areas associated with testing at elevated temperatures. Recommendations for future work in specific areas selected as a result of the survey testing conducted under this program are presented.

ASD TDR 62-667

October 1962

SUBJECT: DEVELOPMENT OF IMPROVED VANADIUM-BASE ALLOYS FOR ELEVATED-TEMPERATURE USE

INVESTIGATOR: V. C. Peterson, H. B. Bomberger

CONTRACT: AF33(616)-7288, Crucible Steel Co.

ABSTRACT: Work was done to develop vanadium-base alloys having improved mechanical properties at elevated temperatures. The screening criteria included density-corrected hot hardness at 1800°F as well as forgeability performance at 2300°F. The hot-hardness apparatus designed for the work permits loading up to 14 specimens at one time and testing up to 1800°F in vacuum. The validity of hot hardness as a hot strength parameter was established by a correlation with hot-strength data. In the exploratory work, 174 binary and multi-addition alloys were levitation-melted in 10-gram charges. Based on these studies of the microstructures, forgeability, and hot hardness of these alloys, 14 compositions were selected as most promising and were scaled up to larger melts. Screening data (rollability, hot hardness, and hot tensile properties) on the larger melts showed three compositions to be superior: V-20Cb-5Ti, V-25Mo-2.5Hf, and V-35Cr-1Zr. These alloys demonstrated better forgeability and superior 2000°F tensile strength than the well known V-20Cb-5Ti alloy. Iron and tin were also found to be promising alloying elements for further study.

ASD TDR 62-730

August 1962

SUBJECT: AN APPLICATION OF LINEAR HARDENING PLASTICITY THEORY TO CYCLE AND PATH DEPENDENT STRAIN ACCUMULATION

INVESTIGATOR: P. D. Schwiebert, G. J. Moyar

CONTRACT: AF33(616)-8177, University of Illinois

ABSTRACT: This paper is a combination of four distinct but closely related topics. The first is a documentation of the existence of cycle and path dependent plastic deformation. The second is a resume' of existing plasticity theories to determine if any existing theory in the realm of mechanics of solids can include the observed phenomenon. The third and essentially original section involves the specialization of an existing theory of inelastic deformation. Included is a discussion of the nature of the specialized theory from a plasticity view-point and the application of the theory to a particular complex cyclic stress history. Equations are developed that predict, as a function of cycles, the plastic strain accumulation under conditions of constant axial stress and alternating shear stress in a thin wall tube. The fourth topic is a discussion of present experimental results in the light of theoretical applicability and suggested modifications to include a greater range of material behavior.

VIBRATIONS

WADD TR 60-540

September 1962

SUBJECT: BIBLIOGRAPHY AND TABULATION OF DAMPING PROPERTIES OF NON-METALLIC MATERIALS

INVESTIGATOR: S. H. Chi

CONTRACT: AF33(657)-7453, University of Minnesota

ABSTRACT: This bibliography was compiled as an aid for those people interested in damping research and related fields. It contains a nearly complete list of references on the damping properties of non-metallic materials for the period from 1929 to 1959, together with an index of nomenclature, units, and test methods. Tabulations of the in-phase and out-of-phase components of Young's modulus and the shear modulus for the various materials are also shown, and graphical representations of experimental data indicate the loss factor values for different materials. Those values of the loss factor are compared to those of the representative structural metals.

A detailed code classification system for the field of damping is also described, and an abstract of each referenced article together with a coded supplement is also included in this report. The code is provided to aid those using the ASM-SLA punched-card system.

ASD TDR 62-8

February 1963

SUBJECT: DEVELOPMENT OF ULTRA-SONIC TECHNIQUES FOR DEFECT EVALUATION

INVESTIGATOR: J. B. Ramsey, W. M. Rowe

CONTRACT: AF33(616)-6793

ABSTRACT: The results of investigations to determine the effects of several metallurgical and acoustical variables on the ultrasonic signal strength using commercially available ultrasonic flaw detection equipment are reported. Applied to various metals used in aerospace structures and components, these investigations resulted in the separation and determination of important acoustical properties that were expected to indicate the cause for differences in the transmission of ultra-sonic energy in the various materials. A method was developed for applying correction factors to test blocks of one metal in order to estimate the size of defects in other metals. Beam collimation techniques were studied to determine optimum conditions for detecting defects and displaying them on both cathode ray image and C-Scan (plan View) facsimile paper recordings. Some investigations were also performed to separate and identify shear and surface (Rayleigh) waves.

ASD TDR 62-431

February 1963

SUBJECT: DISLOCATION MOBILITY & PINNING IN HARD MATERIALS
THROUGH INTERNAL FRICTION STUDIES

INVESTIGATOR: P. Southgate

CONTRACT: AF33(616)-8132, Armour Research Foundation

ABSTRACT: The internal friction of covalently bonded and partly covalently bonded materials is being investigated at elevated temperatures. The apparatus operates on the electrostatic drive system, and covers the range 0 to 1500°C, 1 to 200 kc/s. Single crystals of silicon, silicon carbide, and zinc oxide have been measured. A steady rise of internal friction is seen in deformed silicon specimens above 500°C, which appears to be thermally activated with an activation energy of 1.61 ± 0.02 e V. The rise is attributed to the dislocation damping; its magnitude is approximately proportional to the dislocation density and inversely to the frequency. Quantitative identification can be made with Brailsford's abrupt-kink theory of dislocation damping if a reasonable kink density is assumed, in which case the activation energy is that of kink mobility.

ASD TDR 62-680

July 1962

SUBJECT: AN EXPERIMENTAL INVESTIGATION OF THE RANDOM
EXCITATION OF A TAIL-PLANE SECTION BY JET NOISE

INVESTIGATOR: B. L. Clarkson, R. D. Ford

CONTRACT: AF61(052)-504, University of Southampton

ABSTRACT: The response of a section of tailplane structure to both discrete and random noise pressures has been studied in detail. Initially, the specimen was mounted behind a jet engine and the induced strains were analyzed with the object of determining both resonant frequencies and the corresponding modes of vibration. During these tests, a survey was made of the spectrum and correlation pattern of the jet noise on the surface of the model. Secondly, the specimen was mounted in front of a loud-speaker in an acoustics laboratory and the structural resonances were excited by means of discrete frequency sound. The mode shapes were studied in detail with the aid of a stroboscope. It is concluded that tailplane skin on this particular piece of structure only responds to any significant degree in one structural mode. Although reasonable comparison has been obtained between the random and discrete tests, it was not possible to calculate the induced stresses using the observed mode shapes and measured pressure excitation.

ASD TDR 62-681

July 1962

SUBJECT: FURTHER ANALYSIS OF THE RANDOM VIBRATIONS OF THE CARAVELLE TEST SECTION
INVESTIGATOR: B. L. Clarkson, R. D. Ford
CONTRACT: AF61(052)-332, University of Southampton
ABSTRACT: The vibrations, induced by jet-noise, of a row of skin panels in the side of the rear fuselage of a Caravelle aircraft have been analyzed. It has been found that up to three adjacent panels may couple together in the fundamental stringer-twisting mode in which adjacent panels are out-of-phase. The vibrations of the skin panels in the upper surface of the outboard elevator have also been studied and here it has been found that the ribs act as rigid supports. Any pair of panels between two ribs, however, are vibrationally coupled, although the mode-shapes have not been satisfactorily determined.

ASD TDR 62-701

July 1962

SUBJECT: TORSIONAL VIBRATIONS OF A FREE RECTANGULAR PLATE
INVESTIGATOR: D. J. Mead, R. F. Beresford
CONTRACT: AF61(052)-504, University of Southampton, England
ABSTRACT: This report considers effect of bending distortion of cross-section on torsional modes and frequencies of a uniform rectangular plate. Bi-harmonic plate equation is integrated to yield the transcendental frequency equation for modes of any specified longitudinal wave-length, the effects of shear distortion and rotary inertia associated with bending being ignored. The long wave-length solution is shown to be well approximated to by the results from the simple torsional theory based on the St. Venant and Wagner-Kappus torsion theories. At shorter wave-lengths, the simple theory still gives a good approximation to the frequencies of vibration, but considerable distortion of the cross-section is found to occur. Certain limiting conditions of very short wave-length are also investigated.

ASD TDR 62-702, Part I

July 1962

SUBJECT: THE RESPONSE OF A MODEL STRUCTURE TO NOISE:
Flat Panels
CONTRACT: AF61(052)-504, University of Southampton
INVESTIGATOR: R. D. Ford
ABSTRACT: A model structure consisting of a large flat sheet, sub-divided into small panels has been excited by discrete frequency noise in an acoustics laboratory. The response of the panels has been studied in detail and the mode shapes identified.

A Rayleigh-Ritz energy method has been used to

ASD TDR 62-706, Part I (Continued)

calculate the frequencies of two of the modes that have been excited and also a number of regular modes that could theoretically exist.

Attempts have been made to calculate the response of the structure to the applied noise pressures in observed modes of vibration.

ASD TDR 62-706, Part II

May 1963

SUBJECT: THE RESPONSE OF A MODEL STRUCTURE TO NOISE:
Curved Panel
INVESTIGATOR: B. L. Clarkson, R. D. Ford
CONTRACT: AF61(052)-504, University of Southampton
ABSTRACT: A model structure consisting of a curved sheet sub-divided into small regular panels by frames and stringers has been excited by discrete frequency sound. The response of the panels has been studied in detail and several mode shapes identified.

A group of 'fundamental' type of modes has been found to exist and the mode shape and frequency of the lower and upper bound of this group have been calculated by means of the energy method.

The theoretical response in the upper bounding mode show an order of magnitude agreement with experiment.

ASD TDR 62-1030

May 1962

SUBJECT: DAMPING OF MATERIALS UNDER BIAXIAL STRESS
INVESTIGATOR: P. J. Torvik, S. H. Chi, B. J. Lazan
CONTRACT: AF33(657)-7453, University of Minnesota
ABSTRACT: A new theory of combined stress damping is developed and evaluated on the basis of experimental results of several materials. It is concluded that the new theory should be limited in application to materials whose dominant damping mechanisms is known to be plastic deformation. It appears that a separate theory of combined stress damping may be required for each mechanism of damping.

ASD TDR 62-1031

December 1962

SUBJECT: THERMOELASTIC DAMPING
INVESTIGATOR: L. E. Goodman, C. S. Chang, A. R. Robinson
CONTRACT: AF33(657)-7453, University of Minnesota
ABSTRACT: The non-linear coupled field equations of thermo-elasticity are herein solved for the quasi-static behavior of a solid

ASD TDR 62-1031 (Continued)

bounded by two parallel planes. The mechanical energy converted to heat during each cycle of the loading process is explicitly evaluated and its spectral variation computed. From this exact solution an approximate, but quite general, expression for the thermoelastic energy dissipated in solids as a result of elastic deformation is developed.

ASD TDR 62-1096

January 1963

SUBJECT: THE DAMPING OF ALUMINUM HONEYCOMB SANDWICH BEAMS
INVESTIGATOR: D. J. Mead, G. R. Froud
CONTRACT: AF61(052)-504, University of Southampton
ABSTRACT: The various sources of damping in a transversely vibrating plate of honeycomb construction are considered. and an expression is derived for the total damping ratio of an unjointed honeycomb sandwich beam. It is shown that the energy dissipation by reason of bending strain in the adhesive layer is of major importance. A damping parameter is defined whereby the amount of damping supplied by bending energy loss in the adhesive may be predicted for a given adhesive layer. Non-linearity of the adhesive damping is taken into account. The parameter and the non-linear coefficients have been derived experimentally from tests on honeycomb sandwich beams.

For large wavelength vibrations, it has been found that the damping ratio of an unjointed beam or plate is inversely proportional to the skin thickness, showing that the adhesive bending loss is the predominant source of damping. The low values of damping measured (.01% to .04% of critical) suggest that the contributions of the material and adhesive of a honeycomb plate structure to the total damping will be very small compared with the contributions expected from the joints at the panel boundaries.

ASD TDR 62-1098

January 1963

SUBJECT: STRUCTURAL CONFIGURATIONS FOR INCREASED RESONANCE FATIGUE LIFE
INVESTIGATOR: G. Kurtze
CONTRACT: AF61(052)-547, Grunzweig & Hartmann AG, Ludwigshafen, Germany
ABSTRACT: After a short summary of the existing methods used for the damping of bending waves, an analysis is given for the calculation of three types of sandwich panels with dissipative cores. These sandwich panels seem to represent the optimum solution with

ASD TDR 62-1098 (Continued)

respect to high damping and are characterized by equally thick metal skin layers. The core may be either thin or thick as compared to the skins or may be composed of a thin dissipative layer and a spacer.

In the second part of the report, experimental data are given, obtained from loss tangent measurements as a function of temperature with sandwich panels of the above mentioned three types.

The report furthermore gives an outline of future work in which the optimum samples determined in the first part of the investigations will be made subject to fatigue testing.

ASD TDR 63-91

January 1963

SUBJECT: THE UNIVERSITY OF SOUTHAMPTON RANDOM SIREN FACILITY
INVESTIGATOR: B. L. Clarkson, S. A. Pietruszewicz
CONTRACT: AF61(052)-504, Southampton University, England
ABSTRACT: The report describes the construction and performance of the Southampton University Random Siren Facility. The configuration is of the grazing incidence type designed to take specimens 3 ft. x 2 ft. The maximum noise level which can be achieved in the working section is 150 db in the random mode of operation and somewhat higher for discrete frequencies. The noise source is an electromagnetic air modulator capable of broad band response in the frequency range up to 800 - 1000 cps. A lower frequency limit of 110 cps is imposed by the exponential horn. Thus, a noise pressure spectrum approximating that associated with jet noise can be obtained.

WELDING AND BRAZING

WADC TR 59-404, Part III

July 1962

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

INVESTIGATOR: N. Bredzs, et al

CONTRACT: AF33(616)-6882, Armour Research Foundation

ABSTRACT: It has been shown thermodynamically and confirmed experimentally that a high vapor pressure constituent of a given filler metal can be volatilized in a shorter time, or at a lower temperature, or to a greater extent, if to the given filler metal are added other constituents, which have a larger free energy of solution in the given filler metal than the volatile constituent has. Specifically, it has been experimentally demonstrated that additions of aluminum, chromium, silicon, and to a lesser extent iron and germanium, increase the rate of volatilization of indium from nickel. This order of effectiveness confirms, according to available thermo-chemical data, the above hypothesis. In a practical sense, the effects of the joint geometry on the ability to volatilize are discussed. Limitations on joint depth must be recognized if appreciable volatilization is to be obtained with reasonable times at temperature.

ASD TR 61-313, Part II

January 1963

SUBJECT: DEVELOPMENT OF LOW TEMPERATURE BRAZING ALLOYS
FOR TITANIUM HONEYCOMB SANDWICH MATERIALS

INVESTIGATOR: W. C. Troy

CONTRACT: AF33(616)-7249, Solar Aircraft

ABSTRACT: For brazing titanium under 1100°F, binary information formed the basis of selecting two promising ternary basis alloys for development, (1) Ag-Cu-Ge and (2) Ag-Cu-Sn. Melt and flow behavior was favorable with Ag-Cu-Sn base alloys, but work was discontinued because of excessive joint brittleness related to inter-metallic films formed on the substrate. Alloys based on the Ag-Cu-Ge ternary also formed inter-metallic films, but the joints had intermediate strength which justified evaluation. Average shear strength of lap joints varied from 10,750 psi for aged specimens to 11,900 psi for specimens after brazing at 1100°F. These marginal properties would justify only limited applications of the Ag-Cu-Ge based alloys.

ASD TR 61-678

April 1962

SUBJECT: DETERMINATION OF CAUSES OF CRACKING IN WELDING
AGE HARDENABLE HIGH TEMPERATURE ALLOYS

INVESTIGATOR: B. S. Blum

CONTRACT: AF33(616)-7670, Republic Aviation Corp.

ABSTRACT: Tensile, hot ductility, metallographic and weldability studies were conducted to determine the cause of base metal cracking in A-286 weldments. A mechanism for cracking was proposed, based on grain size, interstice formation by grain boundary sliding or Fe₂Ti-Fe liquation, embrittlement by Ni₃(Al, Ti) general and Ti-Fe liquation, embrittlement by Ni₃(Al, Ti) general and TiC grain boundary precipitation on insufficiently rapid cooling. The effect of stress on weld metal cracking was studied with variable restraint specimens, and optimum filler wires was evaluated on segmented finger specimens.

The strain-age cracking study conducted on Rene' 41 and Inconel X concentrated on the non-equilibrium time between the annealed condition and the aged condition in the 1050° to 1500°F temperature range. Tensile and dilatometric studies indicated a possible transient embrittlement on initial heating. The time, temperature and prior material history dependence of the aging contraction was studied, and based on these results, and metallographic analysis of Inconel X, a mechanism to account for strain-age cracking was developed. The difficulties encountered in developing a valid strain-age cracking specimen are discussed.

ASD TDR 62-292

May 1962

SUBJECT: INVESTIGATION OF WELDING OF COMMERCIAL
COLUMBIUM ALLOYS

INVESTIGATOR: J. M. Gerken, J. M. Faulkner

CONTRACT: AF33(616)-7796, Thompson Ramo Wooldridge

ABSTRACT: An investigation was made of the welding characteristics of three commercial columbium base alloys, FS82, D31, and F48. Tungsten inert gas, electron beam spot, and flash butt welding methods were included in this investigation. The effect of the welding variables travel speed, shielding gas composition and purity, filler metal additions, pre-heat and post heat were studied. Thermal cycles were measured in the fusion zone and heat affected zone of TIG welds to help explain mechanical properties and microstructure on the basis of physical metallurgy of each alloy. The effect of welding on all alloys was to increase the ductile to brittle transition temperature. This temperature was increased 200°F for FS82, 800°F for D31, and 500°F to 800°F for F48 over that of the as received wrought sheet. Of several post heat treatments investigated,

ASD TDR 62-292 (Continued)

May 1962

those most beneficial in lowering the transition temperature of welds were: 2000°F for 4 hours for FS82; 2100°F for 24 hours for D31; and 2500°F for 4 hours for F48. The electron beam welding process consistently produced more ductile welds than the TIG process.

ASD TDR 62-317

June 1962

SUBJECT: INVESTIGATION TO DETERMINE CAUSES OF FISSURING
IN STAINLESS STEEL AND NICKEL-BASE-ALLOY WELD
METALS

INVESTIGATOR: J. N. Cordea, R. M. Evans, D. C. Martin

CONTRACT: AF33(616)-7702, Battelle Memorial Institute

ABSTRACT: This report summarizes the first year's work in a longer term research program being conducted to investigate causes of fissuring in Type 310 stainless steel and Inconel alloy weld metals. Characteristics of fissures in welds in hot-tension specimens were studied and materials examined were: (1) Inconel and Type 310 stainless steel welds made from commercial electrodes, (2) Hot-tension specimens made from commercially available Inconel and Type 310 stainless steel materials (3) Hot-tension specimens made from alloys prepared with electrolytic grade of iron, chromium, and nickel. All fissures were intergranular or interdendritic. In hot-tension specimens, they were usually oriented perpendicular to the tensile axis. The onset of fissuring corresponded to a drop in hot ductility and hot strength at high temperatures. Commercial alloys having high impurity concentrations always had lower hot-tensile properties than high-purity vacuum-melted alloys. When controlled amounts of carbon, silicon, and sulfur were added to vacuum-melted heats of Type 310 stainless steel, their hot-tensile properties approached those of the commercial alloys. The occurrence of intergranular fissuring was attributed to increased amounts of grain-boundary segregation in the specimens. Electron-probe analyses and metallographic examinations of the grain boundaries verified this effect. Test results showed that large amounts of grain-boundary segregation occurred when: (1) the original impurity concentration in the alloy was high, (2) when large grains were formed in the microstructures at the high temperatures.

ASD TDR 62-993

March 1963

SUBJECT: INVESTIGATION OF WELDABILITY OF HOT-WORK TOOL
STEELS AND A TITANIUM ALLOY

INVESTIGATOR: L. A. Luini, N. A. Sinclair

CONTRACT: AF33(616)-8044, General Dynamics Corp.

ASD TDR 62-993 (Continued)

ABSTRACT: The weldability of H-11 and D6-AC steels and the B-120VCA Ti alloy was investigated. Vacuum-melted and air-melted H-11 steels were compared for both base metal weldments. The vacuum-melted H-11 and D6-AC steels were fully heat treated before welding and given various post-weld treatments. Welding the H-11 steel after full heat treatment resulted in lower notch strength than when fully heat treated after welding. An isothermal transformation post-weld treatment which produced mixed bainitic and tempered martensitic structures gave higher notch strength than the conventional tempered martensitic structures for both base metal and weld metal of D6-AC steel. The B-120VCA titanium alloy showed good ductility in the as-welded condition. Age and overage treatments resulted in lower ductility and notch strength than the base metal. Grain refinement by application of ultrasonic vibrations during welding was obtained. The effects are dependent on material type, welding parameters, vibrational source to weld distance, mode of vibration, weldment geometry, and the method of coupling the vibrational source to the weldment.

ASD TDR 63-88

January 1963

SUBJECT: JOINING OF REFRACTORY METALS BY BRAZING AND DIFFUSION BONDING
INVESTIGATOR: W. R. Young, E. S. Jones
CONTRACT: AF33(616)-7484, General Electric Co.
ABSTRACT: Brazing alloy systems based on Cb, V, Ti, and Zr were evaluated for melting range, wettability and flow, and metallurgical compatibility with Cb-base alloys and unalloyed tungsten. Several alloys were identified and evaluated for effects on high temperature strength and base alloy transition temperature.

Investigation into achieving brazed joint failure temperature above the original brazing temperatures indicates that 500°F increases in failure temperature could be attained. Applied stress during testing was found to be the most critical parameter.

The characteristics of diffusion bonding refractory alloys to each other and to Fe-, Ni-, and Co-base alloys have been investigated by metallographic examination, post-bonding thermal exposure, and bonding shear strength determination.

ASD TDR 63-132

February 1963

SUBJECT: ELECTRON BEAM WELDING OF AEROSPACE MATERIALS
INVESTIGATOR: W. I. Kern, L. E. Lubin
CONTRACT: AF33(657)-7763, United Aircraft Corp.
ABSTRACT: High-voltage electron beam welding techniques were developed for B12OVCA titanium, D6AC steel, molybdenum-0.5% titanium to tungsten, and beryllium. Welds were produced in material thicknesses of 0.125 inch for the B12OVCA titanium; 0.290 inch and 0.090 inch for the D6AC steel; 0.100 inch, 0.050 inch, and 0.005 inch for the molybdenum-0.5% titanium welded to tungsten; and 0.040 inch for the beryllium. The effects of thermal treatments (aging and heat treating) were investigated in the B12OVCA and D6AC by welding and testing a variety of weld/thermal treatment combinations. Butt welds of each material thickness, joint combination, and thermal treatment (where applicable) were evaluated for mechanical properties by tensile testing at temperatures to 2200°F and bend testing at temperatures to 1000°F. Internal-notch tensile properties of B12OVCA titanium and D6AC steel were determined at room temperature. Results of the metallographic and mechanical property investigations of the electron beam welds were compared with base-metal properties and with published data for other joining techniques.

ASD TDR 63-137

March 1963

SUBJECT: STRAIN AGING OF TANTALUM
INVESTIGATOR: P. L. Hendricks
ABSTRACT: An investigation was conducted on electron beam melted tantalum to determine the interstitial solute atom responsible for strain aging in the temperature range of 100° to 200°C.

Strain aging was investigated using return of the yield point technique. The interstitial atom responsible for strain aging was determined to be oxygen when the material was essentially free of hydrogen. Hydrogen when present, changes the kinetics of strain aging and contributes in some measure to the strain aging observed when pre-straining and testing is accomplished at -73°C.

ADHESIVES

WADC TR 55-491, Part VI

June 1962

SUBJECT: RESEARCH ON ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES
INVESTIGATOR: R. J. Forlano, et al
CONTRACT: AF33(616)-6192, University of Illinois
ABSTRACT: Strong and relatively shock resistant inorganic adhesives were developed and improved by addition of metal fillers and recrystallizable materials to the glassy phase. Selected oxides added to develop covalent type bonds improved high temperature strength. Recrystallized type adhesives, however, would not develop a strong cohesive bond unless a glassy phase was present.

Test data showed that tensile shear strength values of 5000 psi at room temperature, 3000 psi at 800°F, 1500 psi at 1000°F and 800 psi at 1500°F could be expected of ceramic adhesives as presently developed.

WADC TR 55-491, Part VII

October 1962

SUBJECT: ELEVATED TEMPERATURE RESISTANT CERAMIC STRUCTURAL ADHESIVES
INVESTIGATOR: R. J. Forlano, et al
CONTRACT: AF33(657)-7422, University of Illinois
ABSTRACT: Ceramic-metal filler adhesives were developed in bonds to Rene' 41 alloy adherends displayed tensile lap-shear strength in excess of 3000 psi over a temperature range of room temperature to 1900°F. The critical materials and processing variables for ceramic-metal adhesive combinations are defined, namely, - maximum softening point of the ceramic phase, wettability of the adherend and bonding metals by the ceramic phase, solubility of the adherend metal surface oxides in the ceramic phase, and minimum firing temperature to permit softening of the metal phase. A technique was developed for observing polished cross sections of lap-joints at elevated temperatures with a hot stage microscope. Glassy type adhesives were evaluated for optimum firing temperature and time, application weight, blending of hard and soft frits, gas content, heat treatment after firing, and addition and precipitation of crystal phases.

May 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON INORGANIC HIGH TEMPERATURE ADHESIVES FOR METALS AND COMPOSITE CONSTRUCTIONS

INVESTIGATOR: O. E. Johnston, W. P. Robbins

CONTRACT: AF33(616)-7196, Aeronca Mfg. Corp.

ABSTRACT: Research on high temperature inorganic non-polymeric adhesives for metal to metal bonding for applications as high as the 2000°F to 3000°F range. The following objectives were accomplished: (a) fabrications and evaluation of experimental inorganic, non-polymeric adhesives, (b) preparation and application of the inorganic adhesives to the metal adherends, (c) improvement of flexibility, toughness, and strength of the adhesive to obtain a highly efficient bond for use in joining the high temperature resistant metals such as 17-7 PH, PH 15-7 Mo, AM-350, Inconel "X", (d) processing research for optimum joining of honeycomb sandwich structures with inorganic adhesives, (e) evaluation of bonded honeycomb panels for structural integrity and capability, (f) refractory metals inorganic adhesive bonded with retention of useful bond strength in the 1800°F to 200°F temperature range.

May 1962

SUBJECT: HEAT RESISTANT LAMINATING RESINS

INVESTIGATOR: L. Gilman, et al

CONTRACT: AF33(616)-6956, Monsanto Chemical Co.

ABSTRACT: Polyisocyanurate laminates prepared had 60,000 to 80,000 psi original R. T. flexural strengths and flexural moduli approximately 4.5×10^6 psi. At 500°F, flexural strengths were 30,000-45,000 psi. Good strength at the target 700°F was not attained. Preliminary results indicate that polyisocyanurate resins have potential as adhesives for metal to metal bonding.

Glass fabric reinforced laminates were made using chelate polymers. Although these laminates had low strength at room and elevated temperatures, the chelate resins are the first known to be sufficiently advanced for laminate fabrication. Progress was made in gaining a better understanding of the variables affecting formation of both types of resins. The decomposition rate at 700°F is tolerably low. Some of the apparent problems with both types of resins are processing steps to satisfactory laminates and improvement in mechanical properties.

January 1962

SUBJECT: HEAT RESISTANT LAMINATING RESINS
INVESTIGATOR: L. Gilman, et al
CONTRACT: AF33(616)-7853, Monsanto Research Corp.
ABSTRACT: Polyisocyanurate laminates were made with original R.T. flexural strengths from 60,000-75,000 psi and flexural moduli near 3×10^6 psi. Flexural strengths at 500°F were 40,000-50,000 psi. Mechanical properties at 500°F have been improved, but good strength at 700°F has not been obtained. A soluble, high molecular weight, infusible thermally stable chelate resin has been prepared with mechanical toughness at room and elevated temperatures. Laminates made with this polymer, however, yielded low strength materials because of hydrolysis during fabrication. Laminates were prepared from various phenolic polymers containing siloxy groups. Some required the use of new laminating techniques. Laminates prepared from low molecular weight phenyl-T polymer (polyphenylsil-sesquioxane) and bis-silylarene monomers had flexural strengths of 19,000 psi at room temperature. High molecular weight phenyl-T polymers could not be plasticized enough to allow homogeneous molding.

October 1962

SUBJECT: RESISTANCE OF ADHESIVE-BONDED METAL LAP JOINTS TO ENVIRONMENTAL EXPOSURE
INVESTIGATOR: W. Z. Olson, H. W. Eickner, R. M. Lulling
CONTRACT: DO33(616)-61-06, Forest Products Laboratory
ABSTRACT: An investigation was made of the relative durability of metal-bonding adhesives in exterior exposure at two sites, Miami, Florida, and Panama Canal Zone. Twelve sets of lap-joint panels of clad 2024-T3 aluminum alloy bonded with 12 different adhesives, and one set of 17-7PH corrosion-resisting steel bonded with another adhesive, were exposed at these two sites. Panels were exposed while stressed in bending and while unstressed. Sets of panels were removed and tested periodically through 3 years of exposure. In addition, several short-term laboratory exposure conditions were used to determine whether such tests might provide information similar to the long-term exposure.

In general, stressed specimens decreased in strength more than the unstressed specimens, Florida exposure caused a greater loss in strength than the Panama exposure, and exposure to the weather was more severe than laboratory exposure cycles.

ASD TDR 62-573

November 1962

SUBJECT: EFFECT OF RATE OF TEMPERATURE RISE ON ADHESIVE BONDS IN STEEL
INVESTIGATOR: M. L. Selbo, R. M. Lulling
CONTRACT: AF33(616)-61-06, Forest Products Laboratory
ABSTRACT: Data are presented on the effect of four different rates of temperature rise to reach 650°F (the test temperature) when heating with infrared heat and of one rate of oven heat on the shear strength of steel lap-joint specimens bonded with three different commercial adhesives.

All three adhesives showed serious reduction in shear values when heated to 650°F, and a silicone-modified epoxy (Metlbond 311) was the least affected by the test conditions used. Infrared heat appeared to cause more deterioration to the adhesive bonds than oven heating.

ASD TDR 62-775

August 1962

SUBJECT: PLASTIC BEHAVIOR IN INORGANIC OXIDE-PHOSPHATE BONDED MATERIALS
INVESTIGATOR: A. G. Wehr, J. H. Lauchner, J. Montgomery
CONTRACT: AF33(616)-8134, Mississippi State University
ABSTRACT: Previous research has shown that certain combinations of phosphate bonded metallic oxides exhibit desirable plastic deformation under applied stress. In this program, the relationships between microstructural formations and crystallographic phases within these bodies and plastic deformation characteristics of the cured specimen were investigated.

The oxide-phosphate bond was obtained by reacting measured amounts of aluminum oxide, cobalt oxide and zinc oxide with phosphoric acid for four hours at 200°C. The resulting powders were then pressed into rectangular bars and sintered for four hours at 650°C. A laboratory apparatus was designed and built which was used to determine the modulus of elasticity of the oxide-phosphate specimens by measuring the natural or resonant frequencies of the rectangular specimens. The modulus of rupture of the test specimens was determined by the flexure method using a three point loading apparatus.

The petrographic and the metallographic microscopes were used to observe the highly stressed areas in the deformed specimens. Micro cracks were discovered which were very irregular and almost circular in path.

ASD TDR 62-775 (Continued)

X-ray diffraction, diffraction, differential thermal analysis, and vibration damping techniques were used to identify the phases present in the oxide-phosphate material, and to determine the possible crystallographic inversions that can occur in the same material. It was discovered that bars which were brittle contained two phases that were not present in bars that were capable of being plastically deformed.

CERAMICS AND CERMETS

WADC TR 59-316, Part IV

July 1962

SUBJECT: MECHANISM OF WEAR OF NONMETALLIC MATERIALS
INVESTIGATOR: C. H. Riesz, H. S. Weber
CONTRACT: AF33(616)-6920, Armour Research Foundation
ABSTRACT: Friction and wear of single-crystal sapphire surfaces were studied at 10^{-6} mm Hg over a temperature range of 25 to 1550°C. Highest coefficients of friction were found at ambient temperatures, particularly when basal planes were in sliding contact. Under certain conditions, fracture tracks were produced. Orientation dependence was observed up to approximately 1300°C. Friction-related basal dislocations were detected at temperatures as low as 600°C. A proposed mechanism of friction and wear suggests that two types of frictional junctions are formed. At ambient temperature, the junctions are adhesive in nature, highly orientation dependent, and related to surface phenomena. At elevated temperature friction is only slightly influenced by orientation, and plastic deformation, based mainly on the highly active basal slip system, leads to "weld-junctions" and flow in bulk. The relative contributions of the two types of junctions provide anomalous coefficients of friction for sapphire at or around 600°C both under vacuum and in air.

WADC TR 59-603, Part III

July 1962

SUBJECT: FRICTION AND WEAR AT ELEVATED TEMPERATURES
INVESTIGATOR: E. Rabinowicz, M. Imai
CONTRACT: AF33(616)-7648, Massachusetts Inst. of Technology
ABSTRACT: A number of substances have been examined for suitability under sliding conditions in the range from room temperature to 2000°F. Low melting metals applied as surface coatings show a peak in their friction at the melting points, unless they form an oxide with good lubricating ability. Pyrolytic boron nitride and graphite have the same friction and wear properties as do ordinary forms of these materials. A discussion is presented on how information may be obtained systematically from friction-temperature runs, and examples of the various techniques are given.

WADC TR 59-603, Part IV

March 1963

SUBJECT: FRICTION AND WEAR AT ELEVATED TEMPERATURES
INVESTIGATOR: E. Rabinowicz, M. Imai
CONTRACT: AF33(616)-7648, Massachusetts Inst. of Technology
ABSTRACT: Measurements have been carried out of the friction coefficient as a function of temperature using surfaces of

WADC TR 53-373 Sup 10

WADC TR 59-603, Part IV (Continued)

stainless steel covered by low-melting metals and non-metals applied in powder form. Some work has also been done with a few other metal and non-metal surfaces. In cases where the interaction between the low melting substance and sliding surface is high, as revealed by the occurrence of wetting, the friction reaches a peak just below the melting temperature of the substance, and then drops to considerably lower values just above the melting point. The peak below the melting temperature is associated with the formation of large adhering fragments of the low melting substance on the sliding surface. When there is no wetting, the low-melting substance has, either below or above its melting point, essentially no effect on the friction.

WADDTR 60-184, Part III

July 1962

SUBJECT: EFFECT OF BASIC PHYSICAL PARAMETERS ON ENGINEERING PROPERTIES OF INTERMETALLICS
INVESTIGATOR: J. H. Westbrook, D. L. Wood
CONTRACT: AF33(616)-7714, General Electric Co.
ABSTRACT: Grain boundary hardening is found in this investigation to exist in many intermetallic compounds having a stoichiometric excess of active metal component and is shown to be associated both with the anomalously high ductile-brittle transition temperatures common in these materials and with the "pest" phenomenon occurring in certain intermetallics. Although experiments show clearly that such grain boundary hardening is due to the adsorption of gaseous impurities such as oxygen and/or nitrogen and their segregation to grain boundary regions, the manner in which the resistance to plastic deformation is increased in these areas is not clear. The most likely mechanism is the formation of a dislocation network as found by Ainslie et al, in FeS alloys. Ternary additions as well as appropriate annealing treatments are shown to be a promising approach to alleviating intergranular brittleness in these materials.

WADD TR 60-184, Part IV

April 1963

SUBJECT: EFFECT OF BASIC PHYSICAL PARAMETERS OF ENGINEERING PROPERTIES OF INTERMETALLICS
INVESTIGATOR: J. H. Westbrook, D. L. Wood
CONTRACT: AF33(616)-7714, General Electric Co.
ABSTRACT: This report is a continuance of the authors' mechanical property measurements and grain boundary studies on AgMg. A general survey of the effects of dilute ternary solute additions on the flow stress of AgMg has been made. A unique, efficient

WADD TR 60-184, Part IV (Continued)

device to measure ductile-brittle transition temperatures on wire specimens has been designed and built. Using this device, the effects of composition, grain size, strain rate, and ternary solute additions on the transition temperature have been documented for AgMg. Further studies of the kinetics of solute-induced grain boundary hardening indicate that the phenomenon is much more complex than previously supposed.

WADD TR 60-749, Part II

April 1962

SUBJECT: FACTORS AFFECTING THERMAL SHOCK RESISTANCE OF POLY-PHASE CERAMIC MODIES
INVESTIGATOR: D. P. H. Hasselman, P. T. B. Shaffer
CONTRACT: AF33(616)-6806, The Carborundum Co.
ABSTRACT: An investigation of the factors which affect the thermal shock resistance of polyphase ceramic systems has been conducted using the model system zirconium carbide-graphite. The effect of graphite on the physical properties of the zirconium carbide is to increase the strain at fracture, thermal conductivity and thermal diffusivity, thereby increasing thermal shock resistance. Of significance is the observation that the presence of the graphite substantially reduces the damage which results from thermal shock. Explanations are given and suitable "thermal shock damage resistance parameters" are introduced. Emissivity was introduced as a variable affecting thermal shock resistance, when radiation is the principal mechanism of heat transfer. A quantitative theory for thermal shock by radiation on heating was developed and confirmed experimentally.

WADD TR 60-782, Part IV

May 1962

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURES: On the Existence of Gaseous Sulphides of the Transition Elements
INVESTIGATOR: R. Colin, P. Goldfinger, M. Jeunehomme
CONTRACT: AF61(052)-225, Universite de Bruxelles, Belgium
ABSTRACT: Thermodynamic considerations permit us to predict the mode of vaporization of transition element sulphides. MnS is the only clear case where the gaseous sulphide molecule has an appreciable concentration in the saturated vapor. This is confirmed by a mass spectrometric investigation which gave us the measurement

$$D_0^{\circ} (\text{MnS}) = 65 \pm 5 \text{ kcal/mole.}$$

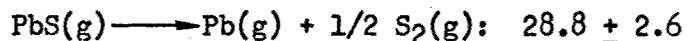
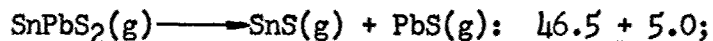
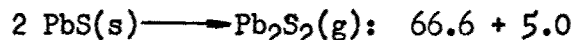
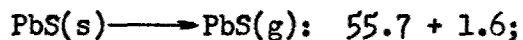
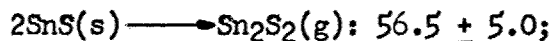
April 1962

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURE: Thermodynamic Study of Tin and Lead Sulfide Using a Mass Spectrometer.

INVESTIGATOR: R. Colin, J. Drowart

CONTRACT: AF61(052)-225, University of Brussels, Belgium

ABSTRACT: Reaction enthalpies, ΔH_{298}° , in kcal/mole, determined were $\text{SnS(s)} \longrightarrow \text{SnS(g)}: 52.6 \pm 1.6$ kcal/mole;



May 1962

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURE: Mass Spectrometric Study of Gaseous Species in the System Boron-Carbon

INVESTIGATOR: G. Verhaegen, et al

CONTRACT: AF61(052)-225, University of Brussels

ABSTRACT: The molecules BC , B_2C , and BC_2 have been identified. Their atomization energies have been measured to be $D_0^{\circ}(\text{BC}) = 105 \pm 10$; $D_0^{\circ}(\text{B}_2\text{C}) = 260 \pm 10$; $D_0^{\circ}(\text{BC}_2) = 302 \pm 10$ kcal/mole.

May 1962

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURES: Correlation of Dissociation Energies of Gaseous Molecules and of Heats of Vaporization of Solids Homonuclear Diatomic Molecules

INVESTIGATOR: G. Verhaegen, et al

CONTRACT: AF61(052)-225, University of Brussels

ABSTRACT: Homonuclear diatomic molecules are classified according to the ratio $\alpha = \Delta H_0^{\circ}(\text{vap.M})/D_0^{\circ}(\text{M}_2)$. For transition elements $\Delta H_0^{\circ}(\text{vap.M}) = A - B \sum_i \epsilon_i(M)$. $\sum_i \epsilon_i(M)$ is the excitation energy of the free atom to a set of electronic levels; A and B are empirical parameters. Experimental conditions for observing unknown

WADD TR 60-782, Part IX (Continued)

homonuclear diatomic molecules of transition elements are estimated for $d = 2$ and 5, using calculated self-consistent set of free energy functions.

WADD TR 60-782, Part X

May 1963

SUBJECT: VAPORIZATION OF COMPOUNDS AND ALLOYS AT HIGH TEMPERATURES: The Dissociation Energy of the Group IV-Group VI Molecules
INVESTIGATOR: R. Colin, J. Drowart
CONTRACT: AF61(052)-225, University of Brussels
ABSTRACT: The available spectroscopic and thermochemical data for the dissociation energy of the group IV-group VI MeX molecules are reviewed and the best present values proposed. Correlations of several excited molecular states with atomic products are proposed.

WADD TR 60-866

July 1962

SUBJECT: EXCITATION PROCESSES IN CERAMICS AND THE ANOMALOUS INCREASE IN THERMAL CONDUCTIVITY AT ELEVATED TEMPERATURES
INVESTIGATOR: D. H. Whitmore
CONTRACT: AF33(616)-6194, Northwestern University
ABSTRACT: The problem of the anomalous increase in the observed thermal conductivity on single-phase ceramics at high temperatures has been considered. At temperatures above the onset of this anomalous rise, account has been taken of the possibility that phonon, electronic, and radiative heat transfer, as well as transport of thermal energy by electron-hole pairs, excitons and dissociated gas molecules, may operate simultaneously and individually contribute significantly to the total heat flow. On the basis of reliable conductivity data on monocrystals of single-phase ceramics, estimations have been made on the magnitudes of these high-temperature components which reveal that excited states of low excitation energy may occur in certain ceramics. In these instances, such excited energy-carrying states are able to diffuse down the temperature gradient in the specimen, thereby producing a non-negligible contribution to its observed thermal conductivity.

July 1962

SUBJECT: INVESTIGATION OF INTERMETALLIC COMPOUNDS FOR
VERY HIGH TEMPERATURE APPLICATIONS
INVESTIGATOR: J. Booker, et al
CONTRACT: AF33(616)-6540, The Brush Beryllium Co.
ABSTRACT: Three different areas were studied in a continuing investigation of intermetallic beryllides and silicides for very high temperature applications. The measured properties of Ta_2Be_{17} were unaffected by minor variations in stoichiometry between 800° and $3000^{\circ}F$, whereas best results for WSi_2 were obtained with a material slightly deficient in tungsten. WSi_2 exhibited poorest oxidation resistance at $2200^{\circ}F$.

Results are reported for oxidation-rate studies on $TaBe_{12}Be_{17}$, $ZrBe_{13}$, and Zr_2Be_{17} in the temperature range from 2300° to $2750^{\circ}F$. The vapor pressures for these same compounds were measured between 2300° and $2650^{\circ}F$ using the Knudsen cell technique. For $TaBe_{12}$ the activation energy for oxidation is lower than the vaporization energy in this temperature range. The oxidation products of Zr_2Be_{17} were found to be $ZrBe_5$ and BeO .

Ternary systems of beryllium and silicon with molybdenum, niobium, tantalum, tungsten, or zirconium were studied. The most promising material from these studies was a mixed-phase preparation with the stoichiometry of $MoBeSi$. This material exhibited less than 2 mils penetration when oxidized in air at $3000^{\circ}F$ for 10 hours.

October 1962

SUBJECT: ORDERING IN OXIDE SOLID SOLUTIONS
INVESTIGATOR: H. H. Wilson
CONTRACT: AF33(616)-6870, Clemson College
ABSTRACT: A study was made of solid solutions of magnesium oxide with manganese, iron, and cobalt oxides to determine the effect of extended heat treatment and of controlled furnace atmospheres on the formation of superlattices. Heat treatments involving temperatures up to $1350^{\circ}C$ and times up to 600 hours were used.

Furnace atmospheres were controlled so as to be either neutral, slightly oxidizing, or slightly reducing with respect to the divalent metallic ions.

Indications of ordering were found in those compositions that were heated in reducing atmospheres. A broad diffraction peak was found at $6.4 \text{ \AA}$ which is three times the (002) spacing of the unordered lattices.

ASD TR 61-481

April 1962

SUBJECT: IMPURITY DEPENDENCE OF CREEP OF ALUMINUM OXIDE
INVESTIGATOR: E. K. Beauchamp, G. S. Baker, P. Gibbs
CONTRACT: AF33(616)-6832, University of Utah
ABSTRACT: Polycrystalline sintered compacts of doped Gulton Alucer MC alumina have been deformed in three point beam loading in the temperature range 1000° to 1350°C. Creep consisted of a transient deformation (ϵ_{tr}) superposed on a steady state deformation ($\epsilon_{s.s.} = C_1 t$). The steady state creep rate of specimens doped with MgO or MnCO₃ from 50 to 5000ppm A exp (-E/kT). The activation energy E was found to be 130 Kcal/Mole independent of added impurity. The constant A was independent of MnCO₃. A decrease in A by a factor of 3-5 in samples doped with MgO may have been due to presence of some large grains. It is suggested that steady state creep of fine grained polycrystalline alumina is controlled by diffusion of vacancies.

Transient creep (ϵ_{tr}) occurring on application of stress approximated the relation $\epsilon_{tr} = C_2 (1 - \exp(-t/\tau_1)) + C_3 (1 - t/\tau_2)$. On removal of the load, recovery was observed which followed the relation $\epsilon_{rec} = C_2 (\exp(-t/\tau_1)) + C_3 (\exp(-t/\tau_2))$ where, as indicated, the amplitude and functional dependence was about the same as for the transient creep. One of the relaxation times was observed to fit the expression $\tau_0 \exp(-E_1/kT)$ where E_1 was about 30-40 Kcal/Mole. It is suggested that this is related to relaxation of stress by grain boundary sliding.

ASD TR 61-628

April 1962

SUBJECT: STUDIES OF THE BRITTLE BEHAVIOR OF CERAMIC MATERIALS
INVESTIGATOR: N. A. Weil
CONTRACT: AF33(616)-7465, Armour Research Foundation
ABSTRACT: Extensive research on factors influencing the behavior of brittle non-metallic ceramics was undertaken. General information on the forms of Al₂O₃ and MgO studies and preliminary work on the effect of porosity on the fracture strengths of Lucalox (Al₂O₃) are presented. Individual problem areas were attacked on separate task programs.

- Task 1 - Effect of Structural Size: "The Zero Strength"
- Task 2 - Effects of Strain Rate
- Task 3 - Effect of Non-Uniform Stress Fields'
- Task 4 - Effect of Microstructure
- Task 5 - Internal Friction Measurements
- Task 6 - Surface Energy on Brittle Behavior

ASD TR 61-628 (Continued)

- Task 7 - Fracture Mechanisms
- Task 8 - Impurity Influences
- Task 9 - Static Fatigue: Delayed Fracture
- Task 10- Effect of Thermal-Mechanical History
- Task 11- Surface Active Environments

ASD TDR 62-203, Part 1

May 1962

SUBJECT: KINETICS OF OXIDATION IN THE Mo-Si SYSTEM
INVESTIGATOR: J. B. Berkowitz
CONTRACT: AF33(616)-6154, Arthur D. Little
ABSTRACT: The oxidation behavior of Mo, Mo₃Si, Mo₅Si₃, and MoSi₂ has been studied at temperatures between 1000° and 1725°C at oxygen partial pressures less than 20 mm. Pure molybdenum metal oxidizers linearly in this range, with a ratio constant

$$k = k_0 e^{-22,000/RT_p^{0.87}},$$

where p is oxygen partial pressure, the gas constant R is in cal/mole, T is °K and k_0 is a constant independent of temperature and pressure. To a good approximation, oxidation of the molybdenum silicides follows a logarithmic rate, $Q = Q_0(1 - e^{-at})$, where Q is the total oxygen consumed between time zero and time t, while Q_0 and a are constants, dependent upon composition, pressure, and temperature. In general, for a given silicide composition, Q_0 decreases and a increases with increasing temperature 1300° and 2000°K, at a constant oxygen pressure. At any fixed temperature, the most oxidation resistant material is MoSi₂; Mo₅Si₃ is oxidized to a larger extent before a protective coating is established, and Mo₃Si is oxidized still more extensively. It is significant, however, that the rates of oxidation of all three compounds eventually reaches a low-limiting value. Metallographic examination of the oxidized samples has established that oxidation proceeds at least in part by diffusion of oxygen inward.

ASD TDR 62-203, Part II

March 1963

SUBJECT: KINETICS OF OXIDATION OF REFRACTORY METALS AND ALLOYS AT 1000°-2000°C
INVESTIGATOR: J. B. Berkowitz-Mattuck
CONTRACT: AF33(616)-6154, Arthur D. Little, Inc.
ABSTRACT: I. Oxidation of Copper: A thermal conductivity apparatus for continuous measurement of oxidation of metallic materials between 900 and 2100°C is described. Results on the oxidation of

ASD TDR 62-203, Part II (Continued)

copper between 975 and 1044°C at oxygen partial pressures of 2-10 Torr are in agreement with literature values obtained by conventional methods.

II. Oxidation of Carbides: Experimental data is discussed in the light of thermodynamic considerations for the carbides of Groups IV-A, V-A, and VI-A metals.

III. Oxidation of Molybdenum Silicides: The oxidation of Mo_3Si , Mo_5Si_3 , and MoSi_2 between 1300 and 2100°K at oxygen partial pressures of 2-20 Torr was studied by oxygen consumption and metallographic techniques.

IV. Oxidation of Miscellaneous Materials: The oxidation of W_5Si_3 and WSi_2 was studied by the thermal conductivity method at temperatures between 1600 and 2030°K. A measurement of the rate of oxidation of $\text{Ta}_2\text{Be}_{17}$ was made at 1664°K and 8.4 Torr.

ASD TDR 62-204, Part I

April 1962

SUBJECT: THERMODYNAMIC AND KINETIC STUDIES FOR A
REFRACTORY MATERIALS PROGRAM
INVESTIGATOR: L. A. McClaine
CONTRACT: AF33(616)-7472, Arthur D. Little, Inc.
ABSTRACT: This report summarizes an integrated program of studies developed to provide thermodynamic and kinetic knowledge needed to describe the chemical behavior of zirconium and hafnium carbides and borides at temperatures to 3000°K and in atmospheres of O_2 , $\text{O}_2 + \text{H}_2\text{O}$, Cl_2 , HF , F_2 , H_2 , CO , NH_3 , and N_2 .

The program consists of both internal and sub-contracted efforts which can (1) provide, through calorimetric measurements, additional thermodynamic data needed for the tabulation of free energy functions of the zirconium and hafnium carbides and borides; (2) identify, by mass spectrometric means, gaseous species formed on vaporization of these carbides and borides and on their interaction with the several atmospheres; (3) provide thermodynamic data for important gaseous species through electron diffraction techniques, "matrix isolation" spectroscopy, and equilibria studies; and (4) develop the kinetics of the reactions of the carbides and borides with the various atmospheres through studies of the over-all rates of reactions and mass transport mechanisms. The status of each investigation is discussed in detail. The preparation and characterization of macrocrystalline, high purity ZrB_2 and ZrC are described.

ASD TDR 62-204, Part I (Continued)

Low temperature heat capacity data for ZrB_2 and heat content data for ZrB_2 , ZrC , and HfB_2 are presented. The results of a study of the vaporization of ZrB_2 are reported.

ASD TDR 62-225

December 1962

SUBJECT: THE ROLE OF THE GRAIN BOUNDARY IN THE DEFORMATION OF CERAMIC MATERIALS
INVESTIGATOR: G. T. Murray, J. Silgailis, A. J. Mountvala
CONTRACT: AF33(616)-7961, Materials Research Corp.
ABSTRACT: The grain boundaries of MgO bicrystal specimens were subjected to creep-rupture tests in the temperature range of 1300-1500°C and at shear stresses varying from 150 to 10,000 gm/mm^2 . The boundary was oriented at 45° to the compression direction. The stress for grain boundary sliding and fracture varied markedly with crystal misorientation; high twist-low tilt boundaries being much weaker than other mis-orientations. For a given misorientation, there was considerable scatter in the fracture and sliding stress data which was found to be a result of boundary irregularities (jog content) present in the as-received material or stress-induced during test. Most specimens did not exhibit controlled grain boundary sliding but rather, would slide uncontrollably to fracture after an incubation period of a few minutes at a critical stress.

ASD TDR 62-272

May 1962

SUBJECT: GROWTH AND ANALYSIS OF ALUMINA "WHISKERS"
INVESTIGATOR: F. W. Vahldiek, C. T. Lynch, L. B. Robinson
ABSTRACT: Alumina whiskers were grown at 1700° to 1800°C in argon and helium atmospheres containing small amounts of hydrogen. Whiskers thus produced were analyzed by X-ray diffraction, by spectrographic emission, and by wet chemical methods. A scheme was developed for establishing impurity levels in the whiskers.

Conditions conducive to growth were investigated, as well as characteristics of whisker growths. Some whiskers which possess unusually complex dendritic growth patterns were examined in considerable detail, and evidence was found of several distinct types of growth forms.

A simple tube furnace apparatus for whisker growth is described.

ASD TDR 62-476

June 1962

SUBJECT: INVESTIGATION OF THE EFFECTS OF PROCESSING VARIABLES AND FABRICATION TECHNIQUES UPON THE PROPERTIES OF INTERMETALLIC COMPOUNDS

INVESTIGATOR: R. S. Truesdale, et al

CONTRACT: AF33(616)-7108, Brush Beryllium Co.

ABSTRACT: Intermetallic compounds of NbBe₁₂, Nb₂Be₁₇, Nb₂Be₁₉, and Ta₂Be₁₇ were fabricated by uniaxial pressing and sintering techniques. Results of sintering studies on Nb₂Be₁₇ are presented and discussed. The effects of particle size, stoichiometry, temperature, and time upon the sintered density, grain size, and strength of Nb₂Be₁₇ are described. A modulus of rupture evaluation is made on Nb₂Be₁₇ up to 2750°F, and results of tensile, Young's modulus, thermal conductivity, and oxidation studies are discussed. The feasibility of scaling-up to large and complicated shapes by isostatic pressing, slip casting, extrusion, hot pressing, and flame spraying is demonstrated. The temperature-pressure flow characteristics of several beryllides at temperatures of 2750° to 3050°F are evaluated together with micro-structural stability. The metallurgical fabrication techniques of forging, forming, and rolling as applied to Nb₂Be₁₇ are examined.

ASD TDR 62-700

October 1962

SUBJECT: INVESTIGATION OF SINTERABLE MgO Powders and CERAMICS MADE FROM THEM

INVESTIGATOR: M. J. Snyder, et al

CONTRACT: AF33(616)-7733, Battelle Memorial Institute

ABSTRACT: The fabrication of dense reproducible specimens suitable for strength measurements and having controlled micro-structural differences was attempted. A sinterable high-purity magnesia powder, developed in an earlier phase of the program, was used. Procedures used earlier to produce small, rather crude specimens, could not be applied directly to controlled production of the quantity of large specimens needed. The sintered densities of the larger specimens were lower, and the variability from one lot to the next was greater. The major source of variability appeared to reside in the isostatic pressing operation. This unexpected sensitivity to forming conditions other than pressure suggests the need for further research on processing effects in ceramics made from sinterable powders. A portion of the effort was devoted to analysis and development of the diametral-compression test wherein short cylinders are broken by applying a load across a diameter. Procedures were established for measuring a tensile strength by this method.

ASD TDR 62-732

April 1963

SUBJECT: A FUNDAMENTAL STUDY OF THE VARIABLES ASSOCIATED WITH THE MIXING OF CERAMIC RAW MATERIALS
INVESTIGATOR: D. W. Fuerstenau, et al
CONTRACT: AF33(616)-7763, University of California
ABSTRACT: This summary report presents the progress completed on the investigation of the mixing and sintering characteristics of Al_2O_3 and MgO particles. Separate inter-related areas of research have been investigated which include particle preparation, dry and wet mixing, and the sintering behavior of Al_2O_3 and MgO.

The techniques of particle reduction and analysis of particle size distribution have been investigated by determining the variables which affect the resulting particles. Emphasis has been placed on control conditions for a jet pulverizer.

Variables encountered in the dry mixing operation have been found to be the mixer design, loading methods, and sampling methods. Analysis of these variables has been made by determining the standard deviation from a statistical number of samples. Surface properties of Al_2O_3 and MgO in aqueous media have been investigated in order to understand wet mixing behavior. Determinations of the nature of the electrical double layer of the particles have been made.

The basic mechanisms and the characteristics of the reaction between MgO and Al_2O_3 have been studied with single crystals. The understanding of the fundamentals of the reaction has enabled the establishment of optimum sintering conditions. Some of the properties of sintered compacts due to varied sintering conditions have been determined.

ASD TDR 62-843

March 1963

SUBJECT: AN INVESTIGATION OF PHYSICAL PARAMETERS OF NON-ELECTRICALLY CONDUCTING FINE PARTICULATES
INVESTIGATOR: D. W. Koester, E. B. Cornelius, J. J. Donovan
CONTRACT: AF33(616)-8032, Houdry Process and Chemical Co.
ABSTRACT: The objective of this research is to find one or more measurable properties of active ceramic powders which can be related to sinterability. Samples of active magnesia were prepared by calcination of basic carbonate at 400° - $900^{\circ}C$ and tested for activity in a closed system. The activity measurements, oxygen exchange and "initial specific adsorption" (of CO_2), indicate that the bulk structure as well as the surface of such materials is extremely active. There are indications that these measurements can

ASD TDR 62-843 (Continued)

be used to characterize ceramic materials with respect to their sinterability.

Data from decomposition experiments and CO₂ adsorption studies are present and discussed. These results emphasize the difficulties involved in preparing uniform, large-scale lots of activated powders, and the care required in their handling to avoid inadvertent contamination by adsorption of atmospheric constituents (H₂O and CO₂).

ASD TDR 62-1029

March 1963

SUBJECT: FRACTURE PROCESSES IN GERMANIUM
INVESTIGATOR: O. W. Johnson, N. Farb, P. Gibbs
CONTRACT: AF33(616)-6832, University of Utah
ABSTRACT: The fracture strength, σ_F , of germanium, at constant loading rate has been determined over a temperature range from -196°C to 500°C. σ_F decreases slowly up to about 200°C, then increases to about 400°C, followed by subsequent decrease. σ_F is found to be essentially independent of ambient for a wide variety of chemical species. These observations are analyzed in terms of fracture nucleation at a flaw near, but not exposed to, the surface, by a presently unknown kinetic process. The role of dislocations apparently is to inhibit fracture by relaxation of stress concentrations up to about 400°, above which general plastic deformation precedes fracture, suggesting the possibility of fracture nucleation by dislocation pileups. The applicability of this analysis to another brittle system, Al₂O₃, is considered in the light of its reported fracture, deformation, wear and friction characteristics. Striking similarities are found in the behavior of these two crystals, suggesting that the underlying mechanisms must have much in common.

The effect of atmosphere on plastic deformation of both single crystal and polycrystalline Al₂O₃ was examined. Heat treatment in dry O₂ and N₂ at temperatures between 1350° and 1580°C for periods up to 50 hours, produced no significant changes in creep behavior.

ASD TDR 62-1086

February 1963

SUBJECT: RESEARCH ON GROWTH AND DEFORMATION MECHANISMS
IN SINGLE CRYSTAL SPINEL
INVESTIGATOR: H. Palmour, et al
CONTRACT: AF33(616)-7820, North Carolina State College

ASD TDR 62-1086 (Continued)

ABSTRACT: The first phases of a continuing program of research on growth and deformation processes in magnesium aluminate spinel are described. Spinel forming reactions, structure and properties of spinel, synthesis of spinel feed materials (including a study of the effects of irradiation on spinel formation), techniques and apparatus for crystal growth with Verneuil oxyhydrogen and R F plasma devices, and the effect of atmosphere on precipitation hardening of alumina-rich spinel have been studied. The report includes a summary of structural and physical properties of $MgAl_2O_4$ spinel.

ASD TDR 63-138

May 1963

SUBJECT: FIELD ION MICROSCOPY OF IRON WHISKERS
INVESTIGATOR: E. Muller, O. Nishikawa
CONTRACT: AF33(616)-6397, Pennsylvania State University
ABSTRACT: The field ion microscope images of iron whiskers from various origins were observed and differentiations between iron whiskers and specimens made of pure iron wire were studied. Indications of crystal defects such as screw dislocations and axial disordered core structures were found. The influence of various gases on the iron surface under the extremely high electric field and the diffusion of potassium ions into the iron lattice was observed. The operation of the field ion microscope with neon gas gives certain advantages for the study of easily field evaporating metals such as iron.

ASD TDR 63-147

March 1963

SUBJECT: FUNDAMENTAL STUDIES OF COMPRESSIBILITY OF POWDERS
INVESTIGATOR: I. Shapiro
CONTRACT: AF33(616)-8006, Hughes Tool Co.
ABSTRACT: The compressibility of a number of powders of different physical characteristics was measured at ambient temperature. The progress of the compaction behavior of the powders was followed by photomicroscopy--this technique permitted observing changes occurring during compression.

The difference between plastic deformation of metals, such as magnesium, and the fragmentation of ceramics, such as thoria, could be clearly demonstrated. The compressibility of molybdenum disulfide, a material commonly used as a lubricant, indicated a high degree of "plastic" quality as well as some fragmentation. Mixtures of molybdenum disulfide with ceramics resulted in both types of compaction.

ASD TDR 63-147 (Continued)

The applicability of several formulas relating porosity or density of compacts with pressure is discussed.

The results of the present study are regarded as most significant towards clarifying erroneous hypotheses as to what forces are operating during compaction of powders. The concept of particles sliding past one another is contrary to experimental findings. The concept of mechanical interlocking of particles as an explanation of strength of compacts does not appear to be valid.

ASD TDR 63-215

March 1963

SUBJECT: MONTE CARLO COMPUTATIONAL EXPERIMENTS ON SINGLE VACANCY MIGRATION IN CUBIC LATTICES
INVESTIGATOR: H. Gegel
CONTRACT: AF33(657)-8473, General Electric Co.
ABSTRACT: A theoretical study of single vacancy migration in cubic AB alloys; i.e., simple cubic, body centered cubic, and face centered cubic (s.c., b.c.c. and f.c.c.), was performed using the Flinn-McManus Monte Carlo method for determining the direction of each vacancy jump. Migration histories of 10^4 to 3×10^4 jumps were considered. The computations dealt with the spatial properties of the vacancy migration process, per se, and the changes this migration induced in the order and configurational energy of the alloy. Physically, these computations correspond to watching a vacancy move through an alloy, originally in a state of equilibrium long range order S_1 at a temperature T_1 after a sudden change to a constant temperature T . Both the ordering and disordering processes were treated.

ASD TDR 63-276

April 1963

SUBJECT: Q VALUES FOR INDEXING X-RAY POWDER PATTERNS ACCORDING TO ITO
INVESTIGATOR: R. L. Prickett
ABSTRACT: A comprehensive table of Q values for all common interplanar spacings was set up to assist in indexing x-ray powder patterns by the Ito method. An IBM 7090 computer was used to calculate the data.

April 1963

SUBJECT: PEST REACTIONS IN INTERMETALLIC COMPOUNDS:
Grain Boundary Hardening in NiGa
INVESTIGATOR: A. U. Seybolt, J. H. Westbrook
CONTRACT: AF33(657)-7980, General Electric Co.
ABSTRACT: The phenomenon of grain boundary hardening has been explored for the CsCl structural intermetallic compound NiGa. NiAg has a homogeneity range of a few per cent and it was possible to examine the effect of stoichiometry upon the grain boundary hardening due to preferential oxygen diffusion down grain boundaries. While some grain hardening was noticeable just below 50A/o Ga, the effect was much less pronounced than at 52A/o Ga.

It was possible to estimate both bulk diffusion and grain boundary diffusion rates for oxygen.

The results suggest that hardening is due to lattice distortions which arise from the formation of a Ga-O complex.

COATINGS

WADD TR 60-758, Part III

April 1962

SUBJECT: PHOTODEGRADATION OF HIGH POLYMERS. Photolysis of Poly(Methyl Methacrylate) in Vacuum and in Air

INVESTIGATOR: R. B. Fox, L. G. Isaacs, S. Stokes, et al

CONTRACT: AF33(616)-61-10, U. S. Naval Research Laboratory

ABSTRACT: The photodegradation of poly(methyl methacrylate) films in vacuum and in air by ultraviolet radiation from a medium-pressure mercury lamp has been investigated at room temperature. Changes in molecular weight in terms of intrinsic viscosities, the formation of volatile substances, and the increase in the ultraviolet absorption of the films were followed as a function of energy absorbed. Quantum yields for random chain scission in poly(methyl methacrylate) are about 0.04 and 0.02 in vacuum and in air, respectively. Cross-linking was not observed in air. Quantum yields in vacuum are the same at 2 and 0.02 pressure. The quantum yield for methyl formate is approximately that for chain scission; the formate is accompanied by larger amounts of methanol and very small quantities of monomer. Increased ultraviolet absorption occurs after extensive degradation in either air or vacuum and is due to chromophores formed in the polymer rather than in low molecular weight fragments. These observations are rationalized in a mechanism for the photolysis of this polymer.

ASD TR 61-151, Part II

July 1962

SUBJECT: REFLECTIVE COATINGS ON POLYMERIC SUBSTRATES

INVESTIGATOR: R. B. Belser, M. D. Carithers

CONTRACT: AF33(616)-6980, Georgia Inst. of Technology

ABSTRACT: Methods of overcoating metal surfaces with polymer coatings to provide a surface of high gloss plus reflective metal films for controlling the optical properties of a metal surface, were examined. Polymers of the species epoxy, polyurethane, or silicone were applied to stainless steel substrates and were overcoated with metal films of gold, silver, aluminum, copper, nickel, or combinations by chemical reduction or vacuum deposition techniques. The polymers and the metal films were examined for adherence, survival from 25 to 200°C, specular reflectance and emittance at various temperatures. Reflectance values of metallic films evaporated or chemically deposited on polymeric substrates were generally equivalent and were equal to that obtained for similar metallic films on glass substrates.

ASD TR 61-558

July 1962

SUBJECT: SOLAR ABSORPTANCE AND TOTAL HEMISPHERICAL
EMITTANCE OF SURFACES FOR SOLAR ENERGY
COLLECTION

CONTRACT: MIPR33(616)-60-3 & 61-29, U. S. Naval Radio-
logical Defense Laboratory

ABSTRACT: The solar absorptance and the total hemispherical
emittance are reported between 200°C and 800°C for 6 polished metals,
8 metals with porous surfaces, and 15 coated metals. The surfaces
evaluated were selected because their optical properties suggested
that they might be applicable for solar power collection. The method
of measurement is described wherein the specimen, approximately 0.75
inches in diameter and 0.040 inches thick, is suspended by thermo-
couple wires in a black walled evacuated chamber. The solar absorp-
tance is determined from the rate of temperature rise when the speci-
men is exposed to the radiation from a cored carbon arc whose spectral
distribution simulates that of sunlight. The total hemispherical
emittance is determined from the rate of temperature decay when the
arc radiation is removed. The effect of microscopic roughness of
the surface on the optical properties is investigated by spectro-
metric measurements.

An expression for the efficiency of a solar
energy collection surface in terms of its solar absorptance, total
hemispherical emittance, solar power concentration (mirror or lens),
the solar irradiance, and the operating temperature is developed.
The efficiencies of all surfaces treated are reported as a function
of temperature.

ASD TDR 62-92, Part 1

June 1962

SUBJECT: INORGANIC FILMS FOR SOLAR ENERGY ABSORPTION

INVESTIGATOR: R. C. Langley

CONTRACT: AF33(616)-7849, Englehard Industries, Inc.

ABSTRACT: A number of thin films (about 1250 Å) was
obtained on glass by thermal decomposition of metallo-organic so-
lutions. This proved to be a practical method for a broad search
for a film which absorbs solar energy of wavelengths below 1.5
microns while reflecting well above 1.5 microns.

A film containing gold, rhodium, and oxides
of bismuth, barium, silicon, and chromium has the properties sought
and is potentially useful for space purposes. This film can be
obtained by a single application and firing of an organic solution of
these metals.

ASD TDR 62-92, Part 1 (Continued)

The best film found has a purity of only 89.5% Au, yet reflects as well as pure Al above 1.5 microns. In this six component film, it was found that additions of barium or silicon in small amounts or of platinum or palladium in substantial amounts cause a decreased reflection.

ASD TR 62-443

May 1962

SUBJECT: RESEARCH FOR LOW AND HIGH EMITTANCE COATINGS
INVESTIGATOR: E. A. Schatz, L. C. McCandless
CONTRACT: AF33(616)-7276, American Machine & Foundry Co.
ABSTRACT: Eight transparent protective coatings for gold were evaluated with respect to their ability to withstand temperatures of 1000°C and not to significantly increase the low total emittance of the substrate. Thin coatings, of the order of m, of vacuum evaporated SiO, Al₂O₃, and WO₃ were found to be highly satisfactory. Samples with a protective topcoat over gold which in turn was applied over a diffusion barrier coating of CeO₂, were able to maintain a total normal emittance of less than 0.1 for up to twenty hours at 1000°C in air.

A second aspect of the work was the study of the spectral normal emittance in the 1-15 μ wave-length range at 1000°C of sintered binary mixtures of pure compounds to correlate the spectral emittance of a mixture with the crystal structure of the sample and the spectral emittance of the pure components. Forty-seven sintered samples were measured including various mixtures of Y₂O₃-Cr₂O₃, Al₂O₃-Cr₂O₃, MoSi₂-Cr₂O₃, Al₂O₃-SiO₂ and Al₂O₃-NiO. No significant correlation was found between the crystal structure and the spectral emittance.

WADD TR 62-703, Part IV

July 1962

SUBJECT: THE ULTRAVIOLET DEGRADATION OF ORGANIC COATINGS:
Radiation in a Vacuum
INVESTIGATOR: D. E. Field, J. E. Cowling, F. M. Noonan
CONTRACT: MIPR33(616)-61-10, U.S. Naval Research Lab
ABSTRACT: Organic coatings are being investigated as one possible means of achieving temperature control within space vehicles. However, their usefulness in the space environment may be limited by their physical and chemical stability in space. It is shown that the optical properties of most organic coatings are changed on exposure to intense ultraviolet radiation in high vacuum. Of the reflective pigments evaluated, those containing zinc sulfide and leafing aluminum are shown to be most stable to this radiation.

WADC TR 53-373 Sup 10

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January 1963

SUBJECT: PIGMENTED SURFACE COATINGS FOR USE IN THE SPACE ENVIRONMENT

INVESTIGATOR: N. Z. Searle, R. C. Hirt, R. G. Schmitt

CONTRACT: AF33(616)-7958, American Cyanamid Co.

ABSTRACT: Photochemical stabilities of pigmented surface coatings to ultraviolet radiation and vacuum conditions simulating those present in the upper atmosphere were determined for various systems. The amount of photodegradation incurred was determined from the change in solar absorptivity based on reflectance measurements over the range 0.25 to 2.6 microns. Melamine modified polyvinyl butyral and silicone alkyd resins are approximately equal in stability and more stable than polyurethane or CYANOCEL resins. Zinc sulfide and zinc oxide pigmented melamine modified polyvinyl butyral films are more stable than the rutile titanium dioxide pigmented films. Lead titanate, lead carbonate and antimony oxide films are least stable. Increase in titanium dioxide concentration from 30% to 60% by volume has a marked effect in increasing the stability. Hydroxybenzoyl ferrocene stabilizes the pigmented coatings by masking the solarization and imparting a high initial solar absorptivity. Methyl salicylate gives some protection while dibenzoyl resorcinol decreases stability. Samples should be maintained under vacuum during spectrophotometric measurements as well as irradiation to prevent partial reversal of solarization. Intercomparison of photochemical stabilities on the basis of irradiation required for a particular increase in solar absorptivity is a far more sensitive criterion than that based on increase in solar absorptivity after some period of irradiation.

February 1963

SUBJECT: BIBLIOGRAPHY OF VACUUM ULTRAVIOLET SPECTROSCOPY

INVESTIGATOR: R. C. Hirt, R. G. Schmitt

CONTRACT: AF33(616)-7958, American Cyanamid Co.

ABSTRACT: Publications in the field of vacuum ultraviolet spectroscopy have greatly increased in number during recent years, stimulated by the interest in space exploration and aided by the advent of commercial instrumentation. It appeared desirable to compile an extensive bibliography at this time. The bibliography is arranged alphabetically by senior author, with a four-letter code (called CODEN) for the journal reference. The format and presentation are suited for easy punching onto IBM punched cards for machine sorting. A subject index is provided to the more than 1300 references.

ASD TDR 62-917

February 1963

SUBJECT: IMPROVED ORGANIC COATINGS FOR TEMPERATURE CONTROL
IN A SPACE ENVIRONMENT

INVESTIGATOR: H. H. Hormann

ABSTRACT: A wide variety of tailored coatings were prepared to meet the demands for selective solar absorptance values for short term exposures to the space environment. A white silicone-alkyd coating gave 0.20-0.85 values when various amounts of a black enamel were added. Only small changes in optical properties were noted when the coatings were exposed to vacuum-thermal or ultraviolet radiation environments. The high emittance (0.90-0.94) of a black enamel was lowered considerably (0.76-0.79) by adding leafing aluminum pigment.

Stabilization of certain organic coatings to the effects of ultraviolet radiation was accomplished by incorporating novel ultraviolet absorbers into the coating system. Additions of protective absorbers, particularly hydroxybenzoylferrocene, into a white silicone alkyd and acrylic have enabled the formulations to withstand the effects of ultraviolet radiation to a higher degree than the basic formulations themselves.

ASD TDR 62-918

February 1963

SUBJECT: ANODIZED ALUMINUM COATINGS FOR TEMPERATURE
CONTROL OF SPACE VEHICLES

INVESTIGATOR: J. H. Weaver

ABSTRACT: The first attempt to accurately relate the factors associated with the preparation of anodized aluminum coatings to their thermal control properties for aerospace use is presented. An understanding of the expected performance of these coatings has been obtained and conditions for maximizing properties have been elucidated. A coating useful for maintaining a high reflectance of solar energy with high emittance has been prepared.

The effects of the simulated environment on the optical properties of anodic coatings have been determined. Preliminary data indicate that the vacuum-ultraviolet effects are the most detrimental to the optical properties of the anodized aluminum coating. The selection of this coating on space vehicles should be confined to interior use only. The coating is scheduled to be evaluated in an actual space environment to confirm the properties demonstrated in the laboratory.

April 1963

SUBJECT: PROPERTIES REQUIRED FOR COLLECTOR SURFACES OF
SOLAR-POWERED THERMAL SYSTEMS

INVESTIGATOR: R. A. Cross

ABSTRACT: This comprehensive analysis of specific properties required for collector surfaces of solar-powered thermal systems useful as auxiliary power in space vehicles uses FORTRAN programming for the IBM 7090 with various in-put parameters to determine operating characteristics of a Carnot engine under various conditions of concentration factor and radiator temperature. Collector surfaces considered include a blackbody surface, an ideal coating (having an absorptance of 1.0 at wavelengths shorter than some cut-off wavelength and an emittance of 0.0 at longer wavelengths), and a feasible selective coating (having an absorptance of 0.95 at short wavelengths, an emittance of 0.05 at long wavelengths, and a 1.0 micron transition region). Cut-off wavelengths were determined for maximum efficiency. At concentration factors up to approximately 500 the feasible selective coating proved advantageous over the blackbody absorber. Specific research approaches to prepare improved coatings with properties approaching the ideal are recommended.

COMPOSITES

WADC TR 55-319, Sup 3

December 1962

SUBJECT: WEATHERING OF GLASS-FABRIC-BASE PLASTIC LAMINATES
INVESTIGATOR: F. Warren
CONTRACT: AF33(616)-58-1 & 61-06, U.S. Forest Products Lab
ABSTRACT: Eleven different glass-fabric-base laminates were exposed to various environmental conditions for periods up to 3 years. One of the exposures was at jungle conditions at Panama, and the 3-year panels were stolen from the site before exposure was completed.

Nine replacement panels were then sent to Panama, and the 3-year exposure was subsequently completed. This report presents results of flexural tests of these replacement panels, and supplements the data of two earlier reports.

Results of the tests show that exposure at the jungle conditions had less effect on strength, erosion of surface resin, and hardness than outdoor exposure at the other four test sites.

WADC TR 55-319, Sup 4

October 1962

SUBJECT: EFFECTS OF WEATHERING ON THE MECHANICAL PROPERTIES OF FOUR REINFORCED PLASTICS LAMINATES
INVESTIGATOR: K. E. Kimball
CONTRACT: AF33(616)-58-1 and 61-06, Forest Products Lab
ABSTRACT: Four different reinforced plastic laminates were exposed to various environmental conditions for periods up to 36 months. After completion of the exposure, the laminated panels were evaluated under normal, wet, and elevated temperature conditions. Data on the effect of 3, 12, 36 months' exposure on the flexural properties of laminates and their appearance after 36 months are presented.

WADD TR 60-299, Part II

June 1962

SUBJECT: SYNTHESIS OF FIBER REINFORCED INORGANIC LAMINATES
INVESTIGATOR: E. M. Clausen, et al
CONTRACT: AF33(616)-6283, University of Illinois
ABSTRACT: The use of inorganic materials as the matrix for reinforced composites is studied. The objectives of this research

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were to study the compositional and processing variables on matrix strength, the explanation of the observed deformation of matrix bodies, and research on protective fiber coatings.

Type of bonding acid used, pre-milling time of raw materials, reacting temperature, and milling time of reacted materials were considered variables affecting strength.

Reactions occurring during drying, firing, and the mechanism of deformation were studied.

Tin oxide, vapor deposited antimony oxide, molybdenum trioxide, organic-inorganic oxides, and liquid silver were considered as coatings for glass fibers.

Strength and corrosive effects of matrices on Al_2O_3 , TiO_2 , and ZrO_2 rods were examined.

WADD TR 60-791, Part III

June 1962

SUBJECT: HIGH TEMPERATURE RESINS, ANALYSIS OF PROCESS PARAMETERS AND EVALUATION PROCEDURES FOR FILAMENT WOUND COMPOSITES. Evaluation Procedures

INVESTIGATOR: H. S. Daley, T. Siuta, S. Yurenka

CONTRACT: AF33(616)-6737, Narmco Industries, Inc.

ABSTRACT: The objective of the program was to determine the most significant basic mechanical properties of filament wound composites, and to provide optimum standardized test methods and procedures for defining them at both room and elevated temperatures. The evolution and development of the various test configurations are described and construction details for each of the evaluation procedures are presented by drawings. Winding machine operating and maintenance instructions are presented in as much detail as possible. An account of the underlying theories and principles governing the design and use of this machine is given to facilitate understanding of the operating instructions and the capabilities of the machine.

ASD TR 61-218

May 1962

SUBJECT: EXOTHERMIC BONDING OF METAL SANDWICH CONSTRUCTION
BY THE "EXO-FLUX" TECHNIQUE

INVESTIGATOR: W. Bassett, R. Caughey, R. A. Long

CONTRACT: AF33(616)-7115, Narmco Research & Development

ABSTRACT: The objective of this program was to adapt the "exo-flux" overlap bonding process to metal sandwich panel bonding. The process provides a continuous metal bond between the core feet and the facing sheets, and consists of painting the metal surfaces to be bonded with approximately .01 gm./sq. in. of inorganic fluxing agent, placing exo-thermically reactive tape against the opposing external faces, subjecting the core-facing sheet contact area to 100 psi pressure and igniting the tape. The resulting bond exhibits no discernible inter-facial discontinuities at a magnification of 1000 diameters. After the most suitable materials were selected (i.e. PH 15-7 stainless steel base metal, B₂O₃ + cryolite flux, and a tape exotherm) and procedures determined, successful 3" x 4" sandwich panel bonding was achieved, although reproducibility was poor. Bonding of 3" x 8" sandwich panels was marginal, with very poor reproducibility and no panels with 100% of area bonded. The maximum strengths obtained were 184,000 psi in edgewise compression at room temperature, 85,000 psi in edgewise compression at 1000°F.

ASD TR 61-338

April 1962

SUBJECT: FATIGUE TESTING OF HONEYCOMB SANDWICH CON-
STRUCTIONS

INVESTIGATOR: H. Spector

CONTRACT: AF33(616)-7200, Northrop Corp.

ABSTRACT: Dynamic methods, developed by Northrop Corp., Norair Division for determination of Young's modulus for honeycomb sandwich constructions, were successfully applied to obtain honeycomb core fatigue properties. S-N curves for extensional core fatigue were developed for representative samples of brazed, welded, and adhesive bonded honeycomb constructions. For tension-compression, the data shows that core density is a dominant parameter in core fatigue. There are indications that core thickness up to and including 1-inch has little effect upon the data; more investigation is necessary to determine the effect of thickness greater than 1-inch. Feasibility of obtaining core fatigue using a two specimen shear modulus approach was demonstrated, but more development is necessary in the design of testing systems to obtain dynamic balance and proper load levels.

ASD TR 62-202

December 1961

SUBJECT: HIGH TEMPERATURE INORGANIC STRUCTURAL COMPOSITE MATERIALS

INVESTIGATOR: J. H. Lauchner, W. B. Hall, J. M. Fields, Jr.

CONTRACT: AF33(616)-7765, Mississippi State University

ABSTRACT: An aluminum phosphate matrix was developed which has the required properties of low modulus of elasticity and inelastic deformation. Dynamic modulus of elasticity, modulus of rupture, maximum deflection, and firing shrinkage were determined for the matrix. Physical properties of the matrix varied between specimens. Differential thermal analysis and x-ray studies indicated that different phases of aluminum phosphate (analogous to silica phases) may be the cause of matrix inelastic deformation characteristics. The matrix materials developed were found to be corrosive to reinforcement fibers. Fiber protection was studied and a series of coated specimens evaluated. Several coatings indicated potential protection capabilities.

ASD TDR 62-352

SUBJECT: CARBONIZED PLASTIC COMPOSITES FOR HYPERTHERMAL ENVIRONMENTS

INVESTIGATOR: R. K. Carlson, B. A. Forcht, J. A. Medford

CONTRACT: AF33(616)-7947, Vought Astronautics

ABSTRACT: A new class of thermally protective materials were synthesized, which have outstanding resistance to dimensional erosion in simulated re-entry environments. The process of making the materials consisted of controlled pyrolysis of precursory reinforced plastics to form a porous carbonized matrix, which was subsequently impregnated with either an organic or inorganic ablative gas-forming filler. Material and fabrication variables for preparation of improved impregnated matrices were investigated and composites having controlled properties were obtained. The ablative behavior of the composites was determined by exposure in an air plasma arc.

ASD TDR 62-396

April 1963

SUBJECT: CONFERENCE ON STRUCTURAL PLASTICS, ADHESIVES, AND FILAMENT WOUND COMPOSITES - 11-13 DEC 1962

INVESTIGATOR: S. Litvak

ABSTRACT: A three day conference on Structural Plastics, Adhesives, and Filament Wound Composites was held at the Biltmore Hotel in Dayton, Ohio, on December 11-13, 1962. This conference was sponsored by the Plastics and Composites Branch, Nonmetallic Materials Laboratory. The purpose of the conference was to present recent

ASD TDR 62-396 (Continued)

progress by the Air Force and other Government agencies on plastic materials and to review the current state-of-the-art and associated problem areas.

ASD TDR 62-406

September 1962

SUBJECT: STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES AT ELEVATED TEMPERATURES (Silicone-Asbestos, R/M Pyrotex Felt 45-RPD)

INVESTIGATOR: K. H. Boller

CONTRACT: D033(616)-61-06, Forest Products Laboratory

ABSTRACT: This report on a reinforced plastic laminate (silicone-asbestos laminate made of R/M Pyrotex Felt Style 45-RPD) is the sixth of several reports that present strength and elastic properties at elevated temperatures. Properties of this silicone-asbestos laminate were evaluated in flexure, tension, compression, inter-laminar shear, and bearing parallel to the machine direction of the felt. In general, the strength retention with increasing severity of exposure is high. Strength drops with the first absorption of heat, but at constant temperatures of 300° to 700°F., the exposed material hardens and has a tendency to either maintain constant strength or even increase in strength. Continued increasing temperatures above 700°F cause decreases in strength but never a complete loss within the limits evaluated. From the curves and data presented here, the effects of temperatures between 80° and 1000°F and exposure periods between 0.05 hour and 1000 hours on individual strength properties may be judged and interpreted separately.

ASD TDR 62-464

September 1962

SUBJECT: FATIGUE PROPERTIES OF PLASTIC LAMINATES REINFORCED WITH UNWOVEN GLASS FIBERS

INVESTIGATOR: K. H. Boller

CONTRACT: D033(616)-61-06, Forest Products Laboratory

ABSTRACT: Fatigue characteristics are presented for epoxy-type plastic laminates at various resin contents and reinforced with layers of unidirectional unwoven glass fibers. Fatigue properties were determined by axially loading flat laminates that had alternate layers at 0°, +5°, +10°, and +15° to the principal axis. Properties are also presented for three special constructions of unwoven glass fiber laminates in which 71.4 percent of the fibers are parallel to the principal axis, 85.2 percent are parallel to the principal axis, and all layers were parallel, with each layer supported by a "carrier" of 2-mil glass fabric. All experiments were conducted on unnotched

ASD TDR 62-464 (Continued)

specimens at zero mean stress after conditioning in an atmosphere maintained at 73° F and 50 percent relative humidity; loads were applied parallel to the principal axis.

ASD TDR 62-629

October 1962

SUBJECT: EFFECT OF ELEVATED TEMPERATURES ON STRENGTH PROPERTIES OF REINFORCED PLASTIC LAMINATES
INVESTIGATOR: K. H. Boller
CONTRACT: AF33(616)-61-06, Forest Products Laboratory
ABSTRACT: Several reinforced plastic laminates that have shown promise of having good strength properties at elevated temperatures have been investigated to determine their strength within their useful range of temperature and duration of exposure. Results of tension, compression, and interlaminar shear evaluations are summarized for six laminates after exposure to temperatures ranging from room temperature to 1000°F and soak from about 2 minutes to 1000 hours.

Results show that strength usually decreases with heat application. Further application of heat at constant temperatures, however, sometimes results in an increase in strength, but continued exposure at the higher temperatures ultimately results in a complete loss of strength. Exceptions and various degrees of degradation depend on the kind of material, the temperature, and the period of exposure. Hence, curves are presented for six materials, three mechanical tests, and a range of duration of exposure.

ASD TDR 62-635

August 1962

SUBJECT: CARBON-BASE FIBER REINFORCED PLASTICS
INVESTIGATOR: D. L. Schmidt, W. C. Jones
ABSTRACT: Current research on high-temperature materials has led to the development of a new class of non-melting filamentous materials. These fibers are carbonaceous in composition, and hold great promise for use as reinforcing agents in both structural and ablative plastic composites. First generation developments in carbon-base fiber technology are reported. Techniques for synthesizing the fibered materials are discussed, together with the properties and characteristics of available materials. Unique plastic composites were prepared using carbon-base fibers and fabrics. Initial empirical results indicate that the composites are useful for both high-temperature structural and ablative purposes.

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Additional improvements will be required to realize the inherent potential of carbon-base fibers. Several technical recommendations are given for eliminating current material deficiencies and improving fiber properties.

ASD TDR 63-81

March 1963

SUBJECT: AF-994 - A SUPERIOR GLASS FIBER REINFORCEMENT FOR STRUCTURAL COMPOSITES
INVESTIGATOR: R. C. Tomashot
ABSTRACT: As the result of one of the programs sponsored by Nonmetallic Materials Laboratory, a new glass fiber reinforcement for structural composites has been developed.

This report reviews the applied research leading to this development, and presents initial information on the mechanical properties of laminates reinforced with both woven and nonwoven types of this reinforcement, and electrical properties of the bulk glass material.

ASD TDR 63-297

April 1963

SUBJECT: DEVELOPMENT OF CHROMIUM COMPOSITE ALLOY WITH HIGH TEMPERATURE OXIDATION AND EROSION RESISTANCE
INVESTIGATOR: J. F. Masterson
CONTRACT: AF33(657)-8422, Bendix Corp.
ABSTRACT: The effects of extrusion and rolling variables on the quality and mechanical behavior of a powder metallurgy chromium-magnesium oxide composite have been studied.

Hot rolling at 2200°F and finish rolling at 900°F with reductions of 40 to 55 percent provided sound, contamination free sheet having a ductile-brittle transition temperature of 45°F in the recrystallized condition. Oxidation, erosion and nitridation behavior were observed to be improved over unalloyed chromium. Preliminary studies have indicated that a strain aging phenomenon may be responsible for the brittle behavior observed with as rolled and stress relieved sheets. Further work is required to resolve this anomaly.

The results of this initial program have indicated that the full potential of chromium composites can be realized with additional development directed toward strengthening, and further retardation of nitrogen diffusion at elevated temperature.

ELASTOMERIC MATERIALS

WADC TR 56-331, Part VI

May 1962

SUBJECT: HIGH TEMPERATURE RESISTANT ELASTOMER COMPOUNDS
INVESTIGATOR: D. R. McGillvary, et al
CONTRACT: AF33(616)-6998, Firestone Tire & Rubber Co.
ABSTRACT: An extensive compounding investigation of ethylene-propylene elastomer, including some statistically designed experiments, was made. The best heat stabilizing system evaluated for ethylene-propylene elastomer was a combination of antimony trioxide and Hypalon. Compounding data on Diene, Coral, Zetafin, hydrogenated butadiene/methylmethacrylate, and blends of Diene with butyl, SBR, Coral and Hevea elastomers are reported. Stress relaxation measurements were made at 300 and 350°F on some of the more heat resistant compounds. Silicone and Viton elastomers had the slowest relaxation rates. Exposure of certain vulcanizates to radiant heat of 1000°F and contact with an 800°F plate was accomplished and the results photographed. Static fatigue experiments on HT-1, polyester (Terylene) and Nylon 66 tire-cords were conducted. Additional adhesion data on Ht-1 to resin-cured butyl was obtained.

A new technique for measuring the inherent thermal stability of vulcanized compounds has been devised which utilizes a modified high temperature chromatograph. Ethylene-propylene elastomer was one of the most stable elastomer evaluated.

WADC TR 56-331, Part VII

January 1963

SUBJECT: HIGH TEMPERATURE RESISTANT ELASTOMER COMPOUNDS
INVESTIGATOR: L. J. Gaeta, et al
CONTRACT: AF33(616)-6998, Firestone Tire & Rubber Co.
ABSTRACT: Two techniques for measuring the inherent thermal stability of polymers and their vulcanizates have been developed which utilize a modified high temperature chromatograph: programmed temperature aging and progressive isothermal aging. Silicone and fluorocarbon polymers have been found to be more thermally stable than their vulcanizates; butyl and EPR polymers were found to be less thermally stable than their vulcanizates. Equipment was designed and built for testing the tensile properties of vulcanizates at temperatures as high as 800°F in the inert atmosphere of a liquid metal bath. Other high boiling liquids could be substituted for the liquid metal. Silicone and fluorocarbon stocks retained the most strength at 800°F. A polybenzimidazole polymer (PBI) of potential use as a textile fiber was evaluated for tirecord properties. PBI had higher thermal stability than HT-1 but also had lower tenacity, modulus and elongation.

WADC TR 53-373 Sup 10

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WADC TR 59-338, Part V

March 1963

SUBJECT: COMPOSITE INORGANIC RESILIENT SEAL MATERIALS
INVESTIGATOR: L. L. Smith
CONTRACT: AF33(616)-7310, Armour Research Foundation
ABSTRACT: The object of this program is to perform research on composite materials for use as fluid seals in the cryogenic to 2000°F, 0-5000 psi range. The composite material consists of fibers, usually metallic, in a metallic or polymeric matrix.

Stainless steel skeletons impregnated with elastomers have been successfully evaluated as low clamping force seals. Cryogenic seals made of babbitt and other fillers reinforced by stainless steel fibers have sealed at -320°F. Stainless steel skeletons filled with silver have been successfully evaluated as rotating and reciprocating seals at temperatures up to 820°F. Theoretical and experimental investigations of these composites have shown that reinforcement by sintered fibers result in higher recovery and strength in pure metals such as silver, copper, and silver-indium.

WADD TR 60-922, Part II

January 1963

SUBJECT: STRESS RELAXATION OF ELASTOMERS
INVESTIGATOR: F. S. Owens
ABSTRACT: This report covers the investigations of the effects of varying the initial force, elongation, temperature, and the experimental procedure on the short-term stress relaxation behavior of an acrylonitrile-butadiene (Hycar 1002) rubber. Also, the effects of varying the milling time, press cure time, post cure time, the experimental procedures, and each of the compounding ingredients for a fluorohydro-carbon elastomer, Viton B, is reported. The results show that while the stress relaxation behavior of one elastomer is somewhat like that of another one, each has its own innate characteristics. Also, the results show that stress relaxation is a degradative process which depends to a great extent upon the previous stress history, the state of cure, the experimental procedure, and the conditions under which the investigation is carried out.

WADD TR 61-167, Part II

March 1962

SUBJECT: MOLECULAR CONFORMATION AND ULTIMATE PROPERTIES OF ELASTOMERS
INVESTIGATOR: M. Morton, N. Bletso, I. Piirma
CONTRACT: AF33(616)-6986, Akron University

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WADD TR 61-167, Part II (Continued)

ABSTRACT: Several investigations have been carried out in an effort to determine the effect of polymer-solvent interaction on the properties of bulk films cast from different solvents. Swelling, sol and stress-strain evaluations of pale crepe films, cast from the good solvents carbon tetrachloride and methyl cyclohexane and the poor solvent benzene, indicate that the films contained the same magnitude of physical entanglements. This work indicated that the differences initially obtained and previously reported were a result of chain scission processes occurring in the films cast from the good solvents. Critical entanglement molecular weights were determined from polymethylmethacrylate in good and poor solvents at 5.0% concentration. The results were in agreement with those previously determined at 1.5%. In order to determine the concentration at which polymer-solvent interaction is no longer in operation, the ratios of $\eta_{\text{good}}/\eta_{\text{poor}}$ were determined at various concentrations. The data indicate that a concentration of approximately 35%, the polymer-solvent interaction is neutralized by polymer-polymer interaction.

WADD TR 61-167, Part III

February 1963

SUBJECT: MOLECULAR CONFORMATION AND ULTIMATE PROPERTIES OF ELASTOMERS
INVESTIGATOR: M. Morton, A. Gent, J. S. Glazman
CONTRACT: AF33(616)-6986, Inst. of Rubber Research
ABSTRACT: Measurements have been made of permanent set, percent rubber sol (i.e., material soluble in benzene but not in acetone), and equilibrium swelling for films of natural rubber vulcanized by gamma irradiation while held in the stretched state. The results show (i) that the degree of crosslinking is largely unaltered by orientation, for extensions of up to about 700%, and (ii) that the simple two-network theory provides a rather poor estimate of permanent set. Some observations of cavitation in swollen gels subjected to reduced pressures are also reported.

ASD TDR 62-30, Part I

June 1962

SUBJECT: MECHANISM OF CROSS-LINKING IN THE VULCANIZATION OF NEW ELASTOMERIC POLYMERS
CONTRACT: AF33(616)-7798, Wyandotte Chemicals Corp.
INVESTIGATOR: K. L. Paciorek, et al
ABSTRACT: The objective of this program was the elucidation of the mechanism of the peroxide induced cross-linking of fluoroelastomers.

ASD TDR 62-30, Part I (Continued)

Several model compounds: 4,4-dihydroperfluoroheptane (V), 5,6-diperfluoroethyl-4,4,7,7-tetrahydroperfluorodecane (VI), and 3,4-diperfluoroethyl-2,2,5,5-tetrahydroperfluorohexane (VIII) were prepared. Interestingly, VI and VIII as well as 2,2,4,4-tetrahydro-3-perfluoroethyl-5-iodoperfluoroheptane (XIII) were resolved by gas chromatography into their respective diastereoisomers. Treatment of XIII with triethylamine or potassium hydroxide gave either the olefin (XVI) or the diene (XIV) depending on the conditions chosen. VI afforded different olefins depending on whether triethylamine or potassium hydroxide was employed.

Treatment of 4-hydroperfluoroheptene-3(IV) and VIII with benzoyl peroxide failed to yield any interaction products.

The lowest curing temperature of the vinylidene-fluoride perfluoropropene (VF-PFP) copolymer-benzoyl peroxide magnesium oxide-system was found to be ca. 100°C, whereas the optimum curing temperature was found to be 140-150°C. No physical test data could be obtained for the VF-PFP copolymer-benzoyl peroxide system, due to the spongy nature of the product.

ASD TDR 62-311

May 1962

SUBJECT: EXPANDABLE RIGIDIZABLE STRUCTURES
INVESTIGATOR: T. L. Graham
ABSTRACT: A new concept has been preliminarily investigated by which initially flexible impregnated fibrous composite structures can be rigidized after expansion due to crosslinking of the impregnant which is induced by selected inflating gases.

Members of two classes of resin intermediates, the epoxides and the urethanes, which cure when exposed to amine and moisture-vapor environments, respectively, have been investigated. The chemical structures and the reaction mechanism of these resin intermediates are discussed. Data is presented showing the strength of structural laminate samples rigidized by this method in which resin catalyst systems were used. The permeability characteristics of candidate expandable structures for inner liners to an amine catalyst are given. Expandable structures successfully rigidized using amine-epoxy resin systems are described.

ASD TDR 62-400

August 1962

SUBJECT: ABLATIVE ELASTOMERIC INSULATION MATERIALS
INVESTIGATOR: R. E. Headrick
ABSTRACT: This is a report of a preliminary investigation of representative vulcanizates of nearly all types of elastomeric polymers as ablative insulations from oxy-acetylene torch and the arc-plasma jet evaluations. In these evaluations, acrylonitrile/butadiene copolymer (NBR) based compounds, which are now the most widely used in insulation compounds, were rated only fair among the materials investigated. The silicones, the polysulfides, and vinyl pyridine/acrylonitrile vulcanizates exhibited appreciably higher thermal protection than did NBR vulcanizates. Also reported, are the results of a literature survey and a detailed discussion of the evaluation procedures and equipment.

ASD TDR 62-709

January 1963

SUBJECT: RESEARCH ON ELASTOMERIC AND COMPLIANT MATERIALS FOR AEROSPACE SEALANTS
INVESTIGATOR: R. M. Heitz, R. W. Hunter, P. J. D'Anna
CONTRACT: AF33(616)-8258, Northrop Space Laboratories
ABSTRACT: Requirements for elastomeric sealants are described for vehicles typical of four space mission profiles. The effects of natural and induced environments are discussed and the major sealant applications outlined. A survey of technical literature and an industry survey on sealants are documented. Seven classes of elastomers were selected: Silicones, Fluoroelastomers, Chlorosulfonated Polyethylenes, Polychloroprenes, Chlorobutyl and Chlorobutyl-Polychloroprene Blends, Polysulfides and Polyurethane. Compounds were prepared for which the base polymer, compounding ingredients or cure schedules were varied. Specimens were exposed to seven different irradiation conditions, including gamma or combined gamma and ultraviolet radiations in vacuum or air for times of 16 and 100 hours. Tensile strength, 100 percent modulus, ultimate elongation and hardness were determined before and after exposure, supplemented by analysis of the gases generated during the irradiation tests and visual examination of the specimens. The effects of the radiation conditions on materials are compared both within a class and for the different classes. For most of these elastomers, the primary reaction seemed to be cross linking with only minor amounts of chain scission. Self-sealing concepts are compared analytically to several other methods of prevention and control of meteoroid damage and the potential air leakage due to penetration.

ASD TDR 62-902

December 1962

SUBJECT: ETHYLENE-PROPYLENE RUBBER-PHYSICAL AND SOLUTION PROPERTIES

INVESTIGATOR: T. Dudek, F. Bueche

ABSTRACT: A detailed investigation of ethylene-propylene rubber (EPR), du Pont's ECD-330, has been made. The polymer-solvent interaction parameter, χ for EPR (53 mole percent ethylene) was determined as a function of degree of crosslinking in n-heptane and benzene. The parameter for a well characterized EPR gum vulcanizate was determined in fourteen solvents and the cohesive energy density of the co-polymer was evaluated. The composite creep curves for gum and filled EPR vulcanizates were obtained and compared with SBR. The tensile strength and elongation at break of SBR and EPR gum vulcanizates were determined as a function of network chain density. A large viscous contribution to the tensile strength versus chain density curve decreased as more equilibrium conditions were approached. Mullins softening curves were determined for filled EPR vulcanizates and the results interpreted in terms of the molecular theory for the "Mullins Effect". The rate and temperature dependence of tensile strength for black filled EPR were studied. The same limiting degree of cure was obtained for the filled and gum vulcanizates of EPR. The research presented in this report is part of a continuing program aimed toward the establishment of theories which explain the mechanical and physical properties of elastomeric polymers. Such theories are essential in guiding future efforts to attain elastomers of greater capabilities.

ASD TDR 62-903

February 1963

SUBJECT: TENSILE STRENGTH OF AMORPHOUS GUM RUBBERS

INVESTIGATOR: F. Bueche, T. Dudek

ABSTRACT: A new molecular theory of tensile strength has been formulated for amorphous gum vulcanizates at temperatures well above the glass temperature and found to be in agreement with recent experimental data. A primary assumption which is supported by experiment is made, i.e., that the sample will fail when a certain critical equilibrium energy is stored in each chain backbone bond of the gel fraction of the rubber. It is shown that the stress on the rubber at break will consist of two parts, the normal kinetic theory stress on the network, and the highly rate dependent stress held by the fully extended chains. It is shown that the fraction of the chains in the fully extended state at the instant of break is greater for a lightly crosslinked rubber than for a highly vulcanized rubber under non-equilibrium conditions. The theory allows the molecular weight of the freely orienting unit to be calculated. Reasonable values are obtained for EPR and SBR which lend support

ASD TDR 62-903 (Continued)

to the validity of the ideas involved. The kinetic theory stress contributes the major portion of the stress at break in highly crosslinked amorphous gum elastomers. However, the stress at break for low degrees of crosslinking is largely the result of stresses in the most highly elongated chains.

ASD TDR 62-1113

March 1963

SUBJECT: HIGH TEMPERATURE STABILIZATION SYSTEMS FOR
HYDROCARBON ELASTOMERS

INVESTIGATOR: J. K. Sieron

ABSTRACT: Compounding research on butyl and ethylene-propylene-diene (EPDR) elastomers has uncovered promising chemical additives which substantially increased the useful life of both materials at temperatures up to 500°F. New vulcanization systems providing superior heat stability for EPDR were also elucidated. Stannous oxide proved to be the most effective high temperature stabilizer found for resin cured butyl; 2-mercaptobenzothiazole provided maximum heat stability for sulfur donor cured EPDR; and a combination of antimony trioxide and a chlorinated hydrocarbon was best for peroxide cured EPDR. The tensile properties of the best butyl and EPDR compounds were determined after various aging times at temperatures over the 300°F to 500°F range. EPDR compounds exhibited surprisingly good resistance to aging at 500°F. Nitrogen versus air heat aging experiments indicated that all of the stabilization systems elucidated were effective against oxidation rather than heat.

ASD TDR 63-167, Part I

March 1963

SUBJECT: COMPOSITE SEAL MATERIALS

INVESTIGATOR: R. Headrick

CONTRACT: AF33(657)-8271, Armour Research Foundation

ABSTRACT: Fibrous composite materials (soft metals or elastomers reinforced by a strong metal fiber structure) were found to be suitable for fluid seals for extreme aerospace environments. Research was conducted to obtain optimization of such materials. For example, techniques were developed for the extruding and rolling of this material for the economical production of large seals. The material was successfully evaluated by outside organizations and Armour for extreme environment applications such as ball valve seats, reciprocating rod seals, fuel tank O-ring seals, and high vacuum system seals.

FIBROUS MATERIALS

ASD TDR 62-435

July 1962

SUBJECT: INVESTIGATION OF POLYBENZIMIDAZOLE FIBERS AT HIGH TEMPERATURE

INVESTIGATOR: F. S. Dawn, J. H. Ross

ABSTRACT: An investigation was conducted on the behavior of fibers spun from polybenzimidazole (PBI) during and after exposure to high temperatures and gamma radiation environment. The yarn has an initial tenacity of 4.1 grams per denier and 6.8 percent rupture elongation, and at 600°F (with a 15 minute exposure time) has a tenacity of 2.6 grams per denier and 9.2 percent rupture elongation. As a comparison, the tenacity of HT-1 dropped from 5.92 to 2.51 grams per denier for the same temperatures (except exposure time was 5 minutes). (1) The PBI yarn was found to be structurally intact after exposure to 977°F for 5-10 minutes. (Future work will include determination of strength properties at this temperature). The strength of the PBI fiber was approximately 10 percent less than that of HT-1 after gamma radiation exposure to 1.4×10^9 ergs gm^{-1} carbon hr⁻¹ total dosage. These results indicate that fibers of this polymer are definite candidates for utilization in expandable structures and decelerators where temperatures of 600° to 900°F are anticipated.

ASD TDR 62-457

August 1962

SUBJECT: A THEORETICAL INVESTIGATION OF CRIMP INTERCHANGE IN A WOVEN FABRIC UNDER BIAXIAL STRESS

INVESTIGATOR: P. G. Popper

ABSTRACT: This report describes the theoretical behavior of a woven fabric which is subjected to biaxial stresses. The stress-strain equations are derived for an idealized plain-weave fabric which extends by means of crimp interchange. To obtain solutions to these equations it was necessary to use an iterative procedure on a digital computer. The results have been presented in several ways and may be used in the design of such items as pressurized structures. A number of sample solutions using the computer results are given.

ASD TDR 62-518

July 1962

SUBJECT: HIGH STRENGTH, GLASS FIBER WEBBINGS, TAPES,
AND RIBBONS FOR HIGH-TEMPERATURE PRESSURE-
PACKAGED DECELERATORS

INVESTIGATOR: E. S. Cobb

CONTRACT: AF33(616)-7441, Owens-Corning Fiberglas Corp.

ABSTRACT: The purpose of this work was to develop high strength glass fiber tapes, ribbons, and webbings for use in high temperature, pressure packed decelerators. Loss of strength was determined to result from the effects of pressure packaging at high temperatures. Glass fiber tapes and webbings were developed and produced which do not drop in tensile strength beyond the strength of the greige goods when exposed to simulated pressure packing conditions at elevated temperatures. A test conditioning method was developed which establishes the performance characteristics of glass fiber structures for decelerators. Optimum yarn construction for the series of tapes and webbings was determined. A system of partial heat cleaning and the application of a silicone finish was developed as a method to prepare the tapes and webbings so that they have optimum strength retention characteristics after exposure to simulated pressure packaging conditions. Methods of joining were developed in which a glass sewing thread and special stitching patterns produce 90% seam efficiency.

ASD TDR 62-542, Part I

June 1962

SUBJECT: NEW AND IMPROVED MATERIALS FOR EXPANDABLE
STRUCTURES

INVESTIGATOR: D. M. Marco

CONTRACT: AF33(616)-7854, Goodyear Aircraft Corp.

ABSTRACT: Work performed during the first year of Contract No. AF33(616)-7854, "New and Improved Materials for Expandable Structures", is presented. Details of specific research programs which have been initiated are not included. The objective of the program was to establish the general material requirements for expandable structures; review present material research activities in this area; and to recommend and generate specific program areas in which additional research is required. General approach used to fulfill these objectives was to review recent applicable technical publications and reports; and contact active researchers and organizations working in this field. Information thus obtained was reviewed and summarized along with information available at GAC from which recommendations were made as to research programs which should be initiated. Materials review was divided into two areas; one, materials for space station and accessories; and two, re-entry materials. General environmental conditions to which expandable

ASD TDR 542, Part I (Continued)

structures materials will be exposed, general material parameters, results of literature survey in the areas of textile materials, high temperature materials, polyurethane, and ultraviolet degradation of polymers; and the research review with respect to space station and accessories, and re-entry materials have been summarized and are presented. Also reported, is information obtained from contracts with various organizations and a bibliography of documents and references used in this program.

ASD TDR 62-542, Part V

February 1963

SUBJECT: NEW AND IMPROVED MATERIALS FOR EXPANDABLE STRUCTURES (PHASE IV - HIGH-TEMPERATURE PROTECTIVE STUDY)

INVESTIGATOR: R. T. Kolarik, D. M. Marco

CONTRACT: AF33(616)-7854, Goodyear Aircraft Corp.

ABSTRACT: Various methods for providing thermal protection for expandable aerospace vehicles during atmospheric glide re-entry have been analytically evaluated at three wing loading conditions. Consideration was given to radiation, insulation, and ablation protective systems compatible with expandable structures. Information on vehicle configurations assumed and re-entry parameters involved is presented. Thermo-physical and ablative properties of thermal protection materials obtained from a literature review are reported. Analytical techniques used in the evaluation of the various protective systems are given. The effectiveness of these systems in protecting nose and wing leading edge stagnation regions of the vehicles is presented in graphic form. Influence to certain re-entry parameters on effectiveness of thermal protection systems is also presented. Areal weights required to protect the re-entry vehicles to a tolerable temperature level are tabulated for various locations. Conclusions on a comparative performance of thermal protection systems and recommendations for future research and development activities are presented.

ASD TDR 62-613

August 1962

SUBJECT: CRITERIA FOR RUPTURE OF CERTAIN TEXTILE STRUCTURES UNDER BIAXIAL STRESS

INVESTIGATOR: P. Popper

ABSTRACT: This report outlines a procedure for determining the combinations of biaxial stress which will cause failure in certain types of fabrics. Solutions are given for knitted fabrics and

ASD TDR 62-613 (Continued)

"diamond" structures, and are presented in the graphical form of a rupture envelope. Also, a commonly used rupture criteria for solid materials is included for comparison with the graphic results.

ASD TDR 62-727, Part I

August 1962

SUBJECT: ULTRA-FINE HIGH TEMPERATURE HIGH STRENGTH METALLIC FIBERS
INVESTIGATOR: C. A. Gorton, C. C. McMahon
CONTRACT: AF33(616)-8366, Hoskins Manufacturing Co.
ABSTRACT: Eight superalloys: A-286, Elgiloy, Hastelloy B, M-252, Rene' 41, U-500, U-700, and Waspaloy were processed to ultra-fine fibers of approximately .001 inch diameter and evaluated for drawability, room temperature, tensile strength and elongation and effect of reduction of area on tensile properties. The Elgiloy and Hastelloy B alloys processed more readily with less wear than the other alloys.

The tensile strength of all alloys except A-286 were in the range of 160,000 psi as solution heat treated. Alloy A-286 tensile strength was approximately 100,000 psi. The alloys in order of decreasing strengths were U-700, Rene' 41, Hastelloy B, Waspaloy, U-500, M-252, Elgiloy, and A-286.

Yarns composed of seven, nineteen and thirty-seven filaments of Elgiloy and Rene' 41 were successfully processed to less than .003 inch diameter when sheathed with Alloy Chromel C, but the sheath could not be removed without damage to the fibers. High temperature tensile tests of the sheathed yarn at 1600°, 1800°, and 2000°F in air and argon atmospheres indicated increased strength in order of increased filaments. The Chromel C sheath protected the core fibers from oxidation and improved the high temperature strength of the yarn at 1800° and 2000°F.

ASD TDR 62-782

October 1962

SUBJECT: INVESTIGATION OF THE THERMAL BEHAVIOR OF GRAPHITE AND CARBON-BASED FIBROUS MATERIALS
INVESTIGATOR: J. H. Ross, F. S. Dawn
ABSTRACT: The elevated temperature properties of graphitized and carbonized polymeric fibrous materials were investigated as part of a research program on high temperature fibrous materials for use in parachutes and other decelerators, and in expandable structures. Experimental quantities of woven materials

ASD TDR 62-782 (Continued)

were evaluated after they were exposed to various temperatures for specific periods. One of these materials was partially graphitic (as shown by some order in X-ray diffraction). All three had been converted to carbon by controlled pyrolysis of polymers while in fabric form. Characteristics investigated as related to temperature and time at temperature were: breaking load to rupture, breaking elongation, energy absorption, and breaking strength after repeated stressing.

The graphitized fibrous material was superior in tenacity initially and at 600°F, to the carbonized materials investigated. Also, these materials were superior to nylon at temperatures above 350°F. Future investigations will be conducted to characterize the behavior of the carbonized and graphitized fibrous materials in the range of 600° to 2000°F.

ASD TDR 62-810

November 1962

SUBJECT: THERMAL TRANSPORT AND RADIATIVE PROPERTIES OF FIBROUS STRUCTURAL MATERIALS

INVESTIGATOR: G. Engholm, S. J. Lis, R. J. Baschiere

CONTRACT: AF33(616)-8181, General American Transportation Corporation

ABSTRACT: Measurements were made of the thermal conductance and total normal emittance of the following fibrous structural materials: 1N Nylon, 11N Nylon; HT-1 Types I, II, and III; Pluton; Glass Fiber Cloth; Aluminized Glass Cloth; Vitreous silica, uncoated, RTV 60 coated, and Parson's Optical Black coated; Graphite cloth; and Rene' 41 cloth, uncoated and CS-105 coated. Data were obtained with a thermal conductance apparatus and a thermal emittance apparatus both developed during the program.

The thermal conductance apparatus employed a transient technique to measure the thermal conductance of fibrous materials under varying conditions of temperature (200 to 1500°F), environmental pressure (20.2 psia to 0.4 mm Hg), biaxial tension (1 to 109 ppi), and compression (1.9 to 13.7 psi). Based upon the Cenco-Fitch measurements of Mylar film and asbestos paper standards, the thermal conductance apparatus measurements were found to be valid to within 7%. The results of the thermal conductance tests have shown that the presence of a coating on a material does not alter its conductance value. Environmental pressure has been found to affect the conductance values to the extent of causing a two-fold increase in the conductance when the pressure is changed from vacuum to an ambient atmospheric pressure.

The thermal emittance apparatus utilized a goniometric technique to spectrally scan the transmitted and reflected radiation from material samples subjected to biaxial tension. Total normal emittance values were calculated from these data. The test data indicated that the magnitude of biaxial tension has no appreciable effect on emittance. The test results also showed that the emittance values, derived at room temperature, can be used in the determination of higher temperature emittance values. A check made to establish the accuracy of the emittance apparatus has shown it to be suitable for the determination of the emittance of both diffuse and specular surfaces to within 29%.

The validity of the temperature measurements obtained from miniature thermocouples woven into the material samples were evaluated. The large variations of temperature (30 to 100°F) which could be found in small areas (1.8 inch diameter) indicated that an estimate of the local temperature could be obtained, but the exact value of the temperature could be established only within 10%. If a "direct method" of total normal emittance were used, the 10% possible error in temperature measurement would result in a 40% possible error in the determination of the emittance value. This result further justifies the use of the "indirect method" of total normal emittance measurement which was used in the program.

ASD TDR 62-851

October 1962

SUBJECT: CONVERSION OF HIGH MODULUS MATERIALS INTO FLEXIBLE FABRIC STRUCTURES
INVESTIGATOR: M. J. Coplan, et al
CONTRACT: AF33(616)-7222, Fabric Research Laboratories, Inc.
ABSTRACT: Flexible "fabric-like" structures exhibiting a moderate tensile strength during a short time exposure in a 1500° to 2000°F oxidative environment are investigated. The class of candidate materials is characterized by a high modulus of elasticity and low elongation. Structural designs utilizing these materials in filamentous form and other configurations are considered. Theoretical and experimental studies of such structures are undertaken.

The bending recovery of monofilaments, single yarns and fabrics, and the torsion recovery of monofilaments, single yarns and plied yarns are investigated. The rigidity and bending recovery of a nonwoven are also investigated. The effect of temperature on filament, yarn, and fabric strength, flexibility and bending recovery is discussed. The feasibility of weaving a fabric from fine metal filaments without resorting to the complexities of plying

ASD TDR 62-851 (Continued)

twisted singles yarns is demonstrated. The properties of a fabric woven from a yarn composed of ribbon filaments are compared to those of a fabric woven from an equivalent yarn composed of circular filaments of the same material and cross-sectional area. Fabric structural designs incorporating mechanical operators such as flex-points and/or hinge-points are investigated.

ASD TDR 62-964

January 1963

SUBJECT: SYMPOSIUM ON FIBROUS MATERIALS
INVESTIGATOR: J. H. Ross
ABSTRACT: This report is a collection of papers presented at the Directorate of Materials and Processes, "Symposium on Fibrous Materials" held in Dayton, Ohio, on 16-17 October 1962. The purpose of this symposium was to review the recent progress on both contractual and internal research programs sponsored by the Fibrous Materials Branch of the Nonmetallic Materials Laboratory in the area of new fiber forming substances, protective chemical finishes and coatings and translation of fibers into flexible, high strength materials.

ASD TDR 62-988

November 1962

SUBJECT: A STUDY OF THE LOAD BEARING BEHAVIOR AND OTHER CHARACTERISTICS INFLUENCING VIBRATION OR FLUTTER IN DECELERATOR RIBBONS
INVESTIGATOR: R. A. Meyer
CONTRACT: AF33(657)-7235, Cook Technological Center
ABSTRACT: The results of an investigation of the load-bearing behavior and characteristics influencing vibration or flutter in nylon decelerator ribbons are presented. MIL-T-5608E, Class E nylon tapes of 2-inch width have received predominant attention in analytical and experimental studies to determine their static and dynamic properties and flutter characteristics. Verification of the results of theoretical flutter analyses was provided by wind tunnel experimentation. Flutter was evidenced at subsonic speeds but not at supersonic velocity conditions. In most cases, however, the existence of subsonic flutter in component ribbons would not significantly affect over-all parachute performance. Consequently, presently available MIL-T-5608E, Class E nylon tapes are believed to be adequate for parachute fabrication purposes.

ASD TDR 63-57

February 1963

SUBJECT: DETERIORATION OF FIBROUS MATERIALS BY ULTRA-VIOLET LIGHT
INVESTIGATOR: C. V. Stephenson, et al
CONTRACT: AF33(616)-7701, Southern Research Institute
ABSTRACT: This report discusses primarily the effects of ultra-violet light on the tensile properties of four polymeric materials - polyethylene terephthalate (Dacron), polybenzimidazole, a thiazole polymer, and a du Pont experimental material designated as Ht-1.

Among these materials, exceptions were found to the usual trend that degradation is more severe the shorter the wavelength of light. These exceptions are discussed in terms of an absorption of light near the surface of the material that, in effect, prevents the interior from being irradiated.

Among materials studied, those that are known to contain hydrogen are more susceptible to irradiation damage in an oxygen atmosphere than in a nitrogen atmosphere or in a vacuum.

ASD TDR 63-61

April 1963

SUBJECT: STUDY OF ENVIRONMENTAL EFFECTS ON HIGH TEMPERATURE ORGANIC MATERIALS
INVESTIGATOR: J. C. McGrath
ABSTRACT: HT-1, an improved high temperature yarn, developed by the E. I. du Pont de Nemours and Company, Inc., has been woven into fibrous structures such as webbings, tapes, and fabrics for possible use in personnel and other type decelerators, or for other purposes where a high temperature requirement exists. These fibrous structures have been evaluated after exposures to (1) outdoor weathering at the Naval Aux. Air Station, El Centro, Calif., for periods of 1, 2, 3, 4, 5, and 6 weeks during January and February 1962. (Additional exposures of two HT-1 fabrics and two comparable nylon fabrics were conducted in July and August 1962.), (2) accelerated weathering in a Weatherometer, Type XW, for periods of 20, 40, 60, 80, 100, and 150 hours and (3) temperatures of 400°, 500°, and 600° for periods of 1-1/2, 3, 6, 12, and 24 hours. HT-1 materials retained as high, and in some instances a higher percentage of strength after outdoor and accelerated weathering than nylon fabrics. All retained at least 35% up to 600°F.

ASD TDR 63-62

April 1963

SUBJECT: ELEVATED TEMPERATURE BEHAVIOR OF FIBERS
INVESTIGATOR: S. Schulman
ABSTRACT: An investigation was conducted on the behavior of fine filaments at ambient and elevated temperatures. Polymeric and inorganic filaments of diameters ranging from 0.2 mil to 1.8 mil in diameter were included. The ambient temperature tensile strengths ranged from 68,000 psi to 501,000 psi. The effects of temperatures up to 1700°F on the properties were determined.

ASD TDR 63-92

May 1963

SUBJECT: A SURVEY OF TEST PROCEDURES FOR EVALUATING SHORT BRITTLE FIBERS
INVESTIGATOR: S. Schulman, J. Epting
ABSTRACT: A survey was conducted to determine what techniques and devices are available for evaluating the mechanical properties of short brittle fibers and continuous filaments; the most important properties are tensile strength and modulus of elasticity of the material. The results of this survey indicated several characterization methods that may have some merit. A critical analysis of these methods was made, since the results of such an analysis should prove helpful in establishing a standard evaluation procedure for short brittle fibers.

ASD TDR 63-197

April 1963

SUBJECT: SYNTHESIS OF S-TRIAZINYL POLYETHERS BY INTERFACIAL CONDENSATION
INVESTIGATOR: L. G. Picklesimer, T. F. Saunders
ABSTRACT: Several polyphenyl-s-triazinyl ethers have been prepared by interfacial polycondensation of cyanuric chloride or 2-phenyl-4,6-dichloro-s-triazine with various dihydric aromatic compounds. Aromatic compounds used were resorcinol, hydroquinone, 4,4'-dihydroxybiphenyl, and 2,2 bis (p-hydroxyphenyl) propane.

The aromatic compounds were reacted as their sodium salt in water. The s-triazinyl chlorides were dissolved in organic solvents such as benzene, chloroform, dibutyl phthalate, and chlorobenzene.

This is the first instance where polyphenyl-s-triazinyl ethers have been prepared by interfacial polycondensation.

FUELS, FLUIDS, AND LUBRICANTS

WADD TR 60-898, Part III

February 1963

SUBJECT: FLUIDS, LUBRICANTS, FUELS, AND RELATED MATERIALS
INVESTIGATOR: E. E. Klaus, M. R. Fenske, E. J. Tewksbury
CONTRACT: AF33(616)-7590, Pennsylvania State University
ABSTRACT: Report describes continuing program to characterize capabilities of fluids and lubricants for aerospace uses. Preparation of three hydraulic fluid specifications for super-refined mineral oils is noted. Use of a molecular still to extend super-refining is discussed. Instrument bearing lubrication is analyzed and experimental studies and fluids are proposed. Radioactive techniques are proposed for use in studies of lubrication, oxidation, corrosion, and degree of refining. Fundamental studies in boundary lubrication are outlined. Design and calibration of a comparative calorimeter for specific heat determination is discussed. Oxidation severity at reduced pressures is explored. A constant-pressure, long-time thermal stability procedure is developed and used to indicate the stability of aromatic hydrocarbons. Storage stability data are presented. Appendixes discussing the use of petroleum oils in space and the development of experimental gyro-bearing lubricants are included.

FUELS

WADC TR 59-327, Part II, Vol I

February 1962

SUBJECT: EVALUATION OF HYDROCARBONS FOR HIGH TEMPERATURE JET FUELS
INVESTIGATOR: J. O. Smith, et al
CONTRACT: AF33(616)-5799, Monsanto Research Corp.
ABSTRACT: The chemical and physical properties of 146 pure hydrocarbons and petroleum fractions were determined. Best prospective fuels were selected. Correlation methods were developed for estimating properties of pure hydrocarbons. For 15 selected saturated hydrocarbons, the rates of decomposition and rates of particle formation were determined under static and dynamic test conditions at 800°F and in the 1000°-1100°F temperature range, respectively. The structural effect of hydrocarbons on their stability was investigated.

The effect of oxygen, oxygen-, and sulfur-containing contaminants on the decomposition process was investigated. Contaminants showed a selective action with hydrocarbons belonging in different groups: they showed the same inhibiting or promoting effect on decomposition and particle formation.

WADC TR 59-327, Part II, Vol II

February 1962

SUBJECT: EVALUATION OF HYDROCARBONS FOR HIGH TEMPERATURE
JET FUELS. PART II, VOLUME II. Hydrocarbon
Properties
INVESTIGATOR: J. O. Smith
CONTRACT: AF33(616)-5799, Monsanto Research Corp.
ABSTRACT: This volume contains the tabulation for the
physical properties of 146 hydrocarbons.

WADD TR 60-841, Part III

October 1962

SUBJECT: RESEARCH ON MATERIALS FOR USE AS ENDOTHERMIC
FUELS
INVESTIGATOR: J. O. Smith, B. M. Fabuss, et al
CONTRACT: AF33(616)-7845, Monsanto Research Corp.
ABSTRACT: The evaluation of six selected CRC fuels showed
that the paraffinics are to be preferred to the napthenics as endo-
thermic fuels. Heat sink capacities of 800-1065 Btu/lb at 1100°F,
890-1160 Btu/lb at 1200°F and 990-1255 Btu/lb at 1300°F were obtained
with these fuels. Petroleum fractions are as suitable for this use
as the best pure hydrocarbons. High temperature alloys, Hastelloy
L-605 and Inconel-X, promoted coke formation to a greater extent than
did stainless steel. Hydrogen sulfide pretreatment of the tubing and
carbon disulfide as an additive reduced the coke forming activity of
these super-alloys without affecting the course or rate of the cracking
reactions. High temperature experiments showed the feasibility of
carrying out endothermic reactions up to a reactor wall temperature
of 1580°F. A practical method for calculating the bulk fuel tempera-
ture and comparing it with the reactor wall temperature and estimating
the fuel conversion was developed. A study of the effect of tube
geometry on the cracking process showed the important role of the
laminar boundary layer on heat transfer and coke formation.

ASD TR 61-150, Part II

November 1962

SUBJECT: PROPELLANTS FOR ELECTRICAL PROPULSION ENGINES
OF THE CONTACT OR BOMBARDMENT ION TYPE SECOND
ANNUAL (FINAL) REPORT
INVESTIGATOR: V. E. Bedwell, H. E. Dubb, F. E. Holmstrom,
J. F. Hon, W. P. Schilling, M. R. Stevens,
B. L. Tuffly
CONTRACT: AF33(616)-7063, Rocketdyne
ABSTRACT: This technical report describes the second year's
efforts on the investigation of materials and methods for producing
molecular ions and a colloidal ion beam suitable for use in electrical

ASD TR 61-150, Part II (Continued)

propulsion devices. The molecular compounds were selected for study primarily on a basis of expected favorable ionization characteristics and high structural stability to fragmentation. Limiting stable molecular structures were found for several classes of compounds. The experimental techniques for determining physical and chemical properties of candidate materials are outlined, and the experimental results are combined with data from the literature to complete the propellant study program. A theoretical analysis of a colloid producing system which utilizes a convergent-divergent nozzle for rapid condensation of propellant is presented. Equations are given which may be used to optimize the design and operation of a system which produces colloidal particles from condensable vapors. Electrical charging of the droplets is accomplished by ionizing a fraction of the propellant vapor prior to expansion through the nozzle. A graphical approach for obtaining approximate thermodynamic conditions necessary to adiabatically condense propellant vapors in a typical colloid generating system is introduced. Experimental results from operation of a charged, mercury colloid generator are presented. The theoretical and graphical methods may be extended to other propellants if they possess suitable thermodynamic and physical properties.

ASD TR 61-687

April 1962

SUBJECT: THERMAL STABILITY OF EXPERIMENTAL HIGH TEMPERATURE FUELS
INVESTIGATOR: C. L. Mahoney
CONTRACT: AF33(616)-7667, Shell Development Co.
ABSTRACT: The high temperature stability of seven pure hydrocarbon fuels supplied by Wright Air Development Division were tested in the Shell Development Fuel Coker at 600°, 700°, and 800°F under recycle conditions. These fuels were: hexahydroindane, methyl hexahydroindane, pinane, bicyclohexyl, 9-methylperhydrofluorene, 2-ethylperhydrophenanthane, and 1, 1-bis(4-methylcyclohexyl) ethane.

On the basis of very limited data, two trends regarding the effect of structure on high temperature stability were noted. One, condensed polycyclic naphthenes form slightly more deposit than non-condensed structures. Two, the presence of a five-membered ring appears to have a detrimental effect on stability as measured by deposit formation.

ASD TR 61-728

May 1962

SUBJECT: FUTURE AIR FORCE REQUIREMENTS FOR HYDROCARBON FUELS
INVESTIGATOR: J. R. Fultz
ABSTRACT: Future supersonic and hypersonic air-breathing weapon systems will require greatly improved fuels for satisfactory operations. To provide fuels for these systems, the Air Force launched a research program directed toward the development of vastly improved hydrocarbon fuels. This report presents the results obtained so far from the program and the conclusions reached concerning further areas of interest.

ASD TDR 62-254

August 1962

SUBJECT: EVALUATION OF HYDROCARBON MATERIALS AS VAPORIZING FUELS
INVESTIGATOR: K. C. Bachman, E. K. Matthews, D. Zudkevitch
CONTRACT: AF33(616)-7502, Esso Research & Engineering Co.
ABSTRACT: Theoretical and experimental data for determining which hydrocarbon types and structures are most promising for use as vaporizing fuels in Mach 3+ aircraft are presented. Thirty-three hydrocarbons covering a broad range of types and structures were evaluated. The source, e.g. literature, laboratory measurement and correlation method (used also to extend data to 1000°F and 800 psia), of required physical property and thermodynamic data are presented. Calculated liquid and vapor heat transfer coefficients are provided. A heat exchanger designed to check the calculated coefficients, but which could not be made operable during the contract period, is described. Thermal cracking studies on sixteen hydrocarbons are described. Combustion studies in which vaporization was simulated by ultrasonically-produced fuel fogs are described for four hydrocarbons. The results show that condensed or uncondensed dicyclic naphthenes, specifically spiro (4,5) decane, hydrindan, and spiro (5,5) undecane, best fill ASD target requirements for a vaporizing fuel.

ASD TDR 62-328

April 1962

SUBJECT: FLAMMABILITY CHARACTERISTICS OF HIGH TEMPERATURE HYDROCARBON FUELS
INVESTIGATOR: J. M. Kuchta, et al
CONTRACT: DO(33-616)-50-5, Bureau of Mines
ABSTRACT: Experimental data are presented on the auto-ignition temperature characteristics of JP-6 jet fuel and of aircraft engine oils O-60-7, O-60-18, and 5P4E. Minimum auto-ignition tempera-

WADC TR 53-373

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ASD TDR 62-328 (Continued)

tures are given for these materials in various quiescent oxygen-nitrogen atmospheres. The effects of initial environmental pressure, fuel injection pressure, fuel concentration, and test vessel size are described. The variations of auto-ignition temperatures caused by different ignition criteria are discussed.

Quantitative relationships are given which define the variation of ignition delays with auto-ignition temperatures in air under static and dynamic conditions. In addition, auto-ignition temperature data are reported for various JP-6 fuel-oxygen-nitrogen mixtures which were heated in large steel cylinders at low heating rates ($< 15^{\circ}\text{F./min.}$). Included in these data are gaseous product analyses which indicated the extent of oxidation at various temperatures under critical heating conditions.

ASD TDR 62-361

July 1962

SUBJECT: MICROBIOLOGICAL EXAMINATION OF JET FUEL-WATER
SAMPLES RAMEY AND EGLIN AIR FORCE BASES
INVESTIGATOR: A. V. Churchill
ABSTRACT: An investigation was conducted to identify microorganisms permitting microbiological contamination and resulting corrosion of B-52 and KC-135 integral fuel tanks at Ramey and Eglin Air Force Bases. Of the microorganisms isolated, the most predominant groups found were the transparent bacterium and the iron-depositing bacterium. Sulfur-oxidizing bacterium was possibly present in a wet sludge sample from a corroded wing tank. The role of these predominant microorganisms on corrosion of aircraft fuel tanks should be further investigated.

The corrosive activity of jet fuel-water sample from Bulk Storage Tank #55, Ramey AFB, was also investigated, as well as analyses of sludge samples from aircraft based at Ramey AFB.

ASD TDR 62-408

May 1962

SUBJECT: SYNTHESIS OF THIRTY-FIVE HYDROCARBON FUELS
INVESTIGATOR: S. D. Koch, et al
CONTRACT: AF33(616)-7190, AF33(616)-5799, AF33(600)-38448, AF33(600)-39634, Monsanto Research Corp.
ABSTRACT: Thirty-five hydrocarbons were prepared for evaluation as high-temperature jet fuels. Thirty-two of them were prepared in two-liter quantity or more. Synthesis of 59 other

ASD TDR 62-408 (Continued)

polycyclic hydrocarbons for the same purpose under three earlier contracts is also described. Forty-one of the 94 compounds described had never been previously prepared.

ASD TDR 62-451

August 1962

SUBJECT: PROPERTIES OF PLASMAS AS THEY PERTAIN TO THERMAL ARC-JETS
INVESTIGATOR: C. J. Chen, R. Greco, Q. McKenna, J. Meltzer, G. Mitcheltree, R. Price, W. Stoner
CONTRACT: AF33(616)-8173, Plasmadyne Corp.
ABSTRACT: The purpose of this study is to present information which will aid in selecting propellants for use in electro thermal engines. Several potential propellants were examined. These include hydrogen, ammonia, helium, lithium hydride, nitrogen, methane, air, argon, and lithium.

The report includes discussions of important propellant properties and their effect on engine performance and life. The effect of the propellant choice on the operation of the arc-jet is considered. This includes examining theoretical thruster efficiency, engine life and the effect of the engine's mission. Experiments were performed for eight propellants using Plasmadyne's one-kilowatt engine. The results of these tests are discussed.

ASD TDR 62-682

July 1962

SUBJECT: COMBUSTION CHARACTERISTICS OF SPECIAL HYDRO-CARBON JET FUELS
INVESTIGATOR: W. W. Horstman, J. L. Jackson
CONTRACT: AF33(616)-8059, Shell Oil Co.
ABSTRACT: Combustion efficiency, flame radiation, fuel consumption, and altitude relight characteristics of twelve hydrocarbon fuels were evaluated in a General Electric J-79 engine combustor operating at simulated supersonic aircraft speeds and 80,000 feet altitude. Relative combustion efficiency was approximately constant for all fuels at a given test condition but increased as combustion intensity increased. Fuel consumption was inversely proportional to net heat of combustion and correlated with fuel hydrocarbon composition at a constant burner temperature rise. At high heat release rates, the liner temperature and flame radiation were lowered significantly as the paraffin content of the fuel increased. East of altitude relight increased with decreasing ASTM 10% distillation temperature and with increasing paraffin content.

ASD TDR 62-761

October 1962

SUBJECT: SUPERSONIC TRANSPORT FUELS AND LUBRICANTS
INVESTIGATOR: C. J. Johnson
ABSTRACT: The Air Force has the responsibility for research and development of potential fuels and lubricants for Supersonic Transport. Thermal stability requirements of fuels are the most difficult to meet because of the severe operational limitations they impose on the fuel system. This report is a discussion of the potential problems and their elimination. Contractual and in-house effort by the Air Force in studying the causes of thermal instability in fuels is discussed. The responsibility of the Coordinating Research Council, Inc., in conducting large scale Mach 3 simulator tests under Air Force contract is included. The programs discussed will evaluate potential fuels and lubricants with varying capabilities and varying costs. This will allow the aircraft and engine designers a degree of latitude in their design and the selection of the optimum fuel hardware combination.

ASD TDR 62-852

September 1962

SUBJECT: AN INVESTIGATION OF THE THERMAL STABILITY OF POTENTIAL SUPERSONIC JET FUELS
INVESTIGATOR: J. J. Bialy, R. A. Frost, K. L. Dille
CONTRACT: AF33(616)-8045, Texaco, Inc.
ABSTRACT: A Texaco research coker was developed which can thermally stress fuels in a closed reservoir for 0 to 10 hours at 90°F to 500°F under an air, nitrogen or air-nitrogen atmosphere prior to determining fuel thermal stability and vapor phase decomposition. Threshold temperatures for a JP-5, JP-6, Soltrol 170, Alkyl Decalins, Isopropyl Bicyclohexyl, Hydrogenated Lauryl Alcohol, Furfural Extract of LCGO and 3 Kerosines were compared after being thermally stressed for 1 hour at 350°F under nitrogen. Increasing the storage time and the storage temperature in the fuel reservoir was found to affect fuels differently. Investigation of the effect of oxygen availability in the reservoir on fuel stability indicated that there is a relationship between the oxygen content of the blanketing atmosphere and fuel thermal stability, vapor phase deposits and color of the fuel. The effect of repetitive heating and of trace impurities on fuel thermal stability were also investigated.

ASD TDR 62-887

October 1962

SUBJECT: PROPERTIES OF PLASMA AS THEY PERTAIN TO TAJ (THERMAL ARC JET) AND J X B (MHD DUCT) DEVICES
INVESTIGATOR: G. P. Kerr, B. A. Free
CONTRACT: AF33(616)-8235, General Electric Company
ABSTRACT: The objective of the work reported here is to

Establish criteria for working fluids in TAJ (Thermal Arc Jet) and $\bar{J} \times \bar{B}$ (Magnetogasdynamic duct) devices.

TAJ. The range of ΔV for TAJ missions is defined and various criteria set forth for the selection of propellants. The best overall criterion for propellant selection is shown to be weight minimization which also minimizes cost in almost all cases. Hydrogen is shown to be superior to ammonia and helium as a propellant for all missions except highly specialized ones. It is also shown that the best overall performance of hydrogen with present power supplies is secured by maximizing specific impulse only when mission time is unimportant and power supply weight can be minimized.

$\bar{J} \times \bar{B}$. An experimental testing program was carried out with apparatus in which the plasma from an arc jet source passed through crossed electric and magnetic fields to accelerate the plasma for propulsion application studies. Both closed and open accelerator channels were developed. A substantial amount of development work was necessary with the system components, such as the arc plasma source, cathode materials and heating methods, and instrumentation. A flexible system was built which was mounted inside a large vacuum tank. A change in the accelerator cathode operating method was thought to be the only necessary requirement to complete preparations for propellant evaluation.

HEAT TRANSFER FLUIDS

WADD TR 61-186, Part II

April 1962

SUBJECT: ADVANCED HEAT TRANSFER FLUIDS
INVESTIGATOR: S. W. Coffman, L. J. Martin, A. G. Osborne
CONTRACT: AF33(616)-7109, Hughes Aircraft Co.
ABSTRACT: The second year's effort of an electronic dielectric heat transfer fluid study project is described in detail and test results presented in a series of graphs and tables. Organic fluids in the operating range of -65°F to 650°F were studied. Dynamic loop tests were performed on marginal fluids (those for which low-temperature viscosity was too high), since only one fluid of 28 tested passed all screening tests. Siloxane ("Silicone") fluids appear to be most promising overall, although poly(phenylethers) are known to have better thermal-oxidative stability. The dynamic loop apparatus used in the first year's study (WADD TR 61-186) was modified to withstand the higher (650°F) temperature, and the electronic coolant fluids were circulated at maximum hot-spot temperature of 650°F for 100 hours to derive an indication of the thermal-chemical-dielectric-time stability required for present and future electronic equipment used in ground-based, ship-based and satellite vehicles. Only one of the fluids passed all screening tests, but four others were marginal, three in flash and fire points and one in low-temperature viscosity. Others which were marginal in two or more screening tests or "high level" rejects were given supplementary testing, since no firm specification has yet been written. The program also included a continued, extensive literature survey on electronic coolants, electronic equipment cooling, loops, and loop testing.

WADD TR 61-186, Part III

February 1963

SUBJECT: ADVANCED HEAT TRANSFER FLUIDS
INVESTIGATOR: C. W. Mell, K. E. Hostetler, T. D. Hogan
CONTRACT: AF33(616)-7109, Hughes Aircraft Co.
ABSTRACT: The third year's program on extreme temperature range dielectric fluids is described. Test results are presented in a series of tables and graphs. Methods of procuring fluids are described. Organic fluids for use in the operating range of -80°F to 400°F and -65°F to 650°F were studied. Twenty-two new fluids were tested bringing the total for the 3 years to 150 fluids.

All fluids are identified as to the suppliers and their designations. Dynamic loop tests were performed on 9 fluids. Five fluids were successfully operated in the loop at 650°F and three fluids at 400°F . Further improvements were built into the loop. A numerical rating system for the physical and electrical

properties is presented for use as a quick evaluation. The screening and supplementary tests indicated 6 fluids with potential value in the -80°F to 400°F range. Three fluids were found to have potential use in the -65°F to 650°F range. Siloxane ("Silicone") fluids appeared to be most promising fluids, but other types with modification look interesting. An extensive literature survey is included.

ASD TDR 62-434

April 1962

SUBJECT: MEASUREMENT OF POINT-WISE JUNCTURE CONDITION OF TEMPERATURE AT THE INTERFACE OF TWO BODIES IN SLIDING CONTACT

INVESTIGATOR: F. F. Ling, T. E. Simkins

CONTRACT: AF33(616)-8016, Rensselaer Polytechnic Institute

ABSTRACT: An apparatus is described for bringing a rider specimen and a slider specimen into continuous sliding contact so that significant temperatures at the interface are achievable. The design is such that the flow fields of heat in the specimens would be at most two-dimensional, i.e. within engineering approximations; this fact makes possible the measurement of temperatures of the specimens without disrupting the flow fields of heat.

Typical data are presented of speed, normal load, frictional resistance and temperatures at strategic locations on the specimens. Using the heat-equation solutions obtained previously for the configurations concerned, contact-surface temperature distributions of both the specimens are calculated from experimental data. Results give the point-wise, temperature juncture condition at the interface.

ASD TDR 62-738

October 1962

SUBJECT: CONDENSATION OF SODIUM AND RUBIDIUM AT LOW HEAT FLUXES

INVESTIGATOR: J. A. Roth

ABSTRACT: This report presents the design considerations and preliminary results of an experimental program to determine the condensing heat-transfer coefficients and to investigate the modes of condensation occurring with alkali liquid metals. Corrosion information was obtained on synthetic sapphire crystals in a sodium atmosphere.

Sodium and rubidium were condensed on a 304 stainless steel U-tube using air as a coolant. Overall heat-transfer coefficients for rubidium varied from 5.7 to $20.6 \text{ Btu/hr-ft}^2\text{-}^{\circ}\text{F}$ and sodium from 6.1 to $24.1 \text{ Btu/hr-ft}^2\text{-}^{\circ}\text{F}$. The pressures for the sodium

ASD TDR 62-738 (Continued)

condensation varied from 0.7 to 44 psia and from 1.4 to 63 psia for rubidium condensation. Individual heat-transfer coefficients for the rubidium condensation ranged from 8.9 to 70.8 Btu/hr-ft²-°F. These are preliminary results in a low heat flux range.

ASD TDR 62-952

January 1963

SUBJECT: MEANS OF CREATING SIMULATED VARIABLE GRAVITY FIELDS FOR THE STUDY OF FREE CONVECTIVE HEAT TRANSFER

INVESTIGATOR: D. A. Kirk

ABSTRACT: Theories concerning the transport of heat by free convection predict the effect of various physical parameters. Experimental investigators have varied these parameters, except gravity, to determine the validity of these theories. To obtain a simulated gravity different from ground level, an additional force or acceleration must be imposed on the fluid mass. Possible methods, analyzed for creating this condition, were electromagnetic, electrostatic, free fall, airplane flight programs, and centrifugal actions.

Considering the time required to reach equilibrium in a free convection heat transfer experiment and the desirability of conducting experiments under equivalent reduced gravity fields, the electromagnetic body force was the most favorable method. An analysis has been made of the possible effects which might interfere with interpretation of the experimental results. It was concluded that an electromagnetic type experiment is feasible and that the effect of gravity on free convection heat transfer can be determined by this method.

ASD TDR 62-1048, Part I

January 1963

SUBJECT: CONDENSATION HEAT TRANSFER: Effect of Turbulent Disturbance on Film Condensation

INVESTIGATOR: J. Lee

ABSTRACT: A turbulent film condensation problem was devised. The simplifying assumptions, similar to that used by Nusselt were evoked with the exception that the effect of turbulent transports was incorporated in the form of an eddy diffusivity of Deissler's form.

The approximate series solutions corresponding to the first order effect of turbulence were obtained correctly reducing to the laminar case in which no turbulent influence existed. The range of validity of approximation is restricted to

$$(C_p \Delta T \lambda) P_r < 0.17$$

The effect of turbulent transports tends to increase the heat transfer coefficient for $Pr \geq 0.3$, which is attributable to the steeper temperature gradient at the wall caused by the enhanced turbulent heat energy transport. However, for $Pr \geq 0.1$, the converse prevails because the thicker condensate film which manifests the influence of the turbulent momentum transport more pronouncedly, giving a linear temperature distribution. With this approximate solution of restricted range of parameters, a qualitative comparison can be made with the previous works. However, it provides an estimate for the decrease in heat transfer coefficient for some experimental data.

ASD TDR 62-1088, Part I

January 1963

SUBJECT: MATERIALS RESEARCH FOR ULTRA-HIGH-SPEED BEARING
LUBRICANTS AND HEAT TRANSFER FLUIDS
INVESTIGATOR: K. R. Mecklenburg
CONTRACT: AF33(616)-6854, Midwest Research Institute
ABSTRACT: Lubrication experiments were run where ultra-high-speed bearings were lubricated with MIL-L-7808 and MLO 60-294 in an oil-air mist. A lightly loaded size 204 bearing ran for 378 hours with MIL-L-7808 at 350°F and 30,000 rpm. A bearing life of more than 800 hours was obtained with MLO 60-294 at 120° to 140°F and 50,000 rpm; but with 300°F and 50,000 rpm it was less than 9 hours.

Heat transfer properties associated with liquid metal condensation were investigated. Over-all heat transfer coefficients are given for condensing sodium and rubidium. The small boiler and other equipment used are described. A larger boiler is also described.

HYDRAULIC FLUIDS:

WADC TR 57-294, Part III

August 1962

SUBJECT: RESEARCH ON LIQUID METALS AS POWER TRANSMISSION FLUIDS
INVESTIGATOR: R. C. Kumpitsch, J. R. Granan
CONTRACT: AF33(616)-5917, General Electric Co.
ABSTRACT: A liquid metals test loop, consisting of a low-pressure flow control and monitoring loop, and a high-pressure, high-temperature liquid metals pump were constructed and tested. The low-pressure loop was debugged and tested at temperatures up to 550°F. A total operating time of 103 hours was accrued. Techniques for loop repair and maintenance were developed.

A two-staged, gear-type pump, rated at 3000 psi, 1000°F and one gallon per minute was designed, built, and tested. The pump was first operated with petroleum ether (Stoddard solvent) to determine its capability. A total of ten hours running was accumulated during which the pump generated 3000 psi and flows exceeding two gallons per minute at approximately half rated speed.

The pump was run for 3 1/2 hours on NaK-77 at pressures up to 640 psi and temperatures to 780°F. Rated flow was developed at half rated speed. Tests were terminated by failure. Limited data was obtained and over-all efficiency was calculated. Material testing NaK-77 to determine compatibility, friction and wear tests, and a literature survey were also conducted.

WADD TR 60-838, Part III

January 1963

SUBJECT: A CONTINUATION OF THE SYNTHESIS AND EVALUATION OF PYRAZINE DERIVATIVES FOR USE AS FUNCTIONAL FLUIDS IN HIGH TEMPERATURE AND OXIDATION ENVIRONMENTS
INVESTIGATOR: P. T. Kan, J. D. Behun, R. Levine
CONTRACT: AF33(616)-6749, Wyandotte Chemicals Corp.
ABSTRACT: Progress is reported on the synthesis and evaluation of pyrazine derivatives for use as functional fluids in high temperature and oxidation environments. High molecular weight 2,3-disubstituted- and 2,3,6-trisubstituted-pyrazine derivatives of the phenoxy, alkoxy, alkoxy, and amino classes of compounds were prepared in good yields from 2-chloro-3-methylpyrazine and 2-chloro-3,6-dimethyl-pyrazine. Starting from 2,5- and 2,6-dimethyl-pyrazines, and tetramethylpyrazine, a large variety of pyrazine derivatives containing straight and branched alkyl substituents were also prepared.

WADD TR 60-838, Part III (Continued)

Synthetic routes were developed for the synthesis of pyrazine-containing esters. These compounds were derived both from pyrazinoic acids and alkyl β -blocked alcohols and fluorine β -blocked alcohols, as well as from pyrazine-alkanols and carboxylic acids. Potential synthetic routes were proposed for the preparation of high molecular weight perfluoroalkylpyrazines and silicon-containing pyrazines. Based on these proposed routes, a number of fluorine-containing and silicon-containing intermediates were synthesized.

WADD TR 60-838, Part IV

February 1963

SUBJECT: A CONTINUATION OF THE SYNTHESIS AND EVALUATION OF PYRAZINE DERIVATIVES FOR USE AS FUNCTIONAL FLUIDS IN HIGH TEMPERATURE AND OXIDATION ENVIRONMENTS

INVESTIGATOR: J. D. Behun, P. T. Kan, W. G. Lajiness

CONTRACT: AF33(616)-6749, Wyandotte Chemicals Corp.

ABSTRACT: Progress is reported on evaluation of new pyrazine functional fluids for high temperature and oxidation environments. Structure-property relations are described.

Tests that distinguish between two types of thermal stability (kinetic and thermo-dynamic) are discussed. Screening for oxidation stability by a moderate (M) and severe (S) test utilizes air flow rate dependence of oxidation to discern candidates of superior oxidation stability.

Pyrazine fluids that possess good overall properties are singled out. These represent structural compromises combining the thermally and oxidatively stable pyrazine nucleus with less stable substituents to enhance low-temperature fluidity. Fluids have been realized with fluidity to -70°F combined with thermal stability up to 700°F and oxidation stability in the range of 500°F .

ASD TDR 62-268

July 1962

SUBJECT: HIGH TEMPERATURE (-40° TO 550°F AMBIENT) POWER TRANSMISSION FLUIDS

INVESTIGATOR: H. W. Adams

ABSTRACT: The derivation of a power transmission fluid formulation suitable for use over an ambient temperature range of -40° to $+550^{\circ}$ to $+600^{\circ}\text{F}$ was the object of this project. Research and development techniques, background information, laboratory data influencing the formulation, and pump test data are presented and discussed.

ASD TDR 62-268 (Continued)

The work described herein led to the selection of a fluid formulation based on a super-refined, deep dewaxed, paraffinic mineral oil as a suitable power transmission fluid for use over the -40° to $+550^{\circ}\text{F}$ temperature range. The fluid formulations mentioned in this report are limited to nonoxidizing environments in the $+450^{\circ}$ to $+600^{\circ}\text{F}$ range; however, they have good oxidative stability at $+350^{\circ}\text{F}$ and below.

ASD TDR 62-405

April 1962

SUBJECT: FIRE-RESISTANT, HIGH-TEMPERATURE HYDRAULIC FLUIDS
INVESTIGATOR: W. C. Hammann, et al
CONTRACT: AF33(616)-7458, Monsanto Research Corp.
ABSTRACT: The objective of this contract was to develop fire-resistant fluids suitable for use in aircraft hydraulic systems over the temperature ranges of -65° to 450°F and 400° to 800°F .

Apparatus to measure pour point, viscosity from -65° to 900°F , C.O.C. flash and fire point, autogenous ignition temperature, flammability at 1300°F , thermal stability and vapor pressure were installed and operated. A bunsen burner apparatus for flame velocity measurement of both liquids and gases employing a schlieren camera technique was designed and operated.

Flame velocity data are reported for 142 organic compounds from which contributor coefficients for numerous moieties have been derived.

Many model compounds in the classes of halo-carbons, phosphine oxides, aromatic and heterocyclics were synthesized and/or purified for the above physical measurements. Correlations of these data with structure were made.

ASD TDR 62-405, Part II

February 1963

SUBJECT: FIRE-RESISTANT HIGH TEMPERATURE HYDRAULIC FLUIDS
INVESTIGATOR: E. S. Blake, et al
CONTRACT: AF33(616)-7458, Monsanto Research Corp.
ABSTRACT: Synthesis and characterization of three partially fluorinated aliphatics, as model compounds for fire-resistant fluids useful at -65 to $+450^{\circ}\text{F}$ is reported. Work to establish degree of fluorination required for fire resistance and to attain useful fluid

ASD TDR 62-405, Part II (Continued)

properties is recommended. Synthesis and characterization of 14 partially fluorinated aromatic ethers is described. Without exception the partially fluorinated ethers are less thermally stable than their hydrogen parents, and this lower thermal stability, combined with inferior autogenous ignition temperature, fire point, flash point, and fluid properties suggests that this class holds little interest as fluids for the 0 to 700°F range. Characterization of a perfluorinated ether is in progress. Limited effort on heterocyclic-containing molecules is reported.

LUBRICANTS:

WADC TR 58-350, Part V

May 1962

SUBJECT: DEVELOPMENT OF GREASES FOR HIGH
SPEED BASS AND ROLLER BEARINGS
INVESTIGATOR: P.R. McCarthy, et al
CONTRACT: AF33(616)-7822, Gulf Research & Development Co.
ABSTRACT: The objective of this work was the development
of a grease capable of lubricating bearings for a minimum period of
500 hours at 600°F and at speeds between 20,000 and 45,000 rpm.

All greases, although prepared from the best fluids available currently and a wide variety of experimental and commercially available thickeners, failed to meet the minimum requirement for performance life. Of these greases, one composed of ammeline as the thickener and an inhibited silicone as the fluid gave the best life at 600°F and at speeds of 20,000, 30,000, and 45,000 rpm. Life under these conditions was 180, 45 and 18 hours respectively.

WADC TR 59-191, Part IV

February 1962

SUBJECT: RESEARCH ON HIGH TEMPERATURE
ADDITIVES FOR LUBRICANTS
INVESTIGATOR: J.O. Smith, G.R. Wilson
CONTRACT: AF 33(616)-7853, Monsanto Research Corp.
ABSTRACT: Tetraphenyltin and bis (p-phenoxyphenyl)
diphenyltin perform very well as antioxidants for polyphenyl ethers
at 600-650°F. in Bearing Rig test ratings. p-Bix (triphenylstannoxy)
benzene is a third member of the organotin compounds screened that
is an effective antioxidant for polyphenyl ethers. Other anti-oxidants
that are effective for polyphenyl ethers are the metal salts of N, N-
diphenyl-dithiocarbamic acid, triphenylbismuth and the oxides of
copper. The most outstanding EP additive, from among those
screened, for polyphenyl ethers is diphenyltin bis(trichloroacetate)
with an EP weld point > 720 kg. Testing a variety of lubricants in a
modified Shell Four Ball apparatus (modified to detect metal-to-metal
contact electrically) permits following the change from hydrodynamic
to boundary type lubrication with increase in loading. A tentative
mechanism is proposed to explain the results of controlled oxidations
of m- and p-bis(phenoxy) benzenes-oxidation of polyphenyl ethers is

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accompanied by bond breaking (phenyloxygen) and coupling or condensation of intermediate to form higher molecular weight compounds.

WADC TR 58-350, Part VI

April 1963

SUBJECT: DEVELOPMENT OF HIGH TEMPERATURE GREASE FOR HIGH SPEED BALL AND ROLLER BEARINGS.

INVESTIGATOR: P. R. McCarthy, et al

CONTRACT: AF 33(657)-8048, Gulf Research and Development Co.

ABSTRACT: The objective of this work was the development of a grease capable of lubricating bearings for a minimum period of 500 hours at 600° F and at speeds between 20,000 and 45,000 rpm.

A wide variety of materials were investigated as thickener components for greases. These included triazines, high-melting metal derivatives, dyes, pigments, inorganic and organic compounds, salts and substituted aryl ureas. Fluid components investigated were silicones, polyphenyl ethers, and several experimental compounds both alone and in combination with various additives. All of the experimental greases containing the above types of compounds failed to meet the minimum life requirement. Maximum life of approximately 200 hours at 600° F and 20,000 rpm was obtained with a grease composed of an inhibited silicone and a triazene compound (ammeline).

WADC-TR 59-191, Part V

December 1962

SUBJECT: RESEARCH ON HIGH TEMPERATURE ADDITIVES FOR LUBRICANTS

INVESTIGATOR: J. O. Smith, et al.

CONTRACT: AF 33(616)-7853, Monsanto Research Corp.

ABSTRACT: A new series of antioxidants, the nickel and cobalt chelates of N-phenyl-5-nitrosalicylimine, having superior antioxidant properties for the polyphenyl ether base stock (LRO-9) at 600° F were discovered this contract year. They are significantly better than the best organotin compounds discovered earlier.

WADC TR 59-191, Part V (Continued)

From among a variety of antiwear and EP additives screened, the highest weld point was obtained with trichloroacetic acid (>1000 kg).

From all the experimental data obtained from the mechanism studies on the oxidation of polyphenyl ethers, a consistent picture of the types of reactions occurring during oxidation is emerging. The first step in the oxidation is most probably a dehydrogenation to form phenyl-type radicals and the theoretical maximum quantity of water. Oxygenation of the products apparently does not occur until a later stage in the oxidation. Results of thermal decomposition of diphenyl mercury in the presence of tritium-labeled *m*- and *p*-bis (phenoxy) benzenes indicate that the most reactive hydrogens are located on the external rings of both isomers.

WADC TR 60-855, Part III

January 1963

SUBJECT: LUBRICATION BEHAVIOR AND CHEMICAL DEGRADATION CHARACTERISTICS OF EXPERIMENTAL HIGH TEMPERATURE FLUIDS AND LUBRICANTS

INVESTIGATOR: V. Hopkins, A. D. St. John, D. Wilson

CONTRACT: AF 33(616)-6854, Midwest Research Institute

ABSTRACT: Shear stability experiments for four fluids are described. Deep dewaxed mineral oil (MLO 60-294) degraded when pumped at 700°F. An ester of TMP (MLO 60-50) resisted degradation at 550°F and 600°F but degraded at 650°F. Phenyl methyl silicone (QF-258) seems to have insufficient lubricity for use at 400°F in a hydraulic pump. Bis (phenoxyphenoxy) benzene (MLO 59-692) performed satisfactorily in a hydraulic pump for 50 hr. at 400°F but the pump failed during warmup for a 500°F run. Bulk modulus data to 10,000 psig are given for MLO 60-294 from 68° to 600°F and for QF-258 from 68° to 700°F. The theoretical analysis of dynamic stability in the rolling-sliding apparatus is presented. Wear-life data at 700° and 1000°F are presented for seven solid film lubricants. Calibration of the high-pressure viscometer pressure sensor to 108,600 psig and the displacement transducer for a 0.50 in. stroke is discussed. Extreme pressure behavior of MLO 60-294 and MLO 59-692 at 165°, 400°, and 600°F is presented. Run-in at a light load increases seizure load. MLO 59-692 requires a longer run-in than does MLO 60-294.

SUBJECT: BASIC FACTORS IN THE FORMATION AND STABILITY OF NON-SOAP GREASES FOR HIGH TEMPERATURE APPLICATIONS

INVESTIGATOR: J. J. Chessick, et al.

CONTRACT: AF 33(616)-7120, Lehigh University

ABSTRACT: This report is concerned specifically with developing a fundamental background of knowledge concerning inorganic, non-soap grease dispersions such as that developed earlier for dispersions formulated with organic vehicles. The results of experiments designed to achieve this objective are described. These are of sufficient number so that some degree of validity for an assumption, most important to this program, that intimate inter-relationships exist between behavior of characteristics of non-soap greases formulated either with organic or inorganic liquids. The importance of proof of this is obvious. For one, the time-consuming investigations needed to understand, broadly, solid-organic vehicle systems need not be repeated with purely inorganic systems. Miscellaneous studies such as publication efforts, stability testing of systems containing components selected or modified using basic knowledge and routine evaluation of new components, selected by the Air Force, were conducted also.

SUBJECT: PROPELLANT LUBRICATION PROPERTIES INVESTIGATION

INVESTIGATOR: M. F. Butner, R. A. Byron

CONTRACT: AF 33(616)-8189, Rocketdyne

ABSTRACT: This final summary report presents results of bearing tests, gear tests, simple sliding friction and wear, and rolling contact screening test conducted, using a mixture of 50-percent unsymmetrical dimethylhydrazine and 50-percent hydrazine as the lubricant. Conclusions are drawn and recommendations made for future work in this area.

ASD TR 61-643

August 1962

SUBJECT: A STUDY OF THE INFLUENCE OF LUBRICANTS
ON HIGH-SPEED ROLLING-CONTACT BEARING
PERFORMANCE

INVESTIGATOR: L. B. Sibley, et al.

CONTRACT: AF 33(616)-7257, Battelle Memorial Institute

ABSTRACT: Lubrication processes at rolling contacts are being with a rolling-disk machine and x-ray system capable of measuring the dynamic oil-film thickness and deformation at rolling contacts. For low-viscosity lubricants operating under moderate conditions of load, speed, and temperature, measured film thickness correlated well with elasto-hydrodynamic lubrication theory including both the elastic deformation of the contact surfaces and the increase in viscosity of the lubricant under pressure. Measured film thickness decreased significantly below theoretical under increasing load, speed, viscosity, and ambient temperature, at times by as much as a factor of 40.

It is proposed that as the conditions of load, speed, and temperature at rolling contacts become more severe, flow properties of lubricants other than viscosity and pressure-viscosity become important in elasto-hydrodynamic lubrication. The data indicate that some form of reduced shear stress or "yielding" may occur in the high-pressure lubricant film at rolling contacts under these conditions.

ASD TR 61-643, Part II

September 1962

SUBJECT: A STUDY OF THE INFLUENCE OF LUBRICANTS
ON HIGH-SPEED ROLLING-CONTACT BEARING
PERFORMANCE

INVESTIGATOR: L. B. Sibley, et al

CONTRACT: AF 33(616)-7275, Battelle Memorial Institute

ABSTRACT: The thickness of the lubricant film and the shape of the elastically deformed surfaces at rolling contacts, as measured by an X-ray method, indicate that some non-Newtonian flow properties of lubricants may have important effects in rolling-contact lubrication. A high-pressure lubricant rheology device is being developed to measure these properties under simulated rolling-contact conditions. Several difficult equipment and instrumentation

ASD TR 61-643, Part II (Continued)

problems were solved to obtain reliable accurate shear-stress and shear-rate data on the rheology machine. However, a brief consideration of some remaining thermal problems in the rheology machine has led to the temporary abandonment of this experimental approach to obtaining realistic high-pressure rheology data on lubricants in favor of a proposed new approach to the analysis of the disk-machine data. The disk machine has been instrumented to obtain the traction or friction at the rolling contact over a range of known amounts of slip superimposed on the rolling. Initial traction data, together with an analysis for interpreting these data in terms of pressure-viscosity coefficients, indicates considerable promise for this rheological technique.

ASD TR 61-737

January 1962

SUBJECT: MATERIALS RESEARCH FOR LUBRICANTS
AND HEAT TRANSFER FLUIDS

INVESTIGATOR: K. R. Mecklenburg

CONTRACT: AF 33(616)-6854, Midwest Research Institute

ABSTRACT: Friction data are presented for copper on copper and for titanium on nickel for very low sliding speeds and light loads. The stick-slip apparatus used to obtain these data is described. Results are given for the initial part of an investigation in which the film conductance will be determined for liquid metals. A preprototype liquid metal boiler was operated at temperatures up to 1630° F with sodium. The design of a prototype liquid metal boiler for use in the next step of this work at temperatures up to 1800° F is described. Techniques used to handle sodium are outlined. Bearing life data are included for experimental greases run at high temperatures and speeds in the Pope Spindle. Screening runs with experimental grease made on the Navy Spindle at 350° F were not effective in selecting greases which would consistently run more than a few hours at high temperatures in the Pope Spindle. Wear scar data from the Four-Ball Wear Tester are given for experimental fluids.

ASD TDR 62-222

February 1963

SUBJECT: STUDY AND EVALUATION OF THE OXIDATIVE AND DEPOSIT FORMING PROPERTIES OF HIGH TEMPERATURE LUBRICANTS

INVESTIGATOR: H. L. Hepplewhite, E. A. Oberright

CONTRACT: AF 33(616)-8021, Socony Mobil Oil Co., Inc.

ABSTRACT: A high temperature Thin Film Oxidation Unit has been constructed and test repeatability and reproducibility have been established. This equipment offers a means of determining the maximum deposit-free temperature of operation of a lubricating oil under highly oxidative conditions for short contact times.

Six high temperature oils have been evaluated in this equipment and maximum deposit-free temperatures determined. Relative ratings based on cleanliness, developed by ASD from Bearing Rig and/or Engine Test data, are in agreement with test results at 575° - 600° F in the high temperature Thin Film Oxidation Unit.

ASD TDR 62-253

March 1962

SUBJECT: RESEARCH ON THE LUBRICANT FOR A MACH 4 CLASS ENGINE

INVESTIGATOR: B. D. Pfoutz, et al

CONTRACT: AF 33(616)-7893, Koppers Co., Inc.

ABSTRACT: The ASD Seal-Lubricant Machine was modified and installed for testing high temperature lubricants. Ultimately it will be expected to operate at a bulk oil temperature of 800° F and an air temperature of 1400° F. These conditions are being approached in a stepwise, multiphase program. For each temperature rise, a repeatable test method is developed and submitted lubricants tested. Such a repeatable test method was first developed for 300° F bulk oil temperature and 750° F air temperature. A method has now been developed for tests at 425° F bulk oil temperature and 1050° F air temperature, and testing of submitted lubricants is getting under way at these conditions.

ASD TDR 62-407

October 1962

SUBJECT: HIGH TEMPERATURE INORGANIC LUBRICANT STUDY
INVESTIGATOR: P. H. Stahlhuth
CONTRACT: AF 33(616)-6860, Sundstrand Aviation
ABSTRACT: The design and development of a high speed journal bearing test rig is described. Test results are presented for water and liquid potassium lubricated journal bearings. The test program included investigations of bearing torque, lubricant flow rate and stability limits for bearings operating in the laminar regime, through transition to turbulent flow and in the turbulent flow regime. Torque versus speed curves were obtained over a range of loads and bearing-journal clearance ratios. The results of the materials compatibility study which supplemented the bearing study are included.

ASD TDR 62-465

May 1962

SUBJECT: PROCEEDINGS OF THE AIR FORCE-NAVY-INDUSTRY PROPULSION SYSTEMS LUBRICANTS CONFERENCE
INVESTIGATOR: G. A. Beane, K. L. Berkey
CONTRACT: AF 33(616)-7223, Southwest Research Institute
ABSTRACT: This report is a compilation of papers presented at the Air Force-Navy-Industry Propulsion Systems Lubricants Conference held in San Antonio, Texas, on 15, 16, and 17 November 1960. The conference was attended by approximately 300 representatives of Industry, the Department of Defense, and other Government Agencies. A total of twenty-two papers were presented, providing a review of lubrication requirements for future propulsion missions and of current research programs pertinent to gas turbine engines, rocket engines, and flight vehicle power.

ASD TDR 62-541

June 1962

SUBJECT: LUBRICATION IN SPACE ENVIRONMENTS
INVESTIGATOR: R. L. Adamczak, et al
ABSTRACT: Solid, semi-solid, and liquid lubricants, hydraulic fluids, heat-transfer fluids, and novel lubrication techniques are discussed with respect to the current state-of-the-art and the

ASD TDR 62-541 (Continued)

future capabilities of these various materials and/or their application. The severe environmental conditions of space are compared against both the current and future state-of-the-art in the field of lubrication and energy-transfer media. Research efforts currently being pursued by the Nonmetallic Materials Laboratory of ASD to provide new and improved lubricants are described. A brief interpolation is also given of the overall lubricant picture with respect to space technology in terms of reliability and system design.

ASD TDR 62-574

August 1962

SUBJECT: FLUIDS (FUELS, LUBRICANTS, HYDRAULIC FLUIDS, AND ELECTRONIC COOLANTS) FOR HIGH TEMPERATURE APPLICATIONS

INVESTIGATOR: M. P. Dunnam

ABSTRACT: This report deals with the timely area of advanced fluids for high temperature applications. A review of the source and nature of high temperature fuels, lubricants, hydraulic fluids, and electronic coolants are given and the limitations of the more conventional fluids are discussed. Recent information on the types of advanced high temperature fuels, lubricants, hydraulic fluids, and electronic coolants and data on their properties are included.

ASD TDR 62-640

December 1962

SUBJECT: DETERMINATION OF WORKING FLUID LUBRICATION CAPABILITY IN JOURNAL BEARINGS

INVESTIGATOR: J. Hall, et al

CONTRACT: AF 33(616)-8328, Rocketdyne

ABSTRACT: A research program was conducted on journal bearings lubricated by the liquid-alkali metals, potassium, and rubidium. The objective was a 1500-hr endurance run on each fluid, speeds up to 36,000 rpm loads up to 100 lb., and lubricant temperature up to 1200° F. Performance studies were conducted using water, potassium, and rubidium lubricants; results are provided. A closed cycle gas system designed to provide turbine and support bearing operating gas for both liquid-metal systems was constructed; it operated at design temperatures.

ASD TDR 62-660

July 1962

SUBJECT: GREEN'S FUNCTION OF RADIAL DISPLACEMENT IN A CIRCULAR DISC DUE TO UNIT NORMAL AND TANGENTIAL LOADS

INVESTIGATOR: C. W. Ng

CONTRACT: AF 33(616)-8016, Rensselaer Polytechnic Institute

ABSTRACT: Using the method of Muskhelishvili, the Green's function of radial displacement is obtained in a circular disc due to a unit normal and a unit shear traction at the boundary. This Green's function is useful in the study of contact problems in sliding as well as in rolling, i. e., included would be the important problem of elasto-hydrodynamics.

An example is carried out for the distribution of heat generation due to a rigid rider against an elastic slider which is in the form of a circular disc.

ASD TDR 62-755

August 1962

SUBJECT: FACTORS GOVERNING THE FORMATION AND GROWTH OF ORGANIC THICKENER PARTICLES

INVESTIGATOR: J. J. Chessick, J. B. Christian

ABSTRACT: An extended series of preparation of various arylurea grease thickeners were conducted under widely different conditions. While the experimental techniques (because of the complexity of the systems studied) were not the ultimate in scientific precision, the resulting extensive data were utilized to determine the important parameters governing particle growth. Available theory was applied and recommendations advanced to aid in the production of organic, non-soap grease solids possessing desirable thickener properties.

The recommendations are applicable generally to particles which form as a result of "nuclei" formation followed by growth due to deposition of product molecules onto nuclei surfaces. This growth mechanism occurs frequently in practice.

ASD TDR 62-760

August 1962

SUBJECT: A SUMMARY OF THE BASIC FACTORS IN THE
FORMATION AND STABILITY OF NON-SOAP
GREASE GELS AND OTHER DISPERSIONS

INVESTIGATOR: J. J. Chessick, J. B. Christian

ABSTRACT: This report summarizes a research program of seven years duration conducted mainly at Lehigh University in cooperation with Aeronautical Systems Division. The major objective was to provide an understanding of the basic factors responsible for the formation and stability of non-soap lubricating greases to provide guides in the preparation of superior lubricants for use under extreme environmental conditions. General approaches to experimental procedures for all studies conducted are presented. Because of the large number of experimental procedures, detailed descriptions are not included. Published works revealing experimental details are referenced.

ASD TDR 62-815

September 1962

SUBJECT: INORGANIC THICKENED GREASES

INVESTIGATOR: J. B. Christian

ABSTRACT: Experimental inorganic thickened greases were synthesized and characterized using standard and specialized techniques. The greases were formulated from high melting inorganic solids, and polyphenyl ethers, high phenyl content silicones, inhibited silicones, and fluorosilicones. The thickening ability of several inorganic solids was determined. Many of the grease formulations show promise for such applications as anti-friction bearing lubricants, high temperature pneumatic greases, and lubricants for use in applications of sliding and rolling friction under heavy load conditions.

ASD TDR 62-828, Part I

October 1962

SUBJECT: FUNDAMENTAL INVESTIGATIONS ON THE
FRICTION AND WEAR OF SLIDING SOLIDS.
Initial Studies of Sliding Friction of Cu-Cu
System

INVESTIGATOR: T. Liu

ABSTRACT: A survey on the literature of the sliding friction of a copper-copper system was made together with an outline of the recent development of friction theory. Experimental measure-

ASD TDR 62-828, Part I (Continued)

ments are described of dynamic friction of 1/8 in. copper spheres sliding on copper flats. At the speed of 0.001 in/sec, the coefficient of friction of the initial path is relatively low (0.2) since the surface impurities may act as boundary lubricants. Upon repeated sliding, during portions of the path the friction tends to rise, either to a value of 0.4 or 1.1. If the sliding is allowed to continue, eventually the friction coefficient becomes 1.1 for the entire path. No satisfactory explanation for the existence of the intermediate value (0.4) is available based on present friction theories. This value, hitherto unreported, was found to disappear at higher sliding speeds.

ASD TDR 62-1079

February 1963

SUBJECT: EUROPEAN RESEARCH ON LUBRICATION
SPONSORED BY THE USAF NON-METALLIC
MATERIALS LABORATORY

INVESTIGATOR: T. Liu, R. Benzing

ABSTRACT: The work of five basic research contracts in the field of friction, lubrication, and wear is described. These contracts, sponsored by the USAF Nonmetallic Materials Laboratory, are spread throughout Europe

The wear of steel due to impacts was investigated as a function of load, speed, and number of impacts. The reaction rates between iron, copper, and aluminum with sulfur and stearic acid were found to increase after the metal specimens had been stretched. A mathematical analysis was carried out for the stress distribution of a rolling contact system lubricated with a non-Newtonian liquid. The surface reaction rates between iron and di-n-butyl monosulfide, di-n-butyl disulfide, and di-n-butyl trisulfide as well as elementary sulfur were investigated at various temperatures and atmospheric conditions. Theoretical estimation of the adhesion between solids was carried out and are being checked with experimental results obtained from ultracentrifuge techniques.

ASD TDR 62-1088, Part I

January 1963

ASD TDR 62-1088, Part I (Continued)

SUBJECT: MATERIALS RESEARCH FOR ULTRA-HIGH-SPEED BEARING LUBRICANTS AND HEAT TRANSFER FLUIDS

INVESTIGATOR: K. R. Mecklenburg

CONTRACT: AF 33(616)-6854, Midwest Research Institute

ABSTRACT: Lubrication experiments were run where ultra-high-speed bearings were lubricated with MIL-L-7808 and MLO 60-294 in an oil-air mist. A lightly loaded size 204 bearing ran for 378 hr. with MIL-L-7808 at 350° F and 30,000 rpm. A bearing life of more than 800 hr. was obtained with MLO 60-294 at 120° to 140° F and 50,000 rpm; but with 300° F and 50,000 rpm it was less than 9 hr.

Heat transfer properties associated with liquid metal condensation were investigated. Over-all heat transfer coefficients are given for condensing sodium and rubidium. The small boiler and other equipment used are described. A larger boiler is also described.

ASD TDR 63-80

March 1963

SUBJECT: EVOLUTION OF ROCKET ENGINE PROPELLANT - MATERIALS COMPATIBILITY TESTING

INVESTIGATOR: G. A. Beane, IV

ABSTRACT: A summary of the need for determining materials compatibility with rocket engine propellants, a discussion of various modes of reactions suspected of occurring in liquid rocket engines, and the various evaluation techniques considered for predicting materials compatibility performance in rocket engine firings are presented. Considerations in selecting a specific Impact Tester (ABMA type) are given as well as a historical review of its development. A summary of data on the compatibility of a few chemical fluid classes with liquid oxygen is presented. Recommendations are made for additional work in the area of evaluation methods refinement, materials compatibility determinations, and compatible materials development.

February 1963

SUBJECT: THERMOPHYSICAL PROPERTIES OF RUBIDIUM

INVESTIGATOR: F. Tepper, et al

CONTRACT: AF 33(657)-7659, NSA Research Corp.

ABSTRACT: Techniques for experimental determination of the thermophysical properties of rubidium are discussed, and literature surveys for each of the properties are included. The amalgamation procedure appears to be applicable to the analysis of oxygen in rubidium. The nitrogen content of rubidium was found to be < 2 ppm, while the carbon content of rubidium charge stock was 28 ppm. Vapor pressure data for rubidium in the range of 0.04 to 14.59 atmosphere was fitted to the equation:

$$\log_{10} P_{\text{atm}} = \frac{-7,005.20}{T^{\circ}\text{R}} + 4.04369$$

standard deviation = 1.26%

Density of liquid rubidium to 2000 $^{\circ}$ F is given by the equation:

$$g/cc = 1.55643 - 0.00026511(t(^{\circ}\text{F})) - \frac{6.26779}{t^{\circ}\text{F}}$$

standard deviation = 0.0035 g/cc

The C_p of liquid rubidium was determined from its melting point to 2000 $^{\circ}$ F, and was found to be constant at 0.0880 BTU/lb/ $^{\circ}$ F from 260 to 2000 $^{\circ}$ F. Progress in determination of the PVT diagram is discussed. Derived heat of vaporization values appear consistent with literature values.

May 1963

SUBJECT: BASE STOCK CHARACTERIZATION AND FORMULATION DEVELOPMENT FOR HIGH TEMPERATURE GAS TURBINE LUBRICANTS

INVESTIGATOR: R. E. Dolle

ABSTRACT: Phosphonitrile, pyrazine, triazine, polyphenyl ether, and ester candidate base stocks were characterized for -35 $^{\circ}$ to 500 $^{\circ}$ F gas turbine engine lubricant application. Formulations were developed utilizing new antioxidant systems in pyrazine and aromatic

ester base stocks. A phenyl α -naphthylamine and tetraphenyltin inhibitor system exhibited exceptional antioxidant properties in alkyl pyrazine fluids. N, N'-Bis(2-methoxybenzylidene)-p-phenylenediamine was found to be the most effective oxidation inhibitor in the aromatic ester, resorcinyl dineoheptanoate.

Revised micro-oxidation and oxidation-corrosion test procedures were developed to give more accurate and reproducible oxidative results.

All evaluation data has been tabulated showing the physical and chemical characteristics of the candidate fluids examined.

SOLID FILM LUBRICANTS

WADD TR 60-530, Part IV

January 1963

SUBJECT: PHYSICAL AND CHEMICAL PROPERTIES OF CERAMIC BONDED SOLID LUBRICANT FILMS
INVESTIGATOR: Lavik, et al
CONTRACT: AF 33(616)-6854, Midwest Research Institute
ABSTRACT: The wettability of glassy ceramics on metals and solid lubricants was studied in developing bonded solid lubricant films. Evaluation tests at temperatures up to 1500°F show improved performance for newer films. The wear of films was studied by electron microscopy and motion picture techniques. Some physico-chemical properties of lubricants were studied including oxidation, desorption and evaporation, crystal structure, and elastic properties.

WADD TR 61-49, Part II

March 1962

SUBJECT: A STATISTICAL ANALYSIS OF THE FRICTION-AL PERFORMANCE OF SOLID FILM LUBRICANTS. Ceramic Bonded Film in Air
INVESTIGATOR: M. R. Adams, M. D. Lum
ABSTRACT This report presents a statistical analysis of the performance of a ceramic bonded solid film lubricant. The ceramic bonded film, PbS/B_2O_3 in a six to one weight ratio of lubricant to binder, was developed by the Midwest Research Institute under contract with the Air Force and is of interest in the 700 to 1000°F temperature range. Experiments were conducted on the Hohman A-6 tester with two loading shoes. The analysis of variance shows that, within the range of the variables studied, the main effects of bearing load and ambient temperature are significant, and that a response to a change in temperature depends on speed.

The results are analyzed with an approximate and with an exact statistical method. The two methods lead to identical conclusions.

SUBJECT: LUBRICATION STUDIES WITH LAMELLAR SOLIDS
INVESTIGATOR: P. Bryant
CONTRACT: AF 33(616)-7823, Midwest Research Institute
ABSTRACT: A basic research program is being conducted to determine the mechanisms of friction and wear for solid lamellar lubricants.

This report proposes mechanisms of lubrication and wear for graphite and correlates the proposed models with some theoretical calculations and experimental results. A similar and more complete set of results was obtained for the lamellar material, mica. In addition, the rehealed cohesion energy of clean and air-contaminated mica lamellae has provided a basis for the proposed models by way of analogy.

The stress-etch process discovered with mica and reported in ASD-TDR-62-55, is the basis of the lubrication model.

SUBJECT: SOLID FILM LUBRICANT-BINDER PHENOMENA:
PbS-B₂O₃ SYSTEM
INVESTIGATOR: H. R. Thornton, et al
CONTRACT: AF 33(616)-7978, University of Illinois
ABSTRACT: The basic techniques, x-ray diffraction, microscopy, and fusion studies, along with the supplementary techniques of differential thermal analyses and friction and wear measurements, are described as related to the PbS-B₂O₃ system. Data indicated that PbS and B₂O₃ were the only crystalline phases expected in the majority of the specimens. A glassy phase exists between 300° and 1500° F. The lubrication mechanism in the high B₂O₃-low PbS mixtures is a function of the liquid phase present while the liquid phase only effects the low B₂O₃-high PbS mixtures above 980° F. Frictional compatibility is necessary between the lubricating pigment and binder over the entire temperature range.

SUBJECT: CERAMIC STRUCTURAL ADHESIVES AS
BINDERS FOR HIGH TEMPERATURE SOLID
FILM LUBRICANTS

INVESTIGATOR: B. D. McConnell

ABSTRACT: This report covers the initial effort by this Laboratory in the area of structural ceramic adhesives as bonding agents for solid film lubricating pigments in high temperature environments. A brief state-of-the-art in the use of ceramic materials as binders in lubricating systems is presented with emphasis on the problems encountered. Composition of the adhesives and pigments studied are given along with a description of the preparation and application of the resulting solid film lubricants. Friction and wear results are reported and discussed. The preliminary results to date indicate that the ceramic adhesives form a very good bond between the lubricating pigment and substrate bearing material, but the resulting film formulations suffer short wear life and fairly high friction. However, some degree of lubrication is obtained when friction and wear data from the coated specimens are compared to that of the bare metal to metal.

SUBJECT: RESEARCH ON HEAT AND MASS TRANSFER
EFFECTS IN SLIDING METAL SYSTEMS LUBRICATED BY SOLID INTERFACIAL FILMS

INVESTIGATOR: F. F. Ling

CONTRACT: AF 33(616)-8016, Rensselaer Polytechnic Inst.

ABSTRACT: An apparatus is described for bringing a rider specimen and a slider specimen into continuous sliding contact so that significant temperatures at the interface are achievable. The design is such that the flow fields of heat in the specimens would be at most two-dimensional, i. e. within engineering approximations; this fact makes possible the measurement of temperatures of the specimens without disrupting the flow fields of heat.

Data are presented of speed, normal load, frictional resistance, wear and temperatures at strategic locations on the specimens. Using the heat-equation solutions obtained specifically for the configurations concerned, contact-surface temperature distributions of both the specimens are calculated from experimental data. Results give the point-wise, temperature juncture condition at the interface for dry steel vs. steel, Linde LS-4 solid film vs. steel and Plasmacote flame coated MoS_2 solid film vs. steel.

Also, other sliding models are analyzed for interface temperature distributions.

Using the method of Muskhelishvili, the Green's function of radial displacement is obtained in a circular disc due to a unit normal and a unit shear traction at the boundary. This Green's function is useful in the study of contact problems in sliding as well as in rolling, i. e. included would be the important problem of elasto-hydrodynamics. An example is carried out for the distribution of heat generation due to a rigid rider against an elastic slider which is in the form of a circular disc

Finally, the design of an apparatus for measuring friction, wear and sliding temperature for use in vacua down to 10^{-9} torr is described. The apparatus is characterized by the fact that there are no supporting bearings other than the simple sliding surfaces to be studied within the vacuum chamber.

ASD TDR 62-449, Part II

December 1962

SUBJECT: SOLID FILM LUBRICANT-BINDER PHENOMENA:
CLEAVABLE MINERAL AND HALOGENATED
GLASS SYSTEMS

INVESTIGATOR: H. R. Thornton, J. F. Benzel, et al.

CONTRACT: AF 33(616)-7978, University of Illinois

ABSTRACT: Friction and wear characteristics were observed for the B_2O_3 -easily cleavable mineral and highly halogenated glass systems. A correlation between the number of cleavable directions and the friction and wear traits was noted for the cleavable minerals. The tri-directional cleaving minerals, anhydrite, halite, barite, and fluorite, demonstrated the best lubricating potential.

The highly halogenated glass area demonstrated good friction and wear properties over the temperature range 80° through $1500^\circ F$. Addition of oxide materials to the chloride glass increased the stability of the system. This area appears to be promising for the development of compositions which will provide sufficient lubrication to bearing members over the temperature range in question.

GRAPHITE

WADC TR 58-360, Part IV, OTS Release

June 1962

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

INVESTIGATOR: S. Mrozowski, et al

CONTRACT: AF 33(616)-7791, Carbon Research Laboratory

ABSTRACT: In continuation of work on the elastic properties of carbons, families of curves of the dependence of Young's modulus on temperature and on the heat treatment have been obtained for four basic types of carbons, using the shortly completed high temperature equipment. The decisive influence of filler type has been established. As a result of gained experience, improvements in the associated electronics were introduced. The amplitude dependence of the internal friction and dynamic Young's modulus were, for the first time, investigated at room temperature for vibrations in the sonic range of frequencies for several types of carbons. Reproducibility has been achieved in measurements of thermal conductivity with the new high temperature test apparatus and families of curves of the dependence of the conductivity on temperature and on heat treatment obtained for soft carbon. A new technique of determining the heat conductivity of carbons without internal heat generation has been tried and found to work up to 1800°C. Improved results were obtained with the transient state technique of determination of the heat diffusivity by using a new fast responding pyrometer and a new improved apparatus for the "steady" sinusoidal wave technique constructed.

WADD TR 60-143, Part III, OTS Release

April 1962

SUBJECT: RESEARCH STUDY TO DETERMINE THE PHASE EQUILIBRIUM RELATIONS OF SELECTED METAL CARBIDES AT HIGH TEMPERATURES

INVESTIGATOR: R. V. Sara, R. T. Dolloff

CONTRACT: AF 33(616)-6286, National Carbon Co.

ABSTRACT: The work here reported is the result of investigations of phase equilibria in the binary systems, tungsten-carbon and zirconium carbon. A completed phase diagram for the tungsten-carbon system is presented which differs significantly from the one proposed by Sykes in 1930 and which is generally accepted today. The system

WADD TR 60-143, Part III (Continued)

is characterized by three binary phases, W_2C , α and β WC. A new cubic phase (β - WC) has been discovered which is stable only above 2525°C. Eutectic temperatures of 2710°C and 2765°C are observed in the tungsten-rich and carbon-rich sides of W_2C , respectively. A tentative phase diagram for the zirconium-carbon is included which, in general, resembles several versions published in the literature. ZrC is the only phase observed in this system, and like most carbides with a face-centered cubic structure, has a broad solubility range. Solidus temperatures of 1860°C and 2850°C have been observed on the zirconium-rich and carbon-rich sides of ZrC, respectively. The data were obtained by high-temperature differential thermal analysis and classical quenching procedures.

WADD TR 60-143, Part IV

February 1963

SUBJECT: RESEARCH STUDY TO DETERMINE THE PHASE EQUILIBRIUM RELATIONS OF SELECTED METAL CARBIDES AT HIGH TEMPERATURES

INVESTIGATOR: R. Sara, C. E. Lowell, R. T. Dolloff

CONTRACT: AF 33(657)-8025, National Carbon Co.

ABSTRACT: The work summarized in this report is the result of investigations of phase equilibria in the binary systems; zirconium-carbon, tantalum-carbon and boron carbon. A completed phase diagram, which encompasses experimental results and considerations heretofore unreported in the literature, is presented for the zirconium-carbon system. A tentative phase diagram for the tantalum-carbon system is included, which, in general, resembles the version released by Ellinger in 1943. Preliminary results for the boron-carbon system, depicting maximum solubility in the graphite lattice below the eutectic temperature of approximately 2390°C, are also included.

WADD TN 61-18, Part II, OTS Release

July 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON ADVANCED GRAPHITE MATERIALS

INVESTIGATOR: R. M. Bushong, R. C. Stroup, et al

CONTRACT: AF 33(616)-6915, National Carbon Co.

WADD TN 61-18, Part II (Continued)

ABSTRACT: A review is given of activities over the period October 15, 1960 to October 15, 1961, on a three year United States Air Force-Department of Defense program for the research and development of materials, experimental techniques, and equipment for development of premium quality, reproducible graphite-base materials suitable for missile and astronautic applications.

Progress is reported on research and development in the study areas of raw materials, fabrication, and material characterization and evaluation.

WADD TR 61-72, Vol II, OTS Release

July 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON ADVANCED GRAPHITE MATERIALS

INVESTIGATOR: G. B. Spence

CONTRACT: AF 33(616)-6915, National Carbon Co.

ABSTRACT: Eshelby, Read, and Shockley's theory of dislocations in an anisotropic elastic continuum has been used to derive formulas not involving complex numbers for the stress-components of straight dislocations in certain symmetry directions. From these the dependence of stacking fault energy γ_f on the orientation of the Burgers vector and on the width of extended dislocations and triple partial ribbons and the dependence of γ_f on the radius of curvature of extended nodes have been calculated. The results are rigorous for hexagonal crystals and approximate for general directions in (111) planes of FCC crystals. -The theory is applied to graphite and close-packed metals. All three methods of determining γ_f for graphite yield results which are compatible with the value 0.6 ± 0.2 erg/cm². Several examples of the dependence of width of a dislocation on depth from a stress-free surface have been calculated.

WADD TR 61-72, Vol VI, OTS Release

June 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON ADVANCED GRAPHITE MATERIALS: Creep of Carbon and Graphites in Flexure at High Temperatures

INVESTIGATOR: E. J. Seldin

WADD TR 61-72, Vol VI (Continued)

CONTRACT: AF 33(616)-6915, National Carbon Co.
ABSTRACT: Flexural creep tests have been made on molded carbons and several types of graphites over the temperature range from 2200° to 3000° C. The amount of creep was found to decrease for a carbon as the temperature of graphitization increased and as the holding time at graphitization temperature increased for times up to four hours.

Activation energies were determined for several standard and experimental grades of graphite from the steady state creep rates. The activation energies ranged from 23 to 92 kcal/mole and appeared to be constant for each graphite. The flexure experiments demonstrated a marked dependence of the creep on the grain orientation for the more anisotropic graphites. The grain orientation showing the highest room temperature strength shows the lowest high temperature creep. Some of the factors which influenced the creep behavior are density, crystallite size, and the size, type and orientation of the particles.

WADD TR 61-72, Vol III, OTS Release

June 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON
ADVANCED GRAPHITE MATERIALS
INVESTIGATOR: E. A. Neel, A. A. Kellar, K. J. Zeitsch
CONTRACT: AF 33(616)-6915, National Carbon Co.
ABSTRACT: This report describes the mechanical and thermal properties of several types of ZT graphite. Grade ZTA is discussed in detail and its physical properties are compared to those of other premium graphites, including pyrolytic, over the range from room temperature to 3000° C. Specifications for Grade ZTA are established. The properties of other grades of ZT graphite and the results from 79 sub-scale firings in solid fuel rocket motors at various locations are tabulated.

WADD TR 61-72, Vol VIII, OTS Release

June 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON
ADVANCED GRAPHITE MATERIALS. VOL

VIII. ELECTRON SPIN RESONANCE IN
POLYCRYSTALLINE GRAPHITE

INVESTIGATOR: L. S. Singer, G. Wagoner
CONTRACT: AF 33(616)-6915, National Carbon Co.
ABSTRACT: The electron spin resonance of polycrystalline graphite is shown to be due to charge carriers, as for single crystals. For lampblack-base graphite, the variation in g-value with temperature agrees approximately with that for single crystal graphite if it is assumed that the spins effectively average out the anisotropic interactions by rapid intercrystallite diffusion. The magnitude and temperature dependence of the spin susceptibility are found to be in excellent agreement with McClure's theoretical predictions for the charge carriers in perfect graphite. Complications of measuring these materials are discussed, including effects of skin depth and microwave heating and the dependence upon crystallite size and sample purity. It is found that two types of impurities can affect the resonance; those which become ionized and shift the Fermi level, and large unionized atoms which do not shift the Fermi level but act as efficient scattering centers for shortening the spin-lattice relaxation time.

WADD TR 61-72, Vol X, OTS Release

August 1962

SUBJECT: RESEARCH AND DEVELOPMENT ON
ADVANCED GRAPHITE MATERIALS. Thermal
Reactivity of Aromatic Hydrocarbons
INVESTIGATOR: I. C. Lewis & T. Edstrom
CONTRACT: AF 33(616)-6915, National Carbon Co.
ABSTRACT: A general survey is given of the thermal reactivity of aromatic hydrocarbons to provide information basic to the understanding of the conversion of organic materials to carbon. Differential thermal analysis was employed on 84 aromatic hydrocarbons to delineate the thermal sequences during pyrolysis to 750°C. Absorption spectra, electron spin resonance, chromatography, and molecular weight determinations have been used to determine the mechanisms and structural changes which occur.

WADD TR 61-72, Vol XI, OTS Release

September 1962

SUBJECT: RESEARCH & DEVELOPMENT ON
ADVANCED GRAPHITE MATERIALS. Vol XI.
Characterization of Binders Used in the Fabrication of Graphite Bodies

INVESTIGATOR: E. de Ruiter, A. Halleux, V. Sandor, et al

CONTRACT: AF 33(616)-6915, National Carbon Co.

ABSTRACT: A series of chemical and physical methods, previously developed for use in the study of the constitution of coals, has been applied to the definition of structural parameters for thirteen tar and pitch samples which have a wide range of properties and which are of interest in the fabrication of graphite bodies.

Information obtained on the molecular size and composition indicates the presence of polycyclic compounds with a high degree of aromaticity. Evidence also points to the importance of large aromatic clusters.

ASD TDR 62-314, OTS Release

May 1962

SUBJECT: STUDY OF THE MECHANISM OF FAILURE OF
ROCKET MATERIALS AND MATERIALS
RESEARCH

INVESTIGATOR: Y. Baskin, D. C. Schell, W. K. Sumida

CONTRACT: AF 33(616)-7048, Armour Research Foundation

ABSTRACT: Rocket failure mechanisms in conjunction with the development of nozzle materials for solid propellant motors is discussed. Testing graphite in an oxy-acetylene torch and the effects of purity, density, orientation, and microstructure on erosion-resistance were evaluated. Erosion decreased with greater purity. New graphite materials were developed and evaluated including hot pressed compositions and impregnated bodies.

Suitability of ZrC as a nozzle material was evaluated. Incorporation of Ta and W fibers in ZrC results in considerable improvement in resistance to thermal spalling, although not sufficient to prevent cracking. Thoria-base bodies reinforced with W fibers were studied.

Ten promising materials were fabricated into rocket nozzles and tested in static motor stands. Erosion performance is presented along with analysis of failure mechanisms.

ASD TDR 63-195

March 1963

SUBJECT: PYROLYTIC GRAPHITE, ITS HIGH TEMPERATURE PROPERTIES

INVESTIGATOR: W. Bradshaw, J. R. Armstrong

ABSTRACT: A compilation and analysis of ambient and high temperature property data on pyrolytic graphite is presented. The origin and nature of the physical, thermal, and mechanical anisotropies are examined. The profound effects of process variables such as substrate characteristics, deposition temperature, gas pressure, and annealing temperature on material properties are analyzed in relation to the micro-structural variations produced. The advantages, as well as the limitations, imposed upon the design of components by the anisotropic nature of pyrolytic graphite are discussed, principally in regard to thermal stresses, with a view toward aiding the designer exploit the unique characteristics of pyrolytic graphite. An account is also given of the reaction mechanisms involved in the oxidation of the subject material. The conflicting viewpoints and experimental evidence supporting either the reaction controlled or the diffusion controlled rate of oxidation theories in the high temperature region are summarized.

It is concluded that the successful application of pyrolytic graphite requires an understanding of process variables, anisotropic properties, and design limitations.

ASD TDR 63-322

May 1963

SUBJECT: SYNTHESIS AND PYROLYSIS OF METAL ALKOXIDES AS POTENTIAL REFRACTORY OXIDE COATINGS FOR GRAPHITE

INVESTIGATOR: K. S. Mazdiasni, C. T. Lynch

ABSTRACT: The vapor phase deposition of hafnia and zirconia on graphite substrates by decomposition of the isopropyl and tertiary butyl alkoxides has been investigated. The infrared spectra of the isopropoxides of zirconium and hafnium have been obtained. Thorium isopropoxide infrared data have also been obtained and the spectra compared with titanium and aluminum isopropoxides. The tertiary butoxides show more promise for coating applications because of their higher vapor pressures. The vapor pressures and decomposition temperatures of the zirconium and hafnium butoxides have been

ASD TDR 63-322 (Continued)

studied. Preliminary data on the oxidation resistance to 1000°C of various grades of graphite coated by this method are reported. The results indicate a potential route to more protective refractory coatings than currently available.

PLASTICS

WADC TR 328, Part IV

November 1962

SUBJECT: HEAT RESISTANT LAMINATING RESINS
INVESTIGATOR: L. Gilman, et al.
CONTRACT: AF 33(616)-7853, Monsanto Research Corp.
ABSTRACT: Glass fabric-reinforced polyisocyanurate laminates derived from 4, 4' -diisocyanatodiphenylmethane and m-chlorophenyl isocyanate can now be reliably prepared with average flexural strength of over 50,000 psi at 500° F. These laminates, although thermally stable well above 500° F decompose during long-term exposure at temperatures of 550° F or above in air.

A copolymer of 4, 4' -diisocyanatodiphenylether and phenyl isocyanate has been developed that is approximately twice as resistant to oxygen attack above 500° F as the resin described above. Initial tests on a laminate made with this new resin have shown a marked improvement in strength retention after long-term exposure to 550° F and 600° F in air.

A study of the kinetics of mixed isocyanate trimerization reactions has indicated that the adding isocyanate controls the reaction rate in all steps, and that there is no one rate-controlling step in the trimerization. From this we conclude that the product distribution obtained in our resins can be controlled by varying the relative reactivities and concentrations of the monoisocyanate-diisocyanate comonomers.

WADD TR 60-125, Part II

June 1962

SUBJECT: THE EFFECTS OF HIGH VACUUM AND ULTRA-VIOLET RADIATION ON NONMETALLIC MATERIALS
INVESTIGATOR: N. E. Wahl, R. R. Lapp, F. C. Haas
CONTRACT: AF 33(616)-6267, Cornell Aeronautical Lab.
ABSTRACT: This study was concerned with the behavior of non-metallic materials exposed to simulated single and combined elements of space environment. The conditions simulated were those of atmospheric composition, pressure, temperature, and ultraviolet radiation.

WADC TR 53-373 Sup 10

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WADD TR 60-125, Part II (Continued)

The loss in weight and strength of three types of structural plastic laminates after exposure to vacuum and ultraviolet radiation intensities from two to six times that experienced outside the earth's atmosphere were determined.

Evaluation of the effect of vacuum ultraviolet on glass and plastic glazing materials indicated that the transmission of the glass was not affected. The transparent plastic materials yellowed, bubbled, and became translucent but showed little chemical degradation of the polymer chains by infrared spectroscopy.

A white polyurethane coating exposed to separate elements of heat, vacuum and ultraviolet radiation showed significant changes in weight, color, and spectral absorptivity in the range of 0.38 to 2.0 microns.

A method of determining the absorptivity and heat transfer characteristics of plastics while exposed to vacuum ultraviolet is described.

WADD TR 60-746

June 1962

SUBJECT: INVESTIGATION OF MECHANICS OF REINFORCED PLASTICS

INVESTIGATOR: Y. Stavsky, F. J. McGarry

CONTRACT: AF 33(616)-6280, Massachusetts Institute of Technology

ABSTRACT: A theory for the small bending and stretching of laminated aeolotropic plates has been found; bending and stretching are, generally, coupled and a cross-elasticity phenomenon occurs in the stress-resultants and couples relations. Some simple boundary value problems for rectangular plates were treated with the general eighth order system in cases where the bending-stretching and cross-elasticity effects vanish; this revealed that homogeneous plate theory can be used to analyze such specific cases. A second theory was established for the small bending of symmetrically laminated isotropic plates where transverse shear and normal stress deformations are taken into account. The experimental phase of this study investigated resin shrinkage effects on single filaments and on fabric-based laminates.

WADD TR 60-746 (Continued)

Helical buckling of encapsulated single filaments was produced by axial shrinkage of the resin; in laminates, extensive internal cracking took place as postcure temperatures increased. Additional flexural studies of laminates revealed glass-resin interactions under stressed conditions.

ASD TR 61-69, Part I

May 1962

SUBJECT: MECHANISM OF FRACTURE OF RIGID
POLYMERS. Environmental Stress Cracking
of Polyethylene

INVESTIGATOR: P. Hittmair, et al

CONTRACT: AF 33(616)-6222, Polytechnic Institute of
Brooklyn

ABSTRACT: The environmental stress cracking of polyethylene has been studied under a variety of experimental conditions in Section I of this report. The importance of polymer crystallinity and molecular weight has been assessed. Many of the variables such as thermal history of the sample, temperature of measurement, surface tension of the stress cracking agent, concentration of the stress cracking agent, and magnitude of the stress have been treated. The importance of the testing procedure has been emphasized, as well as the relationship of stress cracking in polymers to the same problem in non-polymeric systems. Some microscopic studies of stress cracking were also carried out. Section II describes the influence of radiation on the environmental stress cracking of polyethylene. The polymer was irradiated by Cobalt 60 γ -rays, and studied as a function of a variety of experimental conditions. The deleterious effects of oxygen were noted. In order to prevent oxidative deterioration, the free radicals created had to be terminated by a heat treatment in the absence of oxygen. Some procedures were developed in which polyethylene could be made resistant to environmental stress cracking.

WADD TR 61-252

June 1962

SUBJECT: GROWTH AND MECHANICAL PROPERTIES
OF FILAMENTARY SILICON CARBIDE
CRYSTALS

WADC TR 53-373 Sup 10

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WADD TR 61-252 (Continued)

INVESTIGATOR: L. A. Yerkovich, H. P. Kirchner
CONTRACT: AF 33(616)-7005, Cornell Aeronautical Lab.
ABSTRACT: In this investigation silicon carbide whiskers were grown under various experimental conditions. The strength and modulus of elasticity of several whiskers were determined at room temperature. Methods of heating the whiskers for high temperature measurements were investigated. The specific gravity of the whiskers has been inferred from measurement of the unit cell dimensions by x-ray diffraction patterns.

Silicon carbide whiskers were grown by pyrolysis of methyltrichlorosilane in hydrogen. In some cases, dense growths of whiskers from 1.2 to 1.5 centimeters in length and from 2 to 5 microns in diameter were observed. The longest whisker obtained, thus far, was 5 centimeters in length.

The tensile strength of these whiskers ranges from 100,400 to 1,650,000 psi. The elastic strain at failure varied from 0.41 to 1.10 per cent and the observed values of elastic modulus varied from 12,700,000 to 123,300,000 psi. These results indicate that silicon carbide whiskers can be strong, high modulus of elasticity materials. Much research remains to be done to improve the methods of measurement, and to define the conditions of measurement and the types of whiskers that will give the best performance as structural materials.

ASD TR 61-439, Part II

May 1962

SUBJECT: RESEARCH ON CRITERIA FOR PLASTIC ABLATION MATERIALS AS FUNCTIONS OF ENVIRONMENTAL PARAMETERS: Ablation Abstracts

INVESTIGATOR: V. M. Vila, D. Nilsson
CONTRACT: AF 33(616)-7401, Aerojet-General Corp.
ABSTRACT: Reports presenting ablation information and related technology have been compiled and abstracted. Ablation information abstracts of the various reports with author, corporate, and subject indexes are presented. The abstracted reports presented are believed to represent 35 percent of the available literature on the subject of ablation and related technology. Abstracts of 243 reports are presented.

ASD TDR 62-66

May 1962

SUBJECT: BEHAVIOR OF NON-METALLIC MATERIALS
IN THE SPACE ENVIRONMENT

INVESTIGATOR: G. P. Peterson

ABSTRACT: A review of data obtained on the effect of simulated space environments on various nonmetallic materials is presented. The environments considered individually and combined are pressure temperature and ultraviolet radiation. The materials of interest are reinforced plastics of various types, ultraviolet absorbers and transparent materials.

ASD TDR 62-260, Part I

June 1962

SUBJECT: REFRACTORY REINFORCEMENTS FOR
ABLATIVE PLASTICS. Synthesis and Reaction
Mechanisms of Fibrous Zirconium Nitride

INVESTIGATOR: R. L. Hough

ABSTRACT: The need for improved fibrous materials to reinforce ablative plastics used in high shear force environments has precipitated consideration of metal carbides, borides, and nitrides because of their high melting temperature, high degree of hardness, low density, moderate strength, and resistance to chemical attack. In this report, the synthesis of zirconium nitride filaments was successfully accomplished by the reaction of electrically heated zirconium wires with gaseous nitrogen. Diffusion kinetics of the reaction were examined theoretically and experimentally, and the diffusion-controlled synthesis technique developed is superior to conventional vapor plating for obtaining chemically pure filaments of small diameter. Low-melting eutectic phases in the filaments and their subsequent withdrawal by a helictitic growth process were also studied.

ASD TDR 62-312

May 1962

SUBJECT: AN ARC-IMAGING FURNACE FOR MATERIALS
RESEARCH

INVESTIGATOR: R. W. Farmer

ABSTRACT: An arc-imaging furnace suitable for high-temperature materials research is discussed. The basic design, operation, and calibration of the furnace is presented. The original furnace was modified by incorporating an irradiance timing system.

ASD TDR 62-312 (Continued)

Various controlled atmospheric sample chambers were constructed and installed.

Several suggestions are made for improving the furnace.

ASD TDR 62-746

April 1963

SUBJECT: CHARACTERIZATION OF CARBONIZED PLASTIC COMPOSITES IN HYPER-THERMAL ENVIRONMENTS

INVESTIGATOR: K. Burhard, et al

CONTRACT: AF 33(616)-7938, Avco Corp.

ABSTRACT: Carbonized reinforced plastics were characterized in selected hyperthermal environments. Two air arc heaters and an arcimaging radiation simulator were used to generate the high-temperature conditions, which involved air enthalpies from 3000 to 20,000 Btu/lb, convective heat fluxes from 140 to 270 Btu/ft²-sec, a radiative flux of 50 Btu/ft²-sec, stagnation pressures from 0.01 to 1 atm., and exposure times up to 120 seconds.

The materials investigated consisted of a porous, reinforced carbonaceous matrix with or without a low-temperature ablative impregnant. They possessed remarkable dimensional stability during exposure. Performance limitations were found to include moderate insulative ability per unit weight, oxidation susceptibility, and possibly low mechanical strength.

ASD TDR 62-747

February 1963

SUBJECT: HIGH MODULUS, HIGH STRENGTH, HEAT RESISTANT REINFORCEMENTS

INVESTIGATOR: A. R. Morrison, R. Wong, W. C. Brady

CONTRACT: AF 33(616)-7950, Owens-Corning Fiberglass Corp.

ABSTRACT: A program to optimize the properties of and develop fiberization techniques for X-994 glass resulted in the formation of fibers having an average tensile strength of 650,000 psi at

ASD TDR 62-747 (Continued)

room temperature and 123,000 psi at 1500^oF. Fiberization techniques were developed to the extent that X-994 fiber can now be produced on a semi-commercial basis. A program to optimize the high temperature properties of X-815 glass was discontinued after considering the problems involved and the erratic data obtained from the fibers produced.

Inorganic surface treatments were found to improve the tensile strength and abrasive resistance of X-994 and E glass fibers at elevated temperatures. These treatments also gave some protection to fibers from the corrosive attack of inorganic matrices. The effect of a series of proprietary treatments on the properties of YM31A, X-994, and E glass fibers were determined.

ASD TDR 62-1028, Part I

February 1963

SUBJECT: PROPERTIES AND CHARACTERISTICS OF
ABLATIVE PLASTIC CHARS: Organic Fiber-
Reinforced Plastics

INVESTIGATOR: S. A. Marolo

ABSTRACT: The char layer region of cotton- and nylon-
fiber-reinforced phenolic specimens which had been exposed to
high heat flux in a plasma arc was investigated for physical,
chemical, and mechanical characteristics. The objectives were to
increase understanding of ablation phenomena and to correlate char
layer characteristics with ablation performance.

The most significant finding was that of
secondary pyrolysis of initial gases of decomposition, resulting in
deposition of carbon on the walls of the cellular char structure.

ASD TDR 62-1043, Part I

February 1963

SUBJECT: THERMOGRAVIMETRY OF PLASTICS:
Empirical Homogeneous Kinetics

INVESTIGATOR: R. W. Farmer

ABSTRACT: The thermal decomposition of fabric reinforced
phenolics, silicones and phenylsilanes in air and inert atmospheres

ASD TDR 62-1043, Part I (Continued)

was examined with recording thermobalances. Weight loss was continuously measured for powdered samples while heating at a constant rate of temperature rise to 900°C. Assuming the complex decomposition processes to be grossly analogous to classical pseudo-order homogenous kinetics, empirical rate parameters were determined from the recorded thermograms. Observed weight loss was compared to the predicted thermal decomposition of the materials.

ASD TDR 63-56

January 1963

SUBJECT: SYNTHESIS OF NEW ABLATIVE PLASTICS
FOR ROCKET NOZZLES

INVESTIGATOR: N. Bilow, L. J. Miller

CONTRACT: AF 33(657)-9176, Hughes Aircraft Co.

ABSTRACT: The synthesis and formulation of new ablative plastics and composites were conducted to obtain improved ablative materials for use in rocket exhaust environments. Emphasis was placed on the preparation of promising novel polymeric systems from the following general classes: P-phenylphenol Copolymers, Biaryl-substituted phenols, Polynuclear phenolic analogues, Phenoxy resins, and polyphenylenes. New ablative compositions were screened for desirable performance characteristics in three plasma environments: (a) pure nitrogen; (b) nitrogen plus oxygen; and (c) nitrogen plus entrained alumina powder. Several small scale rocket nozzles were fabricated from the most promising composites.

ASD TDR 63-344

May 1963

SUBJECT: DEVELOPMENT OF A MINIATURIZED
TENSILE TEST PROCEDURE FOR PLASTIC
LAMINATES

INVESTIGATOR: W. M. Powers

CONTRACT: AF 33(657)-10683, University of Dayton
Research Institute

ABSTRACT: This report presents a miniaturized-tensile test method for evaluating strength properties of glass-reinforced plastics. The test specimen and method outlined were designed with the purpose

ASD TDR 63-344 (Continued)

of providing the investigator with one simplified test which would require a minimum of material to evaluate the potential of experimental polymers as binders in reinforced plastics. Room temperature tests were conducted on phenolic, phenylsilane, heat resistant polyester, and silicone laminates. Based on the data obtained, the 45°-to-the-warp tensile test is recommended for the initial evaluation of newly developed polymers and resin systems.

EROSION

ASD TR 61-307

August 1961

SUBJECT: THERMAL EROSION OF ABLATIVE
MATERIALS

INVESTIGATOR: D. L. Robbins

CONTRACT: AF 33(616)-6285, Aerojet-General Corp.

ABSTRACT: The erosion of ablative rocket nozzle specimens exposed to rocket nozzle environments in three sub-scale test facilities were examined. Most of the specimens were investigated using a 150-lb-thrust gaseous-oxygen, gaseous-hydrogen rocket engine operating at 500-psia chamber pressure and 5500° theoretical flame temperature. Other specimens were examined using the same facility, with the fuel modified by the introduction of powdered aluminum. Two specimens were examined in a motor, which burned a thixotropic slurry-type propellant containing suspended aluminum particles. Two other specimens were investigated using an aluminized solid propellant.

POLYMERS

ASD TR 55-221, Part VIII

May 1962

SUBJECT: RESEARCH ON SYNTHESIS OF THERMALLY STABLE FLUORINATED POLYMERS

INVESTIGATOR: D. Knutson

CONTRACT: AF 33(616)-7963, Hooker Chemical Co.

ABSTRACT: The perfluoroalkyltriazine elastomer is prepared in two steps. Perfluoroglutaronitrile (1 mole) is reacted with perfluorobutyramidine (2 moles) to form an adduct. The adduct is further reacted with perfluoroglutarimidine to yield a gel that can be milled to form an elastomer. The elastomer had tensile strengths of 200 to 600 psi with 50 to 100 percent elongation. After aging 5 hours at 800 °F, the elastomer had weight losses of 18 to 20 percent. At elevated temperatures the elastomer has very little strength.

The structures of the perfluoroglutaronitrile-perfluorobutyramidine adduct and related nitrile-amidine adducts are discussed.

WADC TR 57-126, Part VI

July 1962

SUBJECT: PHOSPHINOBORINE POLYMERS

INVESTIGATOR: R. Wagner, A. B. Burg

CONTRACT: AF 33(616)-7810, American Potash & Chemical Corp., U. of Southern California

ABSTRACT: In continuing research toward the development of thermally stable polymers based on the boron-to-phosphorus bond system, experiments were conducted to find conditions suitable for preparation of glass cloth laminates from selected borophanes, to explore the effect of substituents on formation of linear borophane polymers, and to synthesize cyclic triborophanes having active functional groups. Phosphonyl azides and phosphonimidophosphoranes have been found to have unusual thermal stability. Infrared data are providing information bearing on the stability of boron-phosphorus polymers. The phosphinic anhydride based polymer system and that based on cyanophosphinediborane are under investigation.

WADC TR 53-373

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WADC TR 57-126, Part VII

January 1963

SUBJECT: PHOSPHINOBORINE POLYMERS
INVESTIGATOR: R. Wagner, A. B. Burg
CONTRACT: AF 33(616)-7810, American Potash & Chemical Corp.

ABSTRACT: A glass cloth laminate was prepared using the resin, polymethylborophane, and although it had a high heat distortion temperature it was too brittle for practical applications. Copolymer laminates of this resin with poly-P-methylsesquimethyleneborophane resin with or without epoxy resin showed high heat distortion temperatures, good adhesion, and toughness. A number of new functionally P- and B-substituted triborophanes have been prepared for use as intermediates in the syntheses of new resin systems. Acetoxyposphines were studied for their possible dissociation to $(CF_3PO)_n$ polymers. A series of new intermediate methyltrifluoromethylphosphine derivatives has been prepared and used in the synthesis of the triborophane, $(CH_3CF_3PBH_2)_3$, the infrared spectrum of which has been compared with related triborophanes. Borane adducts of cyanophosphines polymerize to give products which appear to be cross-linked borazine rings.

WADC TR 58-382, Part VI

February 1963

SUBJECT: SYNTHESIS AND EVALUATION OF NEW
POLYMERS PREPARED BY STEREOSPECIFIC
CATALYSIS

INVESTIGATOR: C. G. Overberger
CONTRACT: AF 33(616)-6866, Polytechnic Inst. of Brooklyn
ABSTRACT: In attempting to prepare thermally stable Diels-Alder ladder polymers and thermally stable cyclo polymers containing sulfur the attempted polymerization of dibenzylidene succinic anhydride and diisopropylidene succinic anhydride has failed whereas poly s, s' divinyl dithioformal has been prepared while divinyl dithioformal failed to polymerize.

WADD TR 59-95, Part IV

March 1962

SUBJECT: RESEARCH ON SYNTHESIS OF 1000°F STABLE
BASE FLUIDS

WADD TR 59-95, Part IV (Continued)

INVESTIGATOR: J. W. Dale, A. A. Hyatt, E. A. McElhill, et al
CONTRACT: AF 33(616)-7853, Monsanto Research Corp.
ABSTRACT: This contract aimed at the synthesis of base fluids stable at 1000°F, effort centered almost entirely on perfluoro-cyclic structures. The primary target was to have been synthesis of low polymers of the perfluoro-aromatic series $C_6F_5 (X-C_6F_4)_n$ $X-C_6F_5$ where $X = -O-$ or $-(CF_2)_n-$, with isolation of their fully saturated perfluoro-cyclohexyl analogues in a state of high purity as intermediates in one approach to their synthesis.

However, because of a change in emphasis, considerable time was spent on a process for synthesizing C_6F_6 via C_6F_{10} derived from the electrochemical fluorination of benzoyl fluoride. With further work to ensure reliable Simons cell operation, this process could offer several advantages over the route involving the CoF_3 fluorination of benzene. Defluorination of the crude C_6F_{10} obtained can also yield C_6F_5H and the $C_6F_4H_2$ isomers as valuable intermediates. A quantity of 1,4-dibromo-2,3,5,6-tetrafluorobenzene was prepared by a stepwise synthesis route largely avoiding use of the Schiemann reaction.

WADC TR 59-272, Part IV

July 1962

SUBJECT: THERMALLY STABLE PERFLUORINATED POLYMERS
INVESTIGATOR: H. C. Brown
CONTRACT: AF 33(616)-7971, University of Florida
ABSTRACT: The study of intermediate polymer formation from perfluoroalkyl dinitriles and perfluoroalkylamidines has been extended to include perfluorocaprylamidine, perfluoroacetamidine and perfluoroadiponitrile as monomers.

Study of the N' (perfluoroacylimidoyl) perfluoroalkylamidines, the initial intermediates in the deammonation of perfluoroalkylamidines to sym. triazines, has been extended in an effort to formulate the second (ring closing) step in this reaction. Possible mechanisms are discussed for the reaction of perfluoroalkylnitriles with the acylimidoylamidines. An alternative ring closure by treatment of the acylimidoylamidines with perfluoroacyl

WADC TR 59-272, Part IV (Continued)

chlorides is described.

Catalytic cotrimerization of perfluoroacetonitrile with several aromatic nitriles is described.

The initial work on another perfluoroalkyl-heterocycle system in which 1, 3 bis (5-perfluoropropyl-1, 3, 4-oxadiazolyl) perfluoropropane was prepared as a model compound is presented.

WADC TR 59-469, Part III

May 1962

SUBJECT: INVESTIGATION OF ORGANIC SEMI-CONDUCTORS
INVESTIGATOR: S. Aftergut, G. P. Brown
CONTRACT: AF 33(616)-8293, General Electric Co.
ABSTRACT: The temperature dependence of the electrical resistance was obtained on polymers and simple substances including poly (m-phenoxy)ene, poly (p-phenylene sulfide), a P-dimethylborophane polymer, a polybenzimidazole, acridone, diphenylguanidine, carbanilide, and phenothiazine. The resistance versus temperature of imidazole was measured in a zone-refining tube; the energy gap changed on zone-refining but became constant after 39 passes. New (apparently polymeric) complexes of imidazole and copper, zinc, and cobalt were prepared. The recent literature on organic semiconductors was surveyed.

ASD TR 61-1, Part II

March 1962

SUBJECT: THE PREPARATION OF ORGANOMETALLIC DERIVATIVES OF INORGANIC "BENZENOID" COMPOUNDS
INVESTIGATOR: D. Seyferth, M. Takamizawa, H. Yamazaki
CONTRACT: AF 33(616)-7124, Mass. Inst. of Tech.
ABSTRACT: Selected reactions of B-trivinyl-N-triphenylborazine have been investigated. Radical additions of bromotrichloromethane, carbon tetrabromide, benzenethiol and hydrogen bromide were accomplished using benzoyl peroxide as initiator. Crystalline

products could not be obtained from analogous reactions of carbon tetrachloride and dimethylphosphite with the vinylborazine. Addition of dimethylchlorosilane and methyldichlorosilate to B-trivinyl-N-triphenylborazine occurred readily in the presence of chloroplatinic acid, and methylation of B-tris (dimethylchlorosilylethyl)-N-triphenylborazine gave the corresponding trimethylsilylethyl derivative. Phenyllithium in ether completely disrupted the vinylborazine, triphenylborane and aniline being produced.

The first carbon-bridged bis-borazine, 1, 4-bis (B-bis(trimethylsilylmethyl)-N-trimethyl-(B)-borazyl)butane, was prepared by the reaction of the di-Grignard reagent of 1, 4-dichlorobutane with B-chloro-B-bis(trimethylsilylmethyl)-N-trimethylborazine. This bis-borazine was unstable toward hydrolysis, and its thermal stability was much lower than that of B-tris(trimethylsilylmethyl)-N-trimethylborazine.

The complete hydroboration of trimethylvinylsilane and allyltrimethylsilane, as well as the partial hydroboration of trimethylvinylsilane, have been studied. Only one direction of addition is observed with the allylsilane, giving the $(CH_3)_3SiCH_2CH_2CH_2B$ system, but with trimethylvinylsilane both possible directions of addition are observed. Particularly intermediates of the type $RBCl_2$ preparable by such procedures will be of interest in connection with further borazine syntheses.

The following new compounds were prepared during the course of this work:

- B-tris(1-bromo-3, 3, 3-trichloropropyl)-N-triphenylborazine
- B-tris(1, 3, 3, 3-tetrabromopropyl)-N-triphenylborazine
- B-tris (β -phenylthioethyl)-N-triphenylborazine
- B-tris (β -bromoethyl)-N-triphenylborazine
- B-tris (β -dimethylchlorosilylethyl)-N-triphenylborazine
- B-tris (β -methyldichlorosilylethyl)-N-triphenylborazine
- B-tris (β -trimethylsilylethyl)-N-triphenylborazine
- B-chloro-B-bis(trimethylsilylmethyl)-N-trimethylborazine
- 1, 4-bis(B-bis(trimethylsilylmethyl)-N-trimethyl-(B)-borazyl)butane
- Tris (γ -trimethylsilylpropyl)borane

ASD TR 61-1, Part II (Continued)

mixed α - and β -trimethylsilylethyldimethoxyborane
mixed α - and β -trimethylsilylethyldichloroborane
1, 3, 3, 3-Tetrabromopropyltrimethylsilane
3, 3, 3-Tribromo-1-propenyltrimethylsilane
1, 8-Dichloro-2, 2, 7, 7-tetramethyl-2, 7-disilaoctane
1, 9-Dichloro-2, 2, 8, 8-tetramethyl-2, 8-disilanonane

ASD TR 61-1, Part III

March 1963

SUBJECT: THE PREPARATION OF ORGANOMETALLIC
DERIVATIVES OF INORGANIC "BENZENOID"
COMPOUNDS

INVESTIGATOR: D. Seyferth et al

CONTRACT: AF 33(657)-8532, Mass. Inst. of Tech.

ABSTRACT: Addition of Si-H containing siloxanes, of tin hydrides and of diphenylphosphine to B-trivinyl-N-triphenylborazine is described. Reaction of B-(β -chlorosilylethyl)-borazines with lithium trimethylsilanolate and cohydrolysis of these silylborazines with chlorosilanes did not serve well in the preparation of siloxanylborazines. Attempts to prepare $(\phi_2P(O)CH_2BN\phi)_3$ and $(\phi_2PBN\phi)_3$ were unsuccessful. The preparation of $(HC\equiv CBN\phi)_3$ is described. A new synthesis of diphenylphosphine is reported.

The reduction of substituted gem-dibromocyclopropanes to monobromocyclopropanes can be effected in good yield with tri-n-butyltin hydride at temperatures below 40°. This radical reaction gives a mixture of isomers in most of the cases where one has the possibility of forming cis and trans isomers. The use of such bulky cyclopropyl bromides as intermediates in the synthesis of hydrolytically stable borazines may be of value.

ASD TR 61-2, Part III

January 1963

SUBJECT: SYNTHESIS OF INORGANIC POLYMER HIGH
TEMPERATURE FLUIDS

INVESTIGATOR: G. M. Nichols

CONTRACT: AF 33(616)-7158, E. I. du Pont de Nemours & Co.

ABSTRACT: Continued investigation of phosphonitriles

has emphasized aryloxy cyclic phosphonitriles and linear phosphonitrilic polymers stabilized against thermal polymerization by bonding to various metal halides. Aryloxy phosphonitrile liquids with potential utility from 10° F. or lower to 750° F. or higher were prepared. A systematic study of factors leading to improved low-temperature fluidity was initiated. Phosphonitrilic chloride polymers were stabilized against thermal polymerization by bonding to BiCl_3 and CoCl_2 but not by NiCl_2 . Synthesis of linear phenyl phosphonitrile polymers end-capped with FeCl_3 or ZnCl_2 was unsuccessful. Oxidation stability tests indicated that FeCl_3 -stabilized phosphonitrilic chloride polymers have oxidation stability to at least 600° F. Preparation of cyclic and linear $(\text{PNF}_2)_n$ polymers was studied under a variety of conditions.

May 1962

SUBJECT: RESEARCH AND DEVELOPMENT OF HIGH TEMPERATURE ORGANO-PHOSPHORUS COMPOUNDS

INVESTIGATOR: C. F. Baranauckas, R. D. Carlson, et al

CONTRACT: AF 33(616)-7963, Hooker Chemical Corp.

ABSTRACT: A number of symmetrical and unsymmetrical triarylphosphine oxides and sulfides have been prepared, using in general, a Grignard Reaction. Thermal testing at 450° C - 500° C in sealed tubes has been carried out to measure the hydro-carbon(s) formed by thermal decomposition. The oxides are shown to be considerably more stable than the sulfides. The more stable compounds tested contain phenoxy or fluoro groups, in a para position.

Certain heterocyclic systems, based on dibenzophosphole and phenoxaphosphine, show exceptionally good thermal stability, exhibiting less than 0.2% hour decomposition at 500° C.

Some conclusions as to decomposition mechanism and effect of ring substitution are advanced.

May 1962

SUBJECT: MOLECULAR STRUCTURE AND PHYSICAL BEHAVIOR OF POLYMERS

INVESTIGATOR: M. Morton, A. Gent, S. Gadkary, et al.

CONTRACT: AF 33(616)-6965, University of Akron

ABSTRACT: Model branched polymers have been prepared by the reaction of relatively monodisperse polystyryl lithium with silicon tetrachloride. A study of the dilute solution properties of the branched molecules has confirmed the theoretical development of the branching coefficient, g' , by Zimm and Kilb. The second virial coefficient, A_2 , has been found to decrease with increased branching. In addition, the Huggins constant, k' , has been found to be a poor measure of branching in molecules. A study has been carried out on the effect of polymerization temperature and age of active anionic polystyrene on its molecular weight. Results to date have shown that, at least up to 70°C, neither polymerization temperature nor polymer aging (up to six weeks) has any appreciable effect on the polymer, as indicated by intrinsic viscosity measurements. Furthermore, analytical tests failed to detect the presence of equilibrium monomer at 70°C, at least down to 0.5% concentration, based on polymer.

Stress relaxation measurements have been carried out at room temperature using vulcanizates of butyl rubber and natural rubber. Some measurements have also been made over a wide temperature range with vulcanizates prepared from fractionated butyl rubber.

February 1963

SUBJECT: MOLECULAR STRUCTURE AND PHYSICAL BEHAVIOR OF POLYMERS

INVESTIGATOR: M. Morton, A. Gent, S. D. Gadkary

CONTRACT: AF 33(616)-6965, University of Akron

ABSTRACT: Crosslinked polyethylene films have been prepared by gamma irradiation of molten linear polyethylene in vacuo. The stress-temperature relations at constant length have been determined, both for these materials and for crosslinked balata (trans-polyisoprene). The stress changes under isothermal conditions have also been studied. Dilatometric studies have been

ASD TR 61-16, Part III (Continued)

started. Evidence for folded-chain or oriented-bundle crystallization is afforded by the stress changes. The former seems less pronounced in balata; it is eliminated by incomplete melting.

ASD TR 61-22, Part II

October 1962

SUBJECT: POLYMER STRUCTURES AND PROPERTIES
INVESTIGATOR: T. A. Orofino, et al.
CONTRACT: AF 33(616)-6968, Mellon Institute
ABSTRACT: Theoretical studies of the thermodynamical configurational properties of polymers in dilute solution are described. Experimental results are reported on the effects of chain branching in both good and poor solvents and on specific solvent effects on chain dimensions. Refinements have been made in the design of a light scattering photometer, and calibration procedures have been critically evaluated. Melt viscosities of mixtures of polymer fractions appear to depend on the viscosity average molecular weight in a theta solvent rather than a higher molecular weight average. New characterization data have been obtained for the vinyl acetate polymer from emulsion polymerization in the presence of protein. Swelling studies of crosslinked polyethylene indicate no appreciable specific solvent effects for this polymer.

WADD TR 61-79, Part III

November 1962

SUBJECT: RESEARCH ON PHOSPHORUS-CONTAINING AND OTHER INORGANIC AND SEMI-ORGANIC POLYMERS
INVESTIGATOR: I. B. Johns, C. N. Matthews, M. L. Nielsen
CONTRACT: AF 33(616)-7853, Monsanto Research Corp.
ABSTRACT: A program involving several different approaches to thermally stable polymers was continued. Phosphonium polymers (MW 2000) were made from dibromomethane and methylene-bis(diphenylphosphine) in dioxane. Brown-black solid polymers that apparently cyclize from an initial ladder structure when heated to 500°C were formed through cyanohalocarbene intermediates. Novel inner salt complexes formed a polymer that was stable to 500°C after initial heating to 250°C. Dehydrohalogenation or dehalogenation also produced polymers from haloacetamides.

WADD TR 61-79, Part III (Continued)

High pressure condensation of (1) cyanodiarylphosphines, (2) mixtures of cyanodiarylphosphines with cyanodiarylboranes, and (3) perfluoroglutarodinitrile produced stable polymers. The perfluoroglutaronitrile polymerized smoothly through a liquid, to a rubber, and finally to a hard solid stable in glass to at least 400°C. Improved syntheses for starting materials were developed.

Phosphorus amides representing four classes were synthesized and studied as models of potential structures for highly stable polymers. Transamidation was studied as a method for phosphorus amide polymer synthesis. Feasibility of using bis(1-pyrrolyl)phenylphosphine oxide for transamidative polymer synthesis with the strongly basic diamine, 5, 5'-bibenzimidazole was established.

ASD TR 61-237, Part II

September 1962

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

INVESTIGATOR: G. B. Butler, C. F. Hauser, et al

CONTRACT: AF 33(616)-6887, University of Florida

ABSTRACT: Several vinyl and allyl phosphines have been characterized. Attempts to obtain divinyl phenyl-, diallylmethyl-, and diallylethylphosphine oxide in analytically pure form have been unsuccessful to date. Attempts to utilize the phosphorus slides obtained from methylvinylidiphenylphosphonium bromide and allyltriphenylphosphonium bromide in a Diels-Alder type reaction in which the phosphorus atom functions as one member of the diene system were unsuccessful. Attempts to polymerize such systems also were unsuccessful. Allylvinyldiphenylphosphonium bromide was polymerized by the alternating intra-intermolecular mechanism to produce a soluble, linear polymer.

Silane monomers capable of producing seven and eight membered rings by the intra-intermolecular mechanism have been synthesized and their polymerization studied. Representative monomers are 3-butenylallyldimethylsilane and di-3-butenyldimethylsilane. Low molecular weight polymers resulted. A

systematic study of diallyldiphenylsilane with a Ziegler type initiator has shown that 75% conversion to solid polymer can be obtained.

The Wittig synthesis of olefins was investigated as a route to certain phosphorus and silicon-containing olefins. While this approach was not too successful for synthesis of the phosphorus and silicon-containing olefins attempted, this study led to a number of observations concerning the Wittig synthesis. A procedure was developed which permits the use of both aliphatic aldehydes including formaldehyde (both gaseous and paraformaldehyde) and low-molecular weight unsaturated aldehydes in this synthesis. In many cases, the synthesis was found to be stereo-specific. In others, the ratio of cis-isomer to trans-isomer was as high as 4:1, rather than the usually predicted predominance of trans-isomer. A rather astonishing halide effect was observed in that the rate of the reaction when the phosphonium chloride was used was of the order of ten times that when the phosphonium bromide was used.

WADD TR 61-255, Part II

May 1962

SUBJECT: SYNTHESIS AND EVALUATION OF THERMALLY STABLE POLYMERS

INVESTIGATOR: G. P. Brown et al

CONTRACT: AF 33(616)-7076, General Electric Co.

ABSTRACT: An investigation of reaction variables affecting the synthesis of polyphenoxylenes has been continued. As in the past, major emphasis has been on the preparation of the meta-substituted polymer. Analysis of the effect of the major reaction variables indicates carbon-carbon coupling, as opposed to carbon-oxygen coupling, and cyclization reactions are the chief obstacles to obtaining polymer of the desired structure and with high molecular weight. The factors affecting side reaction and cyclization are to some extent opposed so that those favorably influencing the course of the reaction in one direction exert an unfavorable influence in the other. The most promising method of polymerization investigated, to date, is the self-condensation of potassium halophenoxide suspended in aliphatic media. This technique has been used for the production of poly-m-phenoxyene of about 10,000 molecular weight, with investigations still continuing. Other polymers investigated were poly-p-phenoxyene, poly-m-xylylene and poly-m-tolylene.

WADD TR 61-255, Part III

January 1963

SUBJECT: SYNTHESIS AND EVALUATION OF THERMALLY STABLE POLYMERS

INVESTIGATOR: G. P. Brown, A. Goldman, C. D. Doyle

CONTRACT: AF 33(616)-7076, General Electric Co.

ABSTRACT: Phase I. Results of further investigations of the role of several variables on the course of polymerization of metal salts of m-halophenols in aliphatic media are presented. In preliminary curing studies three methods of crosslinking of poly-m-phenoxyene have been attained.

Phase II - Polymer Evaluation. A sample holder is described for use in conducting differential thermal analyses in the Aminco "Thermograv". Microsoftening-range curves are given for several experimental polymers for interlaboratory comparison. Methods are given for forming semi-micro mechanical test specimens from scanty samples and for measuring their flexural characteristics, using the nondestructive vibrating reed test, followed by destructive flexural strength testing.

ASD TR 61-305

July 1962

SUBJECT: RESEARCH ON SILICON AND PHOSPHORUS DERIVATIVES OF VARIOUS NITROGEN COMPOUNDS

INVESTIGATOR: R. A. Shaw

CONTRACT: AF 61(052)-175, University of London

ABSTRACT: The new compound, hexamethyldisilazyl-lithium, has been prepared in solution and its reactions with a variety of halogen derivatives are described. Reactions of cyanuric chloride with trialkyl phosphites and other trivalent phosphorus compounds have been investigated and several new phosphorus derivatives of *s*-triazine are reported. The reaction of cyanuric chloride with diethylaniline is reported, and the preparation of silicon derivatives of *s*-triazine are under investigation.

ASD TR 61-359

July 1962

SUBJECT: RESEARCH ON TRANSITION METAL-CARBON CHEMISTRY

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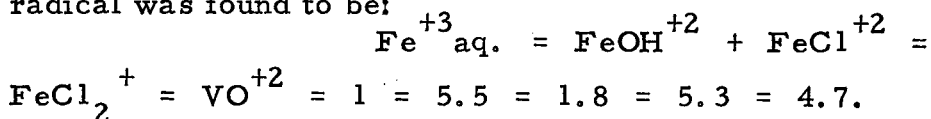
ASD TR 61-359 (Continued)

INVESTIGATOR: G. Wilkinson
CONTRACT: AF 61(052)-176, Imperial College of Science & Technology, London, England
ABSTRACT: The interaction of tri-iron dodecacarbonyl, $\text{Fe}_3(\text{CO})_{12}$, with octafluorocyclohexa-1,3- or 1,4-dienes forms a compound, $\text{C}_6\text{F}_8\text{Fe}(\text{CO})_3$, which must be formulated as a π -bonded olefin complex. In contrast, the interaction of tetrafluoroethylene with $\text{Fe}(\text{CO})_5$ gives a compound, $\text{C}_2\text{F}_4\text{Fe}(\text{CO})_4$, in which the iron atom is bound to carbon by σ -bonds in a perfluorocycloalkane ring. Infrared, high resolution nuclear magnetic resonance, and mass spectroscopic studies support these formulations. Products from the interaction of tetrafluoroethylene with $\text{Co}_2(\text{CO})_8$ and $(\pi\text{-C}_5\text{H}_5)_2\text{Co}$ and of the latter with trifluoromethyl iodide are described.

ASD TDR 61-561, Part II

October 1962

SUBJECT: KINETIC STUDY OF ION-SENSITIZED TERMINATIONS OF RADICAL POLYMERIZATION
INVESTIGATOR: E. A. S. Cavell
CONTRACT: AF 61(052)-376, University of Southampton
ABSTRACT: The investigation of the azo initiated aqueous polymerization of sacrylamide has been extended to consider termination of the growing radical chains by ferric sulfate, ferric chloride and a vanadyl perchlorate. The predicted effects due to radical termination by transition metal ions was found. The relative efficiencies of various species in terminating the acrylamide radical was found to be:



ASD TR 61-735

October 1962

SUBJECT: ORGANOMETALLIC COMPOUNDS AND POLYMERS BASED ON METAL-NITROGEN BOND SYSTEMS
INVESTIGATOR: D. C. Bradley, K. J. Chivers, E. Torrible

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ASD TR 61-735 (Continued)

CONTRACT: AF 33(616)-6934, University of Western Ontario

ABSTRACT: Reactions involving tetrakis-(diethylamido)-titanium, $\text{Ti}(\text{NEt}_2)_4$, and the polyamines, diethylene triamine, triethylene tetramine, and tetraethylene pentamine, gave insoluble non-volatile polymeric products of uncertain composition. Reactions involving n-butylamine, and $\text{Ti}(\text{NEt}_2)_4$ gave rise to solid soluble polymers of the general type $\text{Ti}_n - (\text{NBu}^n)_{2n-2} - 2(\text{NEt}_2)_4$. From molecular weight studies it was deduced that these compounds were probably rigid polymers. Further work on the cobalt chloride-lithium diethylamide reaction has led to an improved method of obtaining a novel cobalt compound.

ASD TDR 62-73, Part II

January 1963

SUBJECT: EFFECTS OF ULTRAHIGH PRESSURES ON THE FORMATION AND PROPERTIES OF ORGANIC SEMIORGANIC, AND INORGANIC MATERIALS

INVESTIGATOR: E. J. Bradbury, H. H. Krause

CONTRACT: AF 33(616)-7471, Battelle Memorial Institute

ABSTRACT: Superpressure effected crosslinking of polybutadiene and apparent graft copolymerization. Methylmethacrylate added to polymethylmethacrylate to give increased-molecular-weight products. Pressurization of other polymers did not produce significant changes. Polymerization of quinone to a relatively heat-stable material was effected by pressurization. Dimethyldicyanosilane was converted to an insoluble polymeric material. Certain aluminosiloxanes have been polymerized by pressure and heat. Pressure stabilized the Al-O-C arsenosiloxane (pentavalent arsenic) and Si-C bonds. Decomposition of an arsenosiloxane (trivalent arsenic) was promoted. Superpressures appeared to inhibit formation of phthalocyanine polymers. Pressurization of aluminum phenoxide gave the same products as obtained by heating. The equilibrium composition of iron monosulfide was shown to be pressure-temperature dependent. A new high-pressure form of aluminum phosphate, probably $\text{Al}_2(\text{PO}_4)_2 \cdot \text{Al}(\text{OH})_3$, and an iron analogue was synthesized. A new high-pressure form of iron phosphate was found. Zirconium pyrophosphate was transformed to a new high-pressure polymorph.

ASD TDR 62-200, Part II

February 1963

SUBJECT: SYNTHESIS OF THERMALLY-STABLE POLYMERS
INVESTIGATOR: Carl S. Marvel et al
CONTRACT: AF 33(616)-7908, University of Arizona
ABSTRACT: Diacrylyl methane has been converted to a low polymer with a cyclic diketone unit but no polyoxime has been obtained. The dimethyl derivative seems more promising.

Further work has been reported on poly-1, 3-cyclohexadiene and Kovacic's polyphenyl has been compared with its dehydrogenation product.

An improved procedure for making poly-benzimidazoles enables one to make 50-60 g. runs. Some derivatives of the basic polymer have been prepared.

ASD TDR 62-251, Part II

January 1963

SUBJECT: RESEARCH ON INORGANIC POLYMER SYSTEMS
INVESTIGATOR: H. Steinberg, R. J. Brotherton, et al.
CONTRACT: AF 33(616)-7303, U. S. Borax Research
ABSTRACT: This report includes investigations by U. S. Borax Research Corporation and a university subcontractor on the chemistry of thermally stable inorganic and semi-inorganic polymers. Polymers based on aluminum-oxygen and boron-nitrogen bonding systems as well as a variety of aromatic polymeric systems linked through boron-nitrogen and boron-oxygen bonds have been studied at U. S. Borax Research Corporation. High-pressure polymerization reactions and a novel approach to boron-nitrogen polymers have been investigated at Indiana University.

ASD TDR 62-261

April 1962

SUBJECT: THERMAL STABILITY OF PLASTICS
INVESTIGATOR: Gerhard F. L. Ehlers
ABSTRACT: This report is a literature survey treating the thermal stability aspects of polymers, more specifically of plastic

ASD TDR 62-261 (Continued)

materials, and includes: (1) the reversible property changes occurring under the influence of heat, especially the phenomena of the "glass transition temperature" and the "melting point;" (2) irreversible property changes (decomposition), the factors contributing to stability, type of decomposition reactions, the chemical reactions occurring during the breakdown of conventional plastics; (3) methods to determine thermal stability; and (4) the structure of polymers correlated to their stability.

ASD TDR 62-263

May 1962

SUBJECT: SYNTHESIS AND REACTIONS OF 2, 2'-
DIHALODIPHENYLAMINE

INVESTIGATOR: C. Tamborski, G. Baum, H. Lloyd

ABSTRACT: The preparation and reactions of 2, 2'-dihalodiphenylamine have been investigated. The dihalodiphenylamine, where the halogen is chlorine or bromine, has been prepared via the Chapman rearrangement. The resulting secondary amines were alkylated successfully to the tertiary amines. The ability of converting the halogens of the tertiary amines to a reactive organometallic, Grignard or lithium derivative, has been studied. The resulting di-organometallic intermediates were reacted with carbon dioxide, diphenylphosphorus chloride and phenylphosphorus dichloride to yield B-alkyl-2, 2'-dicarboxydiphenylamine, N-alkyl-2-diphenylphosphinodiphenylamine and N-alkyl-2, 2'-bisdiphenylphosphinodiphenyl-amine, and 5-alkyl-10-phenyl-5, 10-dihydrophenophosphazine. The last compound represents a new nitrogenphosphorous heterocyclic structure which will be evaluated as an anti-oxidant and lubricity additive for high temperature operating engine oils.

ASD TDR 62-283

March 1962

SUBJECT: UNSOLVED PROBLEMS IN POLYMER
SCIENCE

INVESTIGATOR: Frank R. Mayo

CONTRACT: AF 33(616)-8161, National Academy of
Sciences

ASD TDR 62-283 (Continued)

ABSTRACT: Unsolved problems and gaps in our fundamental knowledge of polymer science are considered in forty-four contributed Sections, arranged in seven Parts: Preparations and Reactions of Organic Macromolecules, Inorganic and Mixed Macromolecules, Constitution of Polymers, Macromolecular Solutions, Solid Polymer Systems, Mechanical Properties, Miscellaneous Properties.

The literature references at the introduction to each Part, at the end of each Section, and at the end of the Survey provide background material for the Sections and also a comprehensive (but not complete) list of recent books and reviews in polymer science.

ASD TDR 62-322, Part II

December 1962

SUBJECT: PHENYLENE SULFIDE POLYMERS
INVESTIGATOR: H. A. Smith, C. E. Handlovits
CONTRACT: AF 33(616)-7251, Dow Chemical Co.
ABSTRACT: The investigation of phenylene sulfide polymers has been divided into seven areas of endeavor. These are monomer synthesis, polymerization studies, large scale preparations, physical properties of the linear polymer, chemical properties of the polymer, crosslinking studies, and applications. With improvements in the monomer synthesis polymerization, one pound batches of polymer can be made. Once made, the polymer has a degree of polymerization in excess of 100 and is inert to air up to 300°C. It can be chemically crosslinked to improve its stability at 400°C in air or heat treated to not only improve its thermal and oxidative stability at 400°C but to improve its properties as well. The polymer can form fibers from the melt and be used in coatings and laminates. However, its most thoroughly investigated property is its adherence. Bond strengths up to 2700 psi have been obtained on 17-7 stainless steel. It also adheres well to glass and aluminum.

ASD TDR 62-372

August 1962

SUBJECT: CONFERENCE ON HIGH TEMPERATURE
POLYMER AND FLUID RESEARCH
INVESTIGATOR: R. J. McHenry

ASD TDR 62-372 (Continued)

ABSTRACT: This report is a collection of the papers presented at the Directorate of Materials and Processes ASD conference on "High Temperature Polymer and Fluid Research" held in Dayton, Ohio, on 8-11 May 1962.

The purpose of this conference was to review the recent progress in both contractual and internal research programs sponsored by the Polymer Branch of the Nonmetallic Materials Laboratory in the area of synthesis of new polymers and fluids.

ASD TDR 62-485

March 1963

SUBJECT: SYNTHESIS AND EVALUATION OF (POLY-PHENYLENETRIAZOLES) AND RELATED POLYMERS

INVESTIGATOR: M. R. Lilyquist, J. R. Holsten

CONTRACT: AF 33(616)-8254, Chemstrand Research Center

ABSTRACT: This report presents the results of investigations toward (1) the synthesis and evaluation of phenylenetriazole polymers as candidate materials for thermally stable film and fiber applications and (2) exploratory research to find new thermally stable materials. Four poly(phenylene)-4-phenyltriazole compositions of high molecular weight (η_{inh} , 0.5-1.5) were prepared from corresponding high molecular weight poly(phenylene) hydrazides by cyclocondensation reactions with aniline in polyphosphoric acid. One composition, poly (m-, p-phenylene)-4-phenyltriazole was picked for evaluation in film and fiber applications. This polymer showed potential usefulness in the temperature range of 300-400°C as fibers, films, protective coatings for metal surfaces and electrical insulating coatings. Further research is recommended to explore modifications in the synthesis procedures and structures of this and related polymers, and to more completely evaluate their potential as candidate materials for film and fiber applications.

ASD TDR 62-863

December 1962

SUBJECT: SYNTHESIS AND REACTIONS OF SUBSTITUTED ETHERS, AMINO-DERIVA-

ASD TDR 62-863 (Continued)

TIVES, THIOETHERS AND DISULFIDES
OF s-TRIAZINES

INVESTIGATOR: G. F. L. Ehlers, G. A. Loughran
ABSTRACT: Several methyl- and phenyl-substituted phenoxy- and amino-derivatives of s-triazines have been prepared by reaction of the corresponding chloro-derivatives with phenol and with amines in the melt and in solution. A number of new substituted thioether and disulfide derivatives of s-triazine have been prepared and identified. In addition tautomerism of several oxygen and sulfur derivatives of methyl or phenyl substituted s-triazines are described. The existence of these derivatives in the keto or thioketo configuration in the solid state is shown.

ASD TDR 62-864

December 1962

SUBJECT: IR-SPECTRA OF s-TRIAZINE DERIVATIVES
INVESTIGATOR: G. F. L. Ehlers, G. A. Loughran
ABSTRACT: Infrared absorption spectra from 2-15 have been obtained from ethers, amino-derivatives, thioethers and disulfides of s-triazines and are discussed.

ASD TDR 62-941

November 1962

SUBJECT: RESEARCH ON LINEAR BORON-NITROGEN POLYMERS
INVESTIGATOR: Michael F. Lappert
CONTRACT: AF 61(052)-419, University of Manchester
ABSTRACT: Attempts to prepare organoboron sulphates were unsuccessful.

The following new organoboron compounds were synthesized: Bis (o-phenylenedioxyboron) sulphamide, $(\text{o-C}_6\text{H}_4\text{O}_2)_2\text{NHSO}_2$

o-phenylene t-butylaminoboronate
o-phenylene ethylaminoboronate
o-phenylene isopropylaminoboronate
o-phenylene phenylaminoboronate
o-phenylene dimethylaminoboronate

ASD TDR 62-941 (Continued)

The last four compounds were all obtained as hydrolytically stable dimers.

The problem of monomer $\rightleftharpoons$ dimer, equilibrium and the influence thereon of substituents on the nitrogen atom has been studied qualitatively.

ASD TDR 62-941, Part II

January 1963

SUBJECT: RESEARCH ON LINEAR BORON-NITROGEN POLYMERS
INVESTIGATOR: Michael F. Lappert
CONTRACT: AF 61(052)-419, University of Manchester
ABSTRACT: Several novel organic-substituted B-N and B-O compounds have been prepared, characterized, examined spectroscopically, and studied chemically. The new classes of compounds are the sulphamidoboranes, $=B-NH-SO_2-NH-B=$, sulphonamidoboranes, $=B-NH-SO_2-$, amidoboranes, $=B-NR'-CO-R''$, carbamato-boranes, $=B-CO_2-NR'R''$, and cyclic boroureas, $-B<(NR'-CO_2>)_2NR''$. Aminoboranes of types $o-C_6H_4O_2>B-NR'R''$ and $o-C_6H_4(NH)_2>B-NR'R''$ were studied, primarily from the standpoint of the equilibrium: 2 monomer $\rightleftharpoons$ dimer (hydrolytically-stable), and its dependence on R' and R'' . Boron-oxygen compounds investigated include $(RO)_2B-O-B(OR)_2$, $(ROBO)_4$ (not yet confirmed), $(R'R''NBO)_3$, and isocyanate-boroxine polymers.

ASD TDR 62-942

November 1962

SUBJECT: RESEARCH ON PROBLEMS OF RESTRICTED ROTATION RELEVANT TO INORGANIC POLYMERS
INVESTIGATOR: Michael F. Lappert
CONTRACT: AF 61(052)-441, University of Manchester
ABSTRACT: Evidence from nuclear magnetic resonance studies for π -bonding between boron and nitrogen in the borazens is presented. Preliminary experiments, by infrared spectroscopy, which indicate that a similar situation might exist between phosphorus (III) and nitrogen, in open chain compounds, are also reported.

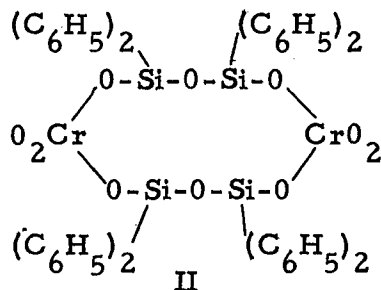
ASD TDR 62-942 (Continued)

The energy barrier to rotation about the BN bond in dimethylaminophenylchloroborane is calculated (from four different observations on the N.M.R. spectrum) as 18 ± 2 Kcals.

ASD TDR 62-1094

January 1963

SUBJECT: INORGANIC HETEROPOLYMERS
INVESTIGATOR: R. N. Hammer, J. B. Kinsinger
CONTRACT: AF 33(616)-6943, Michigan State University
ABSTRACT: Physical and chemical evidence for the cyclic structure of the siloxychromium compound II is complete, and this structure is assigned:



A spectrophotometric method for following the reaction of $(C_6H_5)_3SiOH$ and CrO_2Cl_2 has been developed and kinetic studies of the reaction initiated. Some silylchromates containing a single Si-F bond have been prepared and the reaction of chlorosilanes with silver chromate has been studied.

ASD TDR-63-53

March 1963

SUBJECT: PREPARATION AND PROPERTIES OF AROMATIC POLYSULFONATES
INVESTIGATOR: D. W. Thomson, G. F. L. Ehlers
ABSTRACT: A series of polyesters of aromatic sulfonic acids have been prepared for the first time. The aromatic groups have been varied in order to determine the effect of structure on the thermal stability and molecular weight of the polymer. The physical properties of the polymers have been determined and are reported.

ASD TDR 63-130

January 1963

SUBJECT: INTERMEDIATES FOR HIGH-TEMPERATURE STABLE POLYMERS
INVESTIGATOR: P. Szecsi, J. D. Behun, et al
CONTRACT: AF 33(657)-7613, Wyandotte Chemicals Corp.
ABSTRACT: The synthetic methods and experimental procedures adopted for the preparation of materials specified by ASD that are to be used as intermediates in high-temperature polymer studies, are discussed. The compounds include ferrocenes, α , ω -diolefins and triazine derivatives.

ASD TDR 63-156, Part I

April 1963

SUBJECT: POLYMERIZATION KINETICS BY MEANS OF DIFFERENTIAL THERMAL ANALYSIS, Part I. Apparatus
INVESTIGATOR: W. E. Gibbs, K. R. Fisch
ABSTRACT: A differential thermal analysis (DTA) apparatus has been designed to determine kinetic and thermodynamic data from polymerization reactions. The apparatus was calibrated and the decomposition rate of azobisisobutyronitrile was determined. The data obtained are in good agreement with those reported in the literature.

ASD TDR 63-249

March 1963

SUBJECT: SYNTHESIS OF POLY-HETEROCYCLICS
INVESTIGATOR: Philip E. Brumfield, et al.
CONTRACT: AF 33(657)-7476
ABSTRACT: Polybenzothiazoles were prepared by condensation of 3, 3' -dimercaptobenzidine with dibasic acids. The polymer from terephthalic acid had best physical properties. Products from isophthalic, diphenic, maleic and oxy-bis(4-benzoic)acids were less attractive. The polybenzothiazoles had high thermal stability but in general had low solubility and fusibility. Attempts to join thiazole units by other linkages were not promising.

Study of perfluorovinyl addition polymers dealt primarily with 2-trifluorovinylbenzothiazole. This monomer was

ASD TDR 63-249 (Continued)

prepared but would not form a polymer. A polybenzimidazole from 3, 3' -diaminobenzidine and perfluoroglutaric acid was found to readily lose hydrogen fluoride.

Preparation of poly(1, 3, 4-oxadiazoles) with fluorocarbon linkages was partially successful. Low molecular weight polyhydrazides were obtained and converted to low molecular weight polyoxadiazoles having some residual hydrazide linkages.

ANALYTICAL TECHNIQUES

WADD TR 61-71, OTS Release

May 1962

SUBJECT: CHEMICAL STRESS RELAXATION MEASUREMENTS OF POLYURETHANES
INVESTIGATOR: S. J. Chlystek, et al
CONTRACT: AF 33(616)-7088, Armstrong Cork Co.
ABSTRACT: Three solid polyurethane elastomeric compositions were prepared and evaluated by static and high speed tensile tests, continuous and intermittent stress relaxation measurements, and also by ultrasonic techniques. This preliminary evaluation was conducted to evaluate chemorheological stress relaxation as a means of characterizing polyurethane materials with regard to thermal and mechanical stability. Energies of activation for bond scission were calculated. An annotated bibliography of stress relaxation phenomena was prepared.

ASD TDR 62-275, OTS Release

June 1962

SUBJECT: STUDY FOR IMPROVEMENT OF MATERIALS CENTRAL MASS SPECTROMETER
INVESTIGATOR: G. D. Perkins, D. H. Pollock
CONTRACT: AF 33(616)-7932, Bell & Howell
ABSTRACT: Considerable improvement in average yield and in ease of spark manipulation has been achieved. Photographic emulsion variations have been a serious limitation to sensitivity and to analysis accuracy but with a satisfactory plate sensitivity of approximately 1 in 10^9 has been obtained with exposure durations of approximately one and a half hours. The flexibility of Knudsen Cell operation has been greatly enhanced by a complete redesign of the supporting structure.

ASD TDR 62-519, OTS Release

August 1962

SUBJECT: A PROGRAM OF BASIC RESEARCH TO STUDY X-RAY SPECTRA IN THE REGION 15 TO 50 Å
INVESTIGATOR: D. W. Beard, T. C. Furnas, Jr.

ASD TDR 62-519 (Continued)

CONTRACT: AF 33(616)-8040, Picker X-Ray Corp.
ABSTRACT: Performance tests have been completed on the vacuum spectrographic system described in a previous report. High-intensity X-ray excitation was found to produce low working intensities and was not useful in the detection and analysis of low energy x-rays. Electron excitation provided satisfactory intensities, and made possible the measurement of radiation as "soft" as carbon (44A), as well as the L series spectra from transitional elements. A wave length shift in the L line was observed between metallic iron and iron in the form of the oxide. A Bendix M-304 detector was studied and found to give non-proportional response to incident photon energy, but it did maintain a low noise level.

ASD TDR 62-862, OTS Release

October 1962

SUBJECT: THE INFRARED SPECTRA OF SOME ALDEHYDES BETWEEN 15 AND 35 MICRONS
INVESTIGATOR: J. V. Pustinger, Jr., J. E. Katon
CONTRACT: AF 33(616)-8465, Monsanto Research Corp.
ABSTRACT: Spectral data for 41 aldehydes have been recorded from 700 to 300 cm^{-1} and analyzed for spectra-structure correlations. The information so attained can be applied in identifying aliphatic and aromatic aldehydes. Specific correlations were determined for aliphatic aldehydes with or without α -branching. Tentative correlations were observed for α -olefinic, alicyclic, and aromatic aldehydes.

ASD TDR 62-874, OTS Release

November 1962

SUBJECT: X-RAY POWDER DIFFRACTION DATA FOR SOME FERROCENE DERIVATIVES
INVESTIGATOR: W. L. Baun, M. Goldschmidt
ABSTRACT: Application of X-ray diffraction to identification of polycrystalline ferrocene derivatives is discussed. These compounds are well suited to X-ray diffraction analysis because of their crystalline nature and the individualistic patterns obtained even in closely related substitutions. Data are shown for various mono and di-substituted compounds. Emphasis is placed on effects

ASD TDR 62-874 (Continued)

noted in long chain substitutions and spacings are given for 12 of this type of compound. Powder diffraction data and other physical properties are shown for twenty ferrocene derivatives which were synthesized for research on ultraviolet absorbers for space vehicles.

ASD TDR 62-895, OTS Release

October 1962

SUBJECT: FREQUENCY ASSIGNMENTS OF BENZENE DERIVATIVES IN THE CESIUM BROMIDE REGION

INVESTIGATOR: R. J. Jakobsen

CONTRACT: AF 33(616)-7450, Battelle Memorial Institute

ABSTRACT: Frequency assignments in the CsBr spectral, region (650 to 250 cm^{-1}) have been made from the spectra of 279 benzene derivatives. The Raman spectra of 119 of these compounds were also available and were used as aids in making the assignments. The assigned frequencies include several benzenelike ring deformations and some vibrations that are either internal vibrations of the substituent group or involve the ring-substituent bond. A variation in frequency with symmetry or substitution type (ortho, meta, para, etc.) was found for the ring deformations. Within a given substitution type certain of the ring-deformation vibrations were found to be sensitive to substituent effects. The assigned frequencies involving the substituent group can be classified as characteristic frequencies of benzene compounds with a particular substituent.

ASD TDR 62-940, OTS Release

November 1962

SUBJECT: SUMMARY REPORT ON MOLECULAR STRUCTURE AND MATERIALS CHARACTERIZATION

INVESTIGATOR: J. E. Katon

CONTRACT: AF 33(616)-8465, Monsanto Research Corp.

ABSTRACT: This report summarizes the work accomplished under Contract No. AF 33(616)-8465 between 1 July 1961 and 1 July 1962. This contract concerns the determination of molecular structures, characterization of materials, and the development and application of various analytical techniques. It also concerns various services supplied the Air Force in support of their in-house programs.

ASD TDR 62-940 (Continued)

This report summarizes the three research programs undertaken and the service functions performed. The three research projects, reported in detail in separate ASD reports, concern far infrared spectra-structure correlations of ketones, ethers, alcohols, and aldehydes; spark source mass spectrometry of organic compounds; and application of thermogravimetric and differential thermal analysis techniques to the characterization of high temperature polymeric systems.

ASD TDR 62-1018, OTS Release

February 1963

SUBJECT: SOLUBILITY AND DIFFUSION OF GASES
IN BERYLLIUM

INVESTIGATOR: J. P. Pemsler, et al.

CONTRACT: AF 33(616)-7665

ABSTRACT: Results of an experimental program to determine the solubility and diffusion rate of nitrogen, hydrogen, and oxygen in beryllium are reported. The solubility of nitrogen in beryllium at 1000°C is estimated to be about 60 ppm; this value appears to be temperature independent. The results of nitrogen diffusion experiments are considered tentative. The solubility of hydrogen in beryllium is believed to be very small. Measurement of the growth characteristics of hydrogen bubbles formed in beryllium by proton bombardment indicate a value for the diffusion coefficient of hydrogen in beryllium of 9×10^{-10} cm²/sec at 850°C. Due to limitations of chemical analysis, no new data concerning the beryllium-oxygen system was obtained.

ASD TDR 63-278, OTS Release

April 1963

SUBJECT: LOW TEMPERATURE X-RAY DIFFRACTION
TECHNIQUES

INVESTIGATOR: W. L. Baun, J. J. Renton

ABSTRACT: A review and bibliography of 78 references on low temperature X-ray diffraction techniques are presented. A low temperature specimen mount for the horizontal Siemens diffractometer is described. Construction details, sampling techniques, and low temperature methods are discussed.

ASD TDR 63-290, OTS Release

April 1963

SUBJECT: THE SPECTROCHEMICAL DETERMINATION OF BORON IN MOLYBDENUM

INVESTIGATOR: J. H. Muntz

ABSTRACT: A method for the spectrochemical determination of boron in molybdenum is described. The method provides for the determination of boron in the 50 to 500 ppm range by a carrier-distillation technique with a precision of ± 10 per cent.

ASD TDR 63-310, OTS Release

May 1963

SUBJECT: PREPARATION AND USE OF ORGANIC COMPOUNDS AS DISPERSING DEVICES FOR LONG WAVELENGTH X-RAYS

INVESTIGATOR: W. L. Baun, D. W. Fischer

ABSTRACT: Long spacings and physical properties are shown for a number of metallic salts of n-aliphatic acids. Among the salts made are those of thallium, copper, lead, zinc, uranyl radical, iron, cobalt, nickel, magnesium, and the more soap-like members such as sodium, potassium, lithium and calcium. Spacings ranged from about $15\text{\AA}$ to $40\text{\AA}$ for the salts measured, which is of the correct size for use as a dispersing device in the X-ray spectral region 15 to $60\text{\AA}$. Some salts were found to have properties which negated their use as dispersing devices for X-rays, while others were found to be potentially good analyzing crystals or three dimensional gratings. Of the compounds prepared, zinc, thallium and uranyl salts showed promise for use as dispersing devices for long wave X rays. Almost any desired long spacing may be obtained with the right combination of metal and acid, and these spacings may be calculated once a group of three or more salts of a homologous series has been prepared. Results on some aromatic compounds such as monovalent metal salts of phthalic acid are discussed. These compounds show promise for use as single crystal analyzers for soft X rays. Soft X-ray spectra are shown using a layered barium stearate film on glass and potassium acid phthalate as analyzers.

ASD TDR 63-311, OTS Release

April 1963

SUBJECT: RESEARCH ON A SUITABLE DISPERSING OR ANALYZING CRYSTAL FOR X-RAY SPECTROSCOPY

ASD TDR 63-311 (Continued)

INVESTIGATOR: W. Ruderman, et al.
CONTRACT: AF 33(657)-7756, Isomet Corp.
ABSTRACT: A large number of potential compounds were investigated for application as X-ray analyzer crystals with 2d spacings from 25 to 100A. Four crystals with 2d values from 31 to 90A were developed and show exceptional promise. Although large crystals of these compounds were not grown, X-ray analysis of small crystals indicated excellent reflectivities.

ASD TDR 63-321, OTS Release

April 1963

SUBJECT: INFRARED DATA PROCESSOR
INVESTIGATOR: C. R. Chapman
CONTRACT: AF 33(657)-7941, ITT Federal Labs.
ABSTRACT: The IR DATA PROCESSOR is an analog-to-digital converter capable of digitizing the X and Y coordinates of the infrared spectra. Transmittance, as a shaft position within the spectrophotometer, is converted to absorbance as a dc voltage by utilizing a potentiometer and network whose output is a two-cycle logarithmic function of the input.

Absorbance, as a shaft position within the spectrophotometer, can be directly coupled to a linear potentiometer which would be applied to the IR DATA PROCESSOR. The dc voltage output of either potentiometer is transmitted to the IR DATA PROCESSOR where it is converted to a digital form. Once in the digital form, the PROCESSOR is capable of driving a card punch which records the absorbance at successive wave numbers. The PROCESSOR has the facility of sampling 1, 2, or 4 equi-spaced samples per wave number.

The PROCESSOR is capable of obtaining and recording absorbance units from 0.00 to 1.99. The accuracy of the analog-to-digital conversion of absorbance is $\pm 0.25\%$ or ± 0.005 absorbance units.

ASD TDR 63-356, OTS Release

May 1963

SUBJECT: X-RAY CRYSTALLOGRAPHIC AND POWDER

ASD TDR 63-356 (Continued)

DATA FOR SOME SALTS OF PHTHALIC ACID

INVESTIGATOR: D. W. Fischer

ABSTRACT: Results are shown for X-ray diffraction analysis and X-ray crystallographic analysis of seven salts of phthalic acid. Lattice parameters, space groups, densities and indexed powder data are given for each salt. The results show that crystals of these salts would make quite good dispersing agents for soft X rays in the 10 to 25⁰Å region.

ELECTRICAL & MAGNETIC PROPERTIES

WADD TR 60-787, Part II, OTS Release

March 1963

SUBJECT: INVESTIGATION OF THE STRUCTURAL AND
MAGNETIC PROPERTIES OF THIN FERRO-
MAGNETIC FILMS

INVESTIGATOR: A. Baltz, W. Doyle

CONTRACT: AF 33(616)-8940, Franklin Institute

ABSTRACT: Permalloy films evaporated to normal and ob-
lique incidence to a substance were examined by replication of their
surfaces by the electron microscope. A statistical analysis of the
micrographs was made.

Permalloy films were epitaxially grown on
NaCl and were annealed by electron bombardment in an electron
microscope.

The angular dependence of the torque in Per-
malloy films has been studied. The results agree very well with
the formulas for magnetization reversal in an infinite cylinder.
Various methods of determining the anisotropy have been compared
and a torque technique for measuring dispersion in both magnitude
and direction developed. The anisotropy found in films as a function
of the angle of incidence of the evaporation beam and the anisotropy in
epitaxially grown films is reported. The use of a vibrating sample
magnetometer to determine the hysteretic properties of thin films
is discussed.

ASD TDR 62-427, OTS Release

January 1963

SUBJECT: PREPARATION AND CHARACTERIZATION OF
HIGH-PURITY SINGLE-CRYSTAL BORON

INVESTIGATOR: I. R. King, et al.

CONTRACT: AF 33(616)-7884, Texaco Experiment, Inc.

ABSTRACT: Crystalline boron specimens about 3 to 4 mm in
diameter and several centimeters in length were prepared by chemical
vapor plating and subsequently floating-zone melted. X-ray-diffrac-
tion studies showed them to be the β -rhombohedral phase and single
crystalline over most of the zone-melted portions. Electrical and
optical measurements were made on some of the boron single
crystals produced.

January 1963

SUBJECT: ELECTRICAL CONDUCTORS AT ELEVATED TEMPERATURES

INVESTIGATOR: N. Fuschillo, R. A. Lindberg

CONTRACT: AF 33(616)-8386, Melpar, Inc.

ABSTRACT: Existing data and theory concerning the electrical conductivity, oxidation resistance, and mechanical properties of pure metals, metallic alloys, and metallic binary compounds are presented and analyzed to determine the best approach to developing improved electrical conductors for use at temperatures up to 700° and 1200° C. Recommendations are made for experimental research and development in pure metals, metallic alloys, and metallic binary compounds. Considerable data has been collected and systematized in tables and graphs on the electrical, mechanical, and oxidation resistant characteristics of these conductors. This should enable the design engineer to select the best electrical conductor to suit specific requirements of temperature, oxidation or mechanical environment, and cost.

September 1962

SUBJECT: OBSERVATION OF MAGNETIC DOMAINS BY MEANS OF THE KERR EFFECT

INVESTIGATOR: A. M. Stoffel, K. J. Strnat

ABSTRACT: An experimental apparatus has been built which employs the magneto-optical Kerr effect for visual observation and photographic recording of magnetic domain patterns. The main objective of this work was to familiarize the investigators with the problems of the Kerr technique and thus provide the basis for designing a more sophisticated apparatus.

This report discusses the Kerr effect and its application to the study of magnetic domains and analyzes some of the optical problems incurred in this application. It describes the experimental set-up and presents pictures of domain structures observed on a thin film of Permalloy.

ASD TDR 62-1026, OTS Release

January 1963

SUBJECT: ANNOTATED BIBLIOGRAPHY ON SOLID
STATE OPTICAL AND INFRARED MASER
MATERIALS

INVESTIGATOR: Dieter Feldmann

ABSTRACT: This report contains abstracts of approximately 110 articles from 25 different scientific journals. It covers theory and survey; solid state maser materials; maser optics; properties (coherence and others), related basic research; and organic, semiconducting, and gas masers. The survey includes articles from the beginning of the optical maser research to December 1961.

ASD TDR 63-83, OTS Release

March 1963

SUBJECT: OBSERVATION OF MAGNETIC DOMAINS BY
MEANS OF THE BITTER COLLOID METHOD

INVESTIGATOR: R. J. Patton, K. J. Strnat

ABSTRACT: Experimental equipment has been developed for the light-microscopic observation of ferro-magnetic domains by means of the Bitter technique. The design of an electromagnet-microscope stage which can accommodate both bulk samples and thin sheet strips is described. The formula for the preparation of the colloidal iron-oxide solution is given. Photographs of domains on silicon-iron transformer sheet illustrate the quality of the pictures and the resolution which can be obtained with the set-up.

ASD TDR 63-208, OTS Release

April 1963

SUBJECT: RESEARCH AND DEVELOPMENT TO INVESTI-
GATE THE MICROSTRUCTURE OF THE INTER-
NAL MAGNETIC FIELD IN FERRO-, FERRI-
AND ANTIFERROMAGNETIC SOLIDS

INVESTIGATOR: D. S. Rodbell, et al.

CONTRACT: AF 33(657)-8462, General Electric Co.

ABSTRACT: Research dealing with fundamental interactions and the microstructure of internal fields in selected magnetic materials is described. Static and microwave magnetic properties of single-crystal gadolinium are under investigation. Thermal expansion data

ASD TDR 63-208 (Continued)

of several ferromagnetic metals and one antiferromagnetic oxide are discussed. Cobalt particles precipitated in a copper-cobalt alloy are under study by nuclear resonance and ferromagnetic resonance for separate metallurgically oriented goals. Nuclear resonance studies of MnAs are reported and exploration commenced on electron tunneling into ferromagnetic alloys. High field studies of magnetization processes in the antiferromagnetic compounds, FeCO_3 and CoCl_2 are also reported.

HYPERVELOCITY IMPACT

ASD TDR 62-762

August 1962

SUBJECT: EFFECTS OF HYPERVELOCITY IMPACTS
ON MATERIALS
INVESTIGATOR: V. E. Scherrer
CONTRACT: AF 33(616)-8423, Technical Operations, Inc.
ABSTRACT: A novel exploding-foil gun is described which routinely accelerates small particles (mass 1-100 mg) to velocities up to 60,000 ft/sec when coupled to a slow-capacitor energy-storage system. When the gun was efficiently coupled to a fast-capacitor energy-storage system, a single, solid particle was accelerated to a velocity of 102,000 ft/sec.

A detailed study of various particles impacting quasi-infinite lead targets was made, and preliminary results are given for particle velocities from 7,000 to 40,000 ft/sec. These results indicate a deep penetration phenomenon for a particle velocity of 15,000 ft/sec. If similar phenomena are observed in materials of interest in space vehicle construction, the results will be very important in the design of such structures.

Plans are presented for expanding the hypervelocity facility and improving its performance in the future.

RADIATION CHEMISTRY & RADIATION EFFECTS

ASD TDR 62-10, Part I

July 1962

SUBJECT: BASIC STUDIES IN RADIATION EFFECTS ON MATERIALS
INVESTIGATOR: J. A. Bearden, M. Krause, W. H. Johnston
CONTRACT: AF 33(616)-7678, William H. Johnston Labs., Inc.
ABSTRACT: In the study of fragmentation processes following inner shell ionization of molecules by x-rays, a principle barrier is the extremely small cross sections for x-ray ionization. Although the Coincidence Mass Spectrometer (patented) adapted for these measurements is much more sensitive than other mass spectrometers, a specially designed x-ray tube was required to obtain sufficient gas ionization to produce accurate measurements within a reasonable time. The successful development of the special x-ray tube designed for this purpose incorporated the following unique features; long wave length cut-off, improved beam definition, and ease of assembly and exchange of essential parts. Unpublished data on the attenuation of gases and low energy x-rays by those low atomic number materials suitable for x-ray windows are presented.

ASD TDR 62-10, Part II

July 1962

SUBJECT: BASIC STUDIES IN RADIATION EFFECTS ON MATERIALS
INVESTIGATOR: M. Vestal, et al
CONTRACT: AF 33(616)-7678, William H. Johnston Labs.
ABSTRACT: The combination of a coincidence mass spectrometer (patented) and a special x-ray tube, which was described in Part I, has made possible direct measurements of the ion fragmentation patterns produced by x-ray impact on simple gas phase molecules. Also measurements concerning the secondary electron energy distributions for N₂, propane, n-butane, 1, 3, butadiene, n-pentane, argon, and cyclohexane in radiation chemistry are given, at a fixed angle, using primary ionizing electrons in the energy range of 100 to 1000 ev. Theoretical interpretation of the data in terms of radiation chemical effects is included. Mass spectra are reported for nitrous oxide, and propane under bombardment by low energy x-rays; and spectra were also obtained of these materials under bombardment by 1200 ev

ASD TDR 62-10, Part II (Continued)

primary electrons. The design, construction, and instrumentation, which has made these measurements possible, are described.

ASD TDR 62-52, OTS Release

April 1962

SUBJECT: NEUTRON-GAMMA RAY SPECTROMETER
INVESTIGATOR: J. D. Hall, J. B. Ashe
CONTRACT: AF 33(616)-7787, Texas Nuclear Corp.
ABSTRACT: A neutron-gamma ray spectrometer developed at Texas Nuclear Corporation is described. The spectrometer employs pulse shape discrimination in an organic scintillator detector to selectively detect neutrons or gamma rays in a mixed field by a method not utilizing space charge limiting. Selective detection may be obtained for neutrons of energy higher than 0.7 Mev and for gamma rays above 0.25 Mev. Spectral information is usable to 15 Mev for neutrons and to 4 Mev for gamma rays. Detection efficiencies range from about 6% to 30%. Typical spectra, data analysis procedure, and operating characteristics of the spectrometer are presented.

ASD TDR 62-233, OTS Release

October 1962

SUBJECT: RADIATION EFFECTS ON SEMICONDUCTOR CATALYSTS
INVESTIGATOR: H. C. Sosnovsky
CONTRACT: AF 33(616)-7740, Armour Research Foundation
ABSTRACT: The effect of ion bombardment on carrier lifetime, photoconductivity, and surface structure of germanium (iii) crystals was investigated after bombardment at voltages between 10 and 2000 v. It was found that the lifetime decreased significantly at bombarding voltages above 80 v and that the number of defects in the surface region increased simultaneously by several orders of magnitudes. These defects are most likely dislocations and vacancy clusters. Less stable defects were introduced at very low bombarding voltages but these too, were found to affect the electrical properties of the bombarded surfaces appreciably.

Preliminary results of the effect of adsorbed gases on the properties of ion bombarded surfaces, and the effect of

ASD TDR 62-233 (Continued)

neutron bombardment on the catalytic exchange of hydrogen with deuterium on etched surfaces, are included.

ASD TDR 62-244, OTS Release

March 1962

SUBJECT: ELECTROSTATIC SHIELDING OF HIGH
ENERGY PROTONS

INVESTIGATOR: T. W. Bailey

ABSTRACT: Electrostatic shielding was studied to determine if it should be considered for protecting manned space vehicles against solar cosmic rays. After this study, it was concluded that electrostatic shielding is feasible and that it has some advantages over other types of shields. However, considerable applied research will be necessary.

ASD TDR 62-530, OTS Release

June 1962

SUBJECT: BASES OF NEUTRON DAMAGE MONITORING

INVESTIGATOR: Leonard Solon, et al.

ABSTRACT: The problems of monitoring and reporting neutron damage effects have been reviewed. The commonly used monitors and the analyses generally associated with them are discussed in relation to the problems of damage. Areas requiring further development or revision of technique are indicated. Three analytical procedures are described, each suitable in different situations dependent upon the sample under consideration, the detectors, and the control fluxes available.

Results of monitoring damage to structural steel (obtained by NRL) are examined in some detail to learn what they imply about the state of damage monitoring and analysis.

ASD TDR 62-540, OTS Release

September 1962

SUBJECT: A STUDY OF FREE RADICALS AND OTHER
INTERMEDIATE SPECIES AND INTERNAL
ENERGY TRANSFER PROCESSES IN IRRADIATION

ASD TDR 62-540 (Continued)

DIATED CHEMICAL SYSTEMS

INVESTIGATOR: P. Y. Feng, et al
CONTRACT: AF 33(616)-7587, Armour Research Foundation
ABSTRACT: This report describes a study of the role of internal energy transfer processes in radiation chemical reactions involving free radicals and other intermediate species, using a series of alkyl iodides with different carbon chain lengths as the experimental systems. It was found that, (1) with increasing size of the alkyl group in a homologous series, for example the 1-iodoalkanes, the extent of C-I bond rupture decreases regularly, whereas both the H₂ and the HI yields increase; (2) products with practically identical yields are formed in the radiolyses of two systems with similar elemental compositions, one consisting of pure C₁₆H₃₃I, and the other an equimolecular mixture of C₈H₁₈ and C₈H₁₇I. Analysis of these results, together with those available in the literature, show that energy initially localized elsewhere in an alkyl iodide molecule cannot efficiently cause C-I bond breakage despite the latter's low bond strength, i. e. internal energy transfer does not play an important role in these reactions. Instead, it is concluded that the product yields are dependent on the effective electron fractions of the various groups in the irradiated system, a parameter which permits the quantitative correlation of the outcomes of the radiolytic reactions investigated in this program.

ASD TDR 62-575, OTS Release

July 1962

SUBJECT: VACUUM TECHNIQUES IN RADIATION
CHEMISTRY
INVESTIGATOR: Roger E. Rondeau
ABSTRACT: This report describes the high vacuum apparatus and methods used in the radiation-chemical studies of organic compounds. The design and use of high-vacuum systems for the purification, degassing, filling, and sealing of irradiation ampoules are explained. The post-irradiation techniques of removal, separation, measurement, and collection of the volatile products of radiolysis are also described.

July 1962

SUBJECT: AN ELECTRONICALLY CONTROLLED HIGH SPEED STREAK CAMERA

INVESTIGATOR: O. Van P. Sessoms, III, T. W. Bailey

ABSTRACT: This report describes the design and development of an electronically controlled high speed streak camera. The camera employs a magnetic suspension and drive mechanism which allows a prismatic mirror to be suspended frictionlessly in a magnetic field and to be driven to extremely high rotational velocities. This provides the streak camera with a time resolution and controllability which could not be achieved by more conventional means.

The design, development, and initial operation are described, but no performance data is available as administrative and technical problems forced termination of the project before it could be completed.

December 1962

SUBJECT: THE APPLICATION OF SEMICONDUCTOR RADIATION DETECTORS TO THE OBSERVATION OF FAST NEUTRONS

INVESTIGATOR: E. L. Zimmerman, et al.

CONTRACT: AF 33(616)-8340, Solid State Radiations, Inc.

ABSTRACT: Several methods of utilizing a semiconductor radiation detector for neutron observations have been investigated. Techniques for the application of neutron-sensitive materials to detector surfaces have been developed and the resulting coated detectors have been evaluated as both thermal and threshold detectors. A "good geometry" proton-recoil spectrometer utilizing a semiconductor detector has been built and used to observe fast neutrons. An analytical study of this device resulted in a method which can accurately predict its response as a function of incident neutron energy, hydrogenous radiator thickness, and system geometry. Good qualitative agreement was observed between the predicted and experimental response. Efforts to introduce Li^6 into the sensitive region of a detector in order to make the detector neutron energy sensitive by means of the $\text{Li}^6(n, \alpha) \text{T}^3$ reaction have not been successful to date. However, this effort has contributed substantially to the development of the lithium ion drift detector which in itself is a good 4.5 Mev

ASD TDR 62-838 (Continued)

threshold neutron detector, as well as a good device for observing minimum ionizing particles.

ASD TDR 62-875

October 1962

SUBJECT: RADIATION CHEMISTRY OF BENZENE
SOLUTIONS OF ORGANOMETALLIC COM-
POUNDS OF THE TYPE $M(C_6H_5)_n$

INVESTIGATOR: M. Burton, D. B. Peterson

CONTRACT: AF 33(616)-7075, University of Notre Dame

ABSTRACT: Product yields are reported for Co^{60} -gamma radiolysis of dilute benzene solutions of tetraphenyls of Si, Ge, Sn and Pb and triphenyls of As and Bi. The latter four compounds undergo a concentration-dependent, sensitized decomposition best explained in terms of energy transfer from excited benzene to organometallic. $G(H_2)$ and $G(C_2H_2)$ for benzene are unaffected by any of the solutes indicating that these two products are formed from a state or states of benzene other than the state(s) transferring energy to the Organometallic. The ability of solutes to undergo radiation-sensitized decomposition is compared with ability to quench luminescence of scintillators in benzene solutions. Qualitative agreement is found in that good quenchers undergo sensitized decomposition while poor quenchers ($Si\phi_4$ and $Ge\phi_4$) do not.

ASD TDR 63-65, OTS Release

February 1963

SUBJECT: THE GAS PHASE RADIOLYSIS OF 2-BUTYNE

INVESTIGATOR: R. E. Rondeau, et al

ABSTRACT: Pyrex ampoules of gaseous 2-butyne (dimethylacetylene) were exposed to cobalt-60 gamma rays, and the hundred electron-volt yields of the lower molecular weight products are given. The radiation induced products were studied as a function of sample pressure and total dose. The products, which include hydrogen, methane, acetylene, propene, propyne, cis- and trans- 2-butene, butane, 1,2-butadiene, and vinylacetylene, are explained on the basis of ion-molecule and free radical reactions. Some mechanisms of product formation which are consistent with product distribution and magnitude of the yields are discussed.

ASD TDR 63-89, OTS Release

March 1963

SUBJECT: THE SPM METHOD FOR NEUTRONS
INVESTIGATOR: R. Aronson, J. Heitner
CONTRACT: AF 33(616)-7732, TRG, Inc.
ABSTRACT: The SPM equation has been recoded for the IBM-7090. Both theoretical analysis and some numerical results are given and discussed.

ASD TDR 63-273, OTS Release

March 1963

SUBJECT: ABSTRACTION OF HALOGEN ATOMS BY METHYL RADICALS
INVESTIGATOR: M. Szwarc, et al
CONTRACT: AF 33(616)-7662, College of Forestry
ABSTRACT: Halogen abstraction reactions, described by equation, $RX + \cdot CH_3 \rightarrow R\cdot + XCH_3$ were investigated in solution for $R = CH_3, C_2H_5, iso-C_3H_7, t-C_4H_9, PhCH_2, CH_2Cl, CHCl_2, CCl_3$ and CF_3 . Secondary deuterium effect was investigated for CH_3 and CF_3 addition reactions. Rate constants $k_2 I/k_1$ and $k_2 Br/k_1$ were found to be 45 and 3×10^{-3} respectively. k_1 being the rate constant of the reaction $PhCH_2 + \cdot CH_3 \rightarrow PhCH_2\cdot + \cdot CH_4$. The values of $k_2 I/k_1$ and $k_2 Br/k_1$ are compared with $k_2 H/k_1$.

The activation energies difference $E_2 I - E_1$ was determined as - 1.8 Kcal/mole for the temperature range 55-85°C.

THERMO-PHYSICS

WADD TR 60-370, Part II, OTS Release

August 1962

SUBJECT: THERMAL RADIATION PROPERTIES OF MATERIALS

INVESTIGATOR: R. A. Seban

CONTRACT: AF 33(616)-6630, University of California

ABSTRACT: Spectral emittance data are presented for a number of oxidized and coated materials and these, combined with reflectances measured at low temperature, demonstrate the relative insensitivity to temperature of these spectral properties. The specification of the Hagen Rubens equation of the spectral properties of metals is investigated by comparison to data at high and low temperatures and its usefulness is shown to be due to a combination of photoelectric absorption and of possible effects of surface stress. These are described, and preliminary data are presented from a system for the measurement of emittance at high temperature, with the sample in an environment of inert gas.

WADD TR 60-371, Part II, OTS Release

December 1962

SUBJECT: A SOURCE AND DETECTOR OF RADIATION IN THE WAVELENGTH REGION 1500-50 ANGSTROMS SUITABLE FOR RADIATION EFFECTS STUDIES ON MATERIALS IN VACUO: A Study of Vacuum Ultraviolet Radiation Effects on Materials in Vacuo of Order 10^{-9} mm Hg.

INVESTIGATOR: H. R. Moore, H. Bernstein, R. S. Reynolds

CONTRACT: AF 33(616)-6488, Electro-Optical Systems, Inc.

ABSTRACT: This report describes a space environmental research facility whereby the effects of ultrahigh vacuum and/or vacuum ultraviolet radiation on materials can be experimentally evaluated. The facility consists of an environmental chamber of cylindrical dimensions 18" diameter x 18" depth, which is equipped with various functional and diagnostic controls, and with which is associated a vacuum ultraviolet radiation source and a radiation detector. The radiation source is a pulsed plasma discharge which emits a line and continuum spectrum extending from the visible, through the vacuum ultraviolet, into the soft x-ray region. The radiation detector

utilizes an open photoelectron multiplier tube which is mounted in the chamber for measurements of the radiation pulse energy. The environmental facility vacuum system includes a differentially pumped source-chamber connection and is capable of maintaining chamber pressures of order 10^{-9} mm Hg while the source is in operation. This report includes a description of the results of some exploratory studies of the effects of ultrahigh vacuum and/or vacuum ultraviolet radiation on the characteristics of a silicon solar cell, the electrical surface resistivity of aluminum oxide, and the optical reflectance of metallic mirror coatings.

WADD TR 60-463, Part II, OTS Release

October 1962

SUBJECT: THE VAPORIZATION AND PHYSICAL PROPERTIES OF CERTAIN REFRACTORIES: Experimental Studies

INVESTIGATOR: A. A. Hasapis, et al.

CONTRACT: AF 33(616)-6840, Avco Corp.

ABSTRACT: The vapor pressure-temperature relationships for iridium, rhodium, ruthenium, iridium plus carbon, alumina, thoria, and hafnia were studied by simple effusion and mass spectrometric methods. Vapor phase species were identified. The vapor pressure data and derived heats of vaporization and reaction are presented. The vaporization modes of the rare earth sesquioxides were studied mass spectroscopically and data which demonstrate that the mode of vaporization of these materials is strongly dependent upon the number of electrons in the 4f shell of the rare earth metal, is given. Continuously monitored effusion experiments conducted on the Mo-Si system yielded the following heats of dissociation in kcal/gm atom Si at 298°K: Liquid-MoSi₂, 117.8; MoSi₂-Mo₅Si₃, 121.5; Mo₅Si₃-Mo₃Si, 128.3; Mo₃Si-Mo, 132.9. Continuously monitored experiments on the W-Si system yielded the following approximate dissociation pressures in atms. at 1765°K: liquid-WSi₂, 3.5×10^{-6} ; WSi₂-W₃Si₂, 2.6×10^{-6} ; W₃Si₂-W, 1.7×10^{-6} . The surface tensions of silica and silica plus 1% oxide additives and zirconium plus uranium have been measured by a sessile drop technique. The viscosities of alumina, zirconium plus uranium, pure silica, silica plus 1% V₂O₅, silica plus 1% CoO and silica plus 1% Al₂O₃, have been determined.

June 1962

SUBJECT: THERMAL PROPERTIES OF REFRACTORY MATERIALS

INVESTIGATOR: J. A. Cape, R. E. Taylor

CONTRACT: AF 33(616)-6794, Atomics International

ABSTRACT: Refinements in the transient thermal property apparatus are described. With these modifications, the apparatus has been used to determine the thermal diffusivity of tungsten boride from about 1300°C to 1600°C. The measured values increase from about 0.054 to 0.058 over this temperature interval.

The techniques and apparatus for measuring the specific heat of brittle conductors by pulse heating are also described. Resistivity and specific heat data for uranium silicide of several compositions are reported. The resistivity and specific heat increased with increasing silicon content. For uranium silicide containing 3.8% silicon, the resistivity increased from 56 micro-ohm-cm at 0°C to 75 micro-ohm-cm at 750°C and for uranium silicide containing 5.9% silicon, the resistivity increased from 81 micro-ohm-cm at 0°C to 111 micro-ohm-cm at 800°C. The specific heat for the 3.8% silicon material is given by $c_p = 3.16 \times 10^{-6} T + 0.0412$ cal/gm-°C from 50° to 430°C, and for the 5.8% silicon material, $c_p = 16.1 \times 10^{-6} T + 0.0455$ cal/gm-°C from 50° to 715°C, where T is in C. The thermal conductivity of titanium carbide was measured over the temperature region 400° to 1200°C. The steady-state radial heat flow method was used. The conductivity varies linearly from 0.088 cal/sec-cm-°C at 500°C to 0.109 cal/sec-cm-°C at 1100°C. These results are in marked contrast to values reported in the literature.

January 1963

SUBJECT: CARBONIZATION OF PLASTICS AND REFRACTORY MATERIALS RESEARCH

INVESTIGATOR: J. A. Coffman, et al.

CONTRACT: AF 33(616)-6841, General Electric Co.

ABSTRACT: Presented in this report are the results of continuing investigations of the Carbonization of Plastics, the Vapor Pressure of Refractory Materials and the Spectral Emissivity of Refractories.

Studies of thermal degradation of phenolic, epoxy, polyphenylene and polynapthalene resins were made, utilizing, as earlier, the arc-image furnace, the fluidized bed reactor and the thermogravimetric analysis technique. The effect of semi-carbonized materials used as fillers has been studied and a Bendix time-of-flight mass spectrometer has been adapted to the study of transient and stable species resulting from the flash-photolysed degradation of plastics. Properties of carbon residues have been measured.

Vapor pressure studies using the Langmuir evaporation technique, the matrix isolation method, and resonance line absorption spectroscopy have progressed. Rates of evaporation of tungsten carbides, hafnium carbide and titanium carbide have been measured; further conclusive measurements on zirconium carbide have been made. Preliminary application of the matrix isolation technique to thoria has been made. Resonance line absorption studies have been completed on zirconium carbide and titanium carbide and the accessible thermodynamic data have been deduced.

Normal spectral emissivities of W-2 coated molybdenum, siliconized ATJ graphite, tantalum, molybdenum, tungsten carbide and zirconium have been measured in the temperature range 1400 to 3100°K.

ASD TR 61-260, Part I, Vol II

May 1962

SUBJECT: THERMODYNAMICS OF CERTAIN REFRACTORY COMPOUNDS

INVESTIGATOR: S. L. Bender, R. E. Dreikorn, et al

CONTRACT: AF 33(616)-7327, Avco Corp.

ABSTRACT: A theoretical and experimental study over the temperature range from 298.15° to 6000°K was made of the thermodynamics of oxides, borides, carbides, and nitrides of the metals in groups IVB, VB, VIB, and VIIB of the Periodic Chart in addition to silicon, boron, scandium, beryllium, magnesium, calcium, strontium, and osmium.

June 1962

SUBJECT: NEW METHOD FOR THE DETERMINATION OF THERMAL CONDUCTIVITIES BETWEEN 1000° AND 3000°C

INVESTIGATOR: Michael Hoch, Donald A. Nitti

CONTRACT: AF 33(616)-7123, University of Cincinnati

ABSTRACT: The heat conduction equation in a finite cylinder, heated by high frequency induction and losing heat only by radiation, has been solved. In the steady state, the cylindrical surface is at a constant temperature, and the flat circular surfaces exhibit a temperature gradient along the radius. This temperature gradient has been analytically correlated to the thermal conductivity. The thermal conductivity of molybdenum has been measured between 2100° and 2400° K; and that of vanadium between 1600° and 1800° K; for molybdenum, $k = -0.191T + 81.0$, for vanadium $k = 0.0320T - 41.4$ BTU/hr-ft-°F.

January 1963

SUBJECT: THERMAL DIFFUSIVITY MEASUREMENTS ON METALS AND CERAMICS AT HIGH TEMPERATURES

INVESTIGATOR: R. L. Rudkin, W. J. Parker, R. J. Jenkins

CONTRACT: MIPR 33(616)-61-7, U. S. Naval Radiological Defense Laboratory

ABSTRACT: The adaption of the NRDL flash method to the measurement of the thermal diffusivity of metals and ceramics at high temperatures is described. A high intensity short duration light pulse from a xenon flash lamp is absorbed in the front surface of a thermally insulated specimen a few millimeters thick and the resultant temperature history of the rear surface is measured by a lead sulfide cell radiation detector or a thermocouple, displayed on an oscilloscope and photographed by a Polaroid Land camera. The thermal diffusivity of the material is determined from this temperature versus time curve provided the theoretical boundary conditions are experimentally satisfied. Measurements of the thermal diffusivity of Armco iron, molybdenum, titanium, zirconia, and alumina have been made up to 1200°C, 1300°C, 1700°C, 1100°C and 1100°C respectively.

August 1962

SUBJECT: THEORY OF THE THERMAL CONDUCTIVITY OF METALS, ALLOYS AND SEMICONDUCTORS

INVESTIGATOR: J. R. Madigan

CONTRACT: AF 33(616)-7374, Borg-Warner Corp.

ABSTRACT: The present status of the theory of thermal conductivity in solid is such that although the processes leading to thermal resistance are understood in a qualitative manner, there is no reliable quantitative theory. To illustrate the successes and failures of the present theory we have calculated the heat conduction in a perfect linear lattice. It is possible to perform the necessary calculations for this model without the danger of becoming lost in a forest of indices. In a perfect crystal of reasonable size phonon-phonon interactions are the only scattering events which determine the thermal resistance and the transition probability vanishes for phonon-phonon scattering in a one-dimensional lattice. We have demonstrated that to simultaneously satisfy the selection rules on the wave vectors and the energy at least one of the wave vectors must be equal to a reciprocal lattice vector (more precisely zero in the one-dimensional case). This causes the transition probability as calculated by first order perturbation theory to vanish for cubic anharmonic terms in the potential energy of the lattice. Thermal conductivity of simple alloys was calculated by assuming additional scattering in the alloy is due to the mass difference of the elements. This "isotope effect" has been used to calculate the thermal conductivity of Si-Ge alloys.

January 1963

SUBJECT: THEORY OF THE THERMAL CONDUCTIVITY OF METALS, ALLOYS, AND SEMICONDUCTORS

INVESTIGATOR: J. R. Madigan

CONTRACT: AF 33(616)-7374, Borg-Warner Corp.

ABSTRACT: The lattice thermal conductivity of simple alloys can be calculated by assuming that the only effects of alloying on the thermal conductivity can be represented by point defect scattering mechanisms. The scattering of phonons by point defects has an inverse relaxation time proportional to the fourth power of the frequency. The proportionality constant depends on the particular scattering mechanism considered. In simple substitutional alloys where one chemically similar element is substituted for another, there are three

scattering processes to be considered besides phonon-phonon scattering. Phonons are scattered by the fluctuation in mass, the change in force constants, and the lattice distortion caused by the impurity atom. The relaxation time for all these processes acting simultaneously has been derived.

The relaxation time for phonon-phonon scattering due to cubic anharmonic terms in the expansion of the potential energy of the crystal in the displacements of the atoms comprising the crystal from equilibrium is proportional to the inverse frequency squared. When the relaxation time for point defect scattering is combined with that for phonon-phonon scattering to obtain an effective relaxation time one may calculate the lattice thermal conductivity as a function of alloy composition. We have done this for the case of Ge-Si alloys and find reasonable agreement with experiment.

The possibility of modifying the theory with the aid of ideas from nonequilibrium statistical mechanics is considered.

ASD TDR 62-269, OTS Release

August 1962

SUBJECT: SUPERCONDUCTIVITY IN METALS AND ALLOYS
INVESTIGATOR: W. H. Cherry, et al.
CONTRACT: AF 33(616)-6405, RCA Laboratories
ABSTRACT: Methods have been developed for the measurement of the interphase energy in superconductors. A description of the method is given along with preliminary results on thin foils and films of tin. Measurements have been made of the thermal contact resistance (Kaptiza resistance) between various solids and liquid helium. These measurements have been made in the normal and superconducting states for lead, tin, and indium, and for the insulator sapphire. Measurements have been made of the transition temperatures in the system $(\text{Nb}, \text{Ta}, \text{V})_3\text{Sn}$. The transition temperatures range from 2.8°K to 18°K and can be related to a simple mass and volume dependence. A new method of preparation of Nb_3Sn has been developed, and is described in detail. With this method it is possible

ASD TDR 62-269 (Continued)

to prepare crystalline Nb₃Sn and to deposit films of Nb₃Sn in various geometries. The application of this technique to the continuous deposition of Nb₃Sn on wire is described and some superconducting properties of this wire are given. Alloying experiments have been made and results indicate ambiguities in the electron-to-atom ratio ascribed to various elements. Resistance measurements on sintered and non-sintered specimens of Nb₃Sn reveal a resistance anomaly near 100°K.

ASD TDR 62-348, OTS Release

July 1962

SUBJECT: A HIGH TEMPERATURE THERMAL CONDUCTIVITY APPARATUS
INVESTIGATOR: R. E. Taylor
CONTRACT: AF 33(657)-7136, Atomics International
ABSTRACT: A high temperature, steady-state thermal conductivity apparatus is described. This apparatus has been used to measure the conductivity of diverse materials, such as graphite, metals, carbides, oxides, and liquid copper. Measurements have been made from 200 to 2500° C. The procedures used, difficulties encountered, limitations, and accuracy are discussed. Results previously obtained with this apparatus on graphite, molybdenum, tantalum, porous carbon, liquid copper, Armco iron, beryllia, magnesia, and titanium carbide are presented graphically and are compared with other literature results.

ASD TDR 62-461, OTS Release

August 1962

SUBJECT: CHARACTERIZATION AND EXTENSION OF SIMULATION CAPABILITIES OF THE LAS HIGH TEMPERATURE ARC FACILITY
INVESTIGATOR: H. Halle, C. F. Price
CONTRACT: AF 33(616)-8115, University of Chicago
ABSTRACT: The operation of an existing subatmospheric plasma arc facility was characterized to assist the user in selecting the proper simulation conditions for materials test application. To this end, eight principal test conditions were established within the operating limits of the arc, and the pertinent parameters describing the thermal environment were determined and tabulated. Subsequently, the hypersonic re-entry conditions corresponding to the thermal

ASD TDR 62-461 (Continued)

environment of the test conditions were computed and plotted on a velocity-altitude chart. In addition, an arc unit with water-cooled, non-consumable electrodes was developed to reduce contamination of the arc plasma. This arc, having double-ring electrode configuration, will generate plasma with a net average enthalpy of 8000 Btu/lb. The final phase of the program concerned testing various materials under different exposure conditions.

ASD TDR 62-608, Part I, OTS Release

November 1962

SUBJECT: THERMAL CONDUCTIVITY OF ANISOTROPIC SOLIDS AT HIGH TEMPERATURES
INVESTIGATOR: M. Hoch, J. Vardi
CONTRACT: AF 33(616)-7123, University of Cincinnati
ABSTRACT: A method has been developed for the determination of the thermal conductivities of anisotropic solids under conditions of two-dimensional, steady-state heat conduction in a cylinder of finite length heated in vacuum by high frequency induction and radiating heat to the surroundings. The method has been used to determine the radial thermal conductivity, k_r , and the axial thermal conductivity, k_z , of molded ZT type and pyrolytic graphite in the temperature range $1200^\circ - 2200^\circ \text{K}$. For ZT type graphite $k_z/k_r = -0.10116 + 2.00191 \times 10^{-4} \times T$ ($1260^\circ \text{K} < T < 2199^\circ \text{K}$); for pyrolytic graphite, $k_z/k_r = 0.0376$ at 1817°K .

ASD TDR 62-633

August 1962

SUBJECT: A HIGH INTENSITY ARC PLASMA TUNNEL
INVESTIGATOR: Edmund J. Rolinski
ABSTRACT: This report describes a design for a 200-kilo-watt high-intensity arc-plasma tunnel. The design is intended to provide maximum versatility both in terms of operating the arc tunnel and in the experiments which would be seen in the tunnel. Specific requirements for arc-tunnel operation are indicated in the design parameters, i. e., power supply, gas supply, cooling water, vacuum, and gas, water, and electrical metering systems.

Shakedown runs of the arc tunnel indicated that the arc plasma is stable for periods of 6 min. Arc-plasma character-

ASD TDR 62-633 (Continued)

ization studies will include determination of heat-flux rates, pressure and velocity profiles, mass flow rates, atomic and molecular species, and temperature of the arc-plasma environment. Heat transfer rates will be correlated for simulation of hypersonic flight conditions. Chemical kinetic studies on the arc plasma are possible future experiments.

ASD TDR 62-765, OTS Release

January 1963

SUBJECT: THE THERMAL PROPERTIES OF TWENTY-SIX SOLID MATERIALS TO 5000°F OR THEIR DESTRUCTION TEMPERATURES

INVESTIGATOR: C. D. Pears, et al.

CONTRACT: AF 33(616)-7319, Southern Research Institute

ABSTRACT: The thermal expansion, heat capacity, thermal conductivity, total normal emittance, electric resistivity, and thermoelectric voltage were investigated for 26 refractory materials, including the borides, carbides, nitrides, oxides, ATJ graphite, tungsten, and alloys of molybdenum and columbium. The temperature range was from 500°F to 5000°F. In addition to these thermophysical properties, the density, chemical analysis before and after temperature exposure, and microscopic pictures before and after temperature exposure, are included to define the materials and assist in the analysis of the data.

ASD TDR 62-916

November 1962

SUBJECT: RESEARCH ON METHOD FOR MEASURING TEMPERATURES BETWEEN 8000° AND 30,000° KELVIN

INVESTIGATOR: Manfred Kopsel, Dr. J. Richter

CONTRACT: AF 61(052)-512, University of Kiel

ABSTRACT: A device is described which permits the measurement of the intensity of spectral lines in the visible spectrum which are emitted from a plasma. Discussion will be made of the conditions under which the measured intensity permits the determination of plasma temperature.

ASD TDR 62-916 (Continued)

For an atmospheric plasma at various pressures, the dependence between the temperature and the intensity will be given for a series of spectral lines. It is shown that in this case a quick temperature determination is possible when the plasma temperature lies between 8000°K and $30,000^{\circ}\text{K}$.

APPLICATION STUDIES

ASD TDR 61-81, Part II

October 1962

SUBJECT: PERFORMANCE CHARACTERISTICS OF HIGH TEMPERATURE MATERIALS EXPOSED TO RE-ENTRY CONDITIONS SIMULATED BY AN AIR-STABILIZED ARC

INVESTIGATOR: L. J. Budnick, H. Halle, C. F. Price

CONTRACT: AF 33(616)-7001, University of Chicago

ABSTRACT: The results obtained from tests of 59 samples of various materials are presented. The samples were exposed to the high-temperature plasma discharge produced in an air-stabilized electric arc. The sample shape, test conditions and test procedure, and the material behavior are reported upon.

ASD TN 61-102

December 1961

SUBJECT: CURRENT VACUUM TECHNOLOGY AND PRACTICE

INVESTIGATOR: M. L. Minges

ABSTRACT: A review of vacuum technology literature is presented. The review includes: 1) a general description of vacuum system components, 2) an outline of the engineering approach to vacuum system design, and 3) a discussion of out-gassing phenomena. This report is an introduction to the vacuum technology field, however, a limited amount of quantitative data is included in selected areas so that it may be of value in preliminary vacuum system design.

ASD TR 61-386, OTS Release

June 1962

SUBJECT: DEVELOPMENT OF NEW AND USEFUL ELEVATED-TEMPERATURE STEELS FOR AIRCRAFT APPLICATIONS

INVESTIGATOR: A. Kasak, V. K. Chandhok, E. J. Dulis

CONTRACT: AF 33(616)-7376, Crucible Steel Co. of America

ABSTRACT: A heat-treatable stainless steel with an outstanding combination of strengths at ambient and elevated temperatures was developed. Of the experimental steels studied, the 0.15C-14.5Cr-

Mo-V-Co type showed the best combination of desired characteristics and properties. Analysis of the effects of the alloying elements show that Mo is an effective room- and elevated-temperature strengthening agent in these steels. The strengthening mechanism is associated with precipitation of a FeMoCr intermetallic compound. On the basis of work on laboratory-sized heats, a nominally 0.15C-14.5Cr-5Mo-0.5V-13.5Co steel (AFC77) was selected for scaling-up for production on mill facilities. It was produced by air-induction and vacuum-arc-remelting methods and processed to bar and sheet products without difficulty. AFC 77 has very high strength (290,000 psi tensile strength) at ambient temperature and retains its strength remarkably well up to about 1200^oF (120,000 psi tensile strength at 1200^oF). It has appreciably higher strength, particularly in prolonged exposures, than any stainless steel known today. The excellent strength properties over a wide range of temperatures are accompanied by good ductility; good formability and weldability are indicated. AFC77 is also resistant to atmospheric corrosion and oxidation.

ASD TDR 62-64, OTS Release

June 1962

SUBJECT: RESEARCH ON MATERIALS AND METHODS
FOR DECONTAMINATION OF TOXIC MISSILE
PROPELLANT SPILLAGE

INVESTIGATOR: J. M. Siegmund, et al

CONTRACT: AF 33(616)-7672, Allied Chemical Corp.

ABSTRACT: Decontaminants for five toxic missile propellants; nitrogen tetroxide, chloride trifluoride, hydrazine, unsymmetrical dimethyl-hydrazine, and pentaborane, were considered for this work.

The work for each propellant is divided into: the practical phase which includes a comprehensive literature survey; the theoretical phase includes a study of possible chemical reactions with respect to probable effectiveness, nature of end products, and heats involved; and laboratory phase where the candidate materials were tested for their neutralizing capability with the various propellants.

The most effective decontaminant for each propellant was developed based on this work; however, if a common de-

ASD TDR 62-64 (Continued)

contaminant for all five propellants is required, dilute aqueous solutions of ammonia appear to offer the best potential.

ASD TDR 62-91, OTS Release

June 1962

SUBJECT: TEST DEVICE FOR THE DYNAMICS EVALUATION OF AIRCRAFT FUEL SYSTEM MATERIALS AND STRUCTURES

INVESTIGATOR: L. G. Middleton

CONTRACT: AF 33(616)-7847, The Boeing Co.

ABSTRACT: A test device that is capable of simulating, on a small scale, the dynamic loads and environmental conditions present in actual aircraft integral fuel tanks was successfully developed and tested. Sixteen integral test tanks were sealed with eight different sealing systems and evaluated on the dynamic test device. Five of the eight sealing systems were considered very satisfactory and judged equal in ability to provide a reliable sealing system. The remaining three systems (a fluorocarbon fillet sealed system and two structural adhesive systems) were considered unsatisfactory because there was excessive leakage.

The effects of a jet fuel anti-icing additive (Phillips Fuel Additive No. 55MB) on the sealing materials used in the test tanks and in laboratory control tests were also evaluated. The anti-icing additive was found to be compatible with all of the sealing materials tested.

ASD TDR 62-415

March 1963

SUBJECT: EVALUATION OF THIN FILM CORROSION INDICATORS

INVESTIGATOR: A. A. Mohaupt

CONTRACT: DO 33(616)-60-9, Forest Products Laboratory

ABSTRACT: Corrosion indicators were developed that consisted of a thin iron film deposited on a plastic substrate under vacuum conditions. The iron film was then treated with chloride and chromate salts of a specific concentration to make the film more sensitive to corrosive conditions.

This report includes the results of an evaluation made at the Forest Products Laboratory to determine the effectiveness of the corrosion indicators under various conditions of temperature and humidity and their performance when used with different preservation methods. The project was divided into the following phases: Phase I -- Comparison of corrosivity of indicators and various metals. Phase II -- Use of indicators with volatile corrosion inhibitors. Phase III -- Indicator performance during dry-down period. Phase IV -- Outdoor shed storage. Phase V -- Packaging recommendations for indicators.

ASD TDR 62-416

March 1963

SUBJECT: DELAYED ACTION, FOAM-IN-PLACE POLYURETHANE FOR USE IN AN AEROSPACE ENVIRONMENT

INVESTIGATOR: S. Schwartz

CONTRACT: AF 33(616)-7925, Hughes Aircraft Co.

ABSTRACT: This report details the materials and techniques developed to meet the requirement of a delayed action, foam-in-place polyurethane for use in an aerospace environment. The methods investigated for preparation of the special polyurethane included a number of chemical blocking reactions, molecular sieves and encapsulation techniques, all designed as restraining agents to inhibit the activity of the isocyanate component. The successful method finally developed utilized the principle of solid components to assure low reactivity and good storability. The optimum compound employed a sterically hindered solid diisocyanate, dianisidine diisocyanate, a solid diol, an ethylene oxide adduct of bisphenol A, a solid triol crosslinker, trimethylol propane, and a solid catalyst, dibutyl tin di-2-ethyl hexoate. The resulting powder mixture was found to have storage stability of at least two months when stored at room temperature. The powder could be activated to produce foams of 2 to 5 pounds per cubic foot density on heating to 175 to 250° F in a vacuum environment. By addition of a water liberating blowing agent, boric acid, foams can also be made under ambient pressure conditions. Structural components made to demonstrate the utility of the powder, consisted of a seven-foot diameter balloon, which using predistributed powder on its surface, was inflated and rigidized at a simulated alti-

ASD TDR 62-416 (Continued)

tude of 150,000 feet, and a full sized man-supporting chair which was also made under the same conditions. In both cases infra-red heat was used as the triggering mechanism.

ASD TDR 62-421, OTS Release

May 1962

SUBJECT: EVALUATION OF COATING SYSTEMS FOR
HIGH STRENGTH, LOW ALLOY STEEL EX-
TERIOR MISSILE AND ROCKET CASINGS

INVESTIGATOR: H. R. Nelson, K. E. Hofer

CONTRACT: AF 33(616)-7739, Armour Research Foundation

ABSTRACT: Existing organic coating systems were evaluated by exposing coated specimens to environments to which missile and rocket casings may be exposed during fabrication, storage, shipment, and readiness. SAE 4340 steel was the substrate material. The protection afforded the substrate was evaluated by direct tests of the coatings or by the change in the performance of the coating or substrate following exposure to various adverse environments.

Sixteen systems were studied. Tension, fatigue, embrittlement, abrasion, adhesion, flexibility, stress-corrosion, thermal change and humidity, accelerated weathering and salt spray corrosion were the tests made.

ASD TDR 62-618

February 1963

SUBJECT: DETERMINATION OF THE EFFECTS OF
PROCESSING REFRACTORY METALS
UNDER VACUUM

INVESTIGATOR: F. R. Cortes

CONTRACT: AF 33(616)-8212, Universal-Cyclops Steel Corp.

ABSTRACT: Ten representative refractory metal alloys were chosen for vacuum rolling studies. The degree and/or the effect of contamination incurred on 90Ta-10W, F-48, D-31, TZM, and Mo+0.5%Ti sheet hot rolled at 0.5 microns and at 100 microns pressure was evaluated over a range of rolling parameters. Data is presented showing qualitative differences in fabricability with pressure of each of these alloys. Mechanical property and metallographic data provide

ASD TDR 62-618 (Continued)

a quantitative indication of vacuum purity levels required to prevent surface contamination or minimize its effect in various alloys. Two alloys - D-41 and W+.6C - could not be successfully processed to starting size sheet for vacuum rolling studies due to the lack of sufficiently developed sheet processing procedures. In addition, technical difficulties prevented the vacuum rolling and evaluation of three tungsten base materials chosen for evaluation.

ASD TDR 62-653

July 1962

SUBJECT: DEVELOPMENT AND INVESTIGATION OF AN
ARC-PLASMA MATERIAL EVALUATION
FACILITY

INVESTIGATOR: H. E. Smith

CONTRACT: AF 33(616)-6198, University of Dayton

ABSTRACT: A facility and a standard test procedure have been developed to predict gross differences in the behavior of materials exposed to a high temperature environment. Material samples may be exposed to a simulated air stream with total enthalpy values ranging from 600 to 4000 BTU/lb. at heat flux levels from 100 to 500 BTU/ft²-sec. Techniques and instrumentation used to determine properties of the discharge stream are described and results of a temperature and pressure survey of the stream are presented. Test results of some typical high temperature materials are also presented.

ASD TDR 62-655

July 1962

SUBJECT: THE EVALUATION OF HIGH TEMPERATURE
MATERIALS SYSTEMS WITH AN ARC-PLASMA-
JET

INVESTIGATOR: H. E. Smith, J. C. Wurst

CONTRACT: AF 33(616)-7838, University of Dayton

ABSTRACT: A small arc-plasma-jet was suitably modified and utilized for the evaluation and screening of high temperature materials at heat flux levels ranging from 10 to 500 Btu/ft²-sec. A "standard" evaluation procedure was employed to characterize a number of systems representing ablating, insulating, and heat sink type materials. Results of these tests are presented in terms of

ASD TDR 62-655 (Continued)

weight loss, density change, depth and volume of erosion, and front and back surface temperatures.

A metallographic analysis of coating failures in the XLR-99 thrust chamber of the X-15 research aircraft and the subsequent development of a thermal shock test with the plasma-jet for sprayed ceramic coatings are discussed. The results of an extensive evaluation of protective coatings and the field test verification of these results are presented.

DESIGN CRITERIA

WADC TR 56-645, Part VII, OTS Release

August 1962

SUBJECT: PROPERTIES OF GLASSES AT ELEVATED TEMPERATURES

INVESTIGATOR: M. J. Kerper

CONTRACT: AF 33(616)-59-4, National Bureau of Standards

ABSTRACT: A program was initiated to investigate the physical properties of several glasses that are candidates for glazing flight vehicles. The objectives of the program were: 1) develop suitable test methods for determining the desired physical properties at room and elevated temperatures, and 2) determining the values of the desired physical properties of individual glasses over a wide temperature range.

This report contains a study and interpretation of several factors associated with the determination of Young's modulus and the modulus of rupture. The tests were performed on seven commercially available glasses and were conducted from room temperature to several degrees above their strain points.

WADC TR 59-762, Part IV, OTS Release

August 1962

SUBJECT: ULTRA-SHORT-TIME CREEP RUPTURE

INVESTIGATOR: J. P. Knight, W. A. Cosby, H. W. Leavenworth

CONTRACT: AF 33(616)-7632, American Machine and Foundry Co.

ABSTRACT: This program involved the design and fabrication of ultra-short-time creep test equipment and utilization of the equipment for determining the short time mechanical properties of a structural refractory metal sheet alloy at elevated temperatures. Tensile test specimens of National Research Corporation Ta-10%W alloy were strained at temperatures of 2000^oF, 2500^oF, and 3000^cF in an argon atmosphere and in a vacuum (3000^oF only). Heating of test specimens to maximum temperature was accomplished in approximately 60 milliseconds. Tensile loading of test specimens at each temperature was accomplished prior to attaining test temperature and 1/2 second, 2.0 seconds, and 5.0 seconds after attaining test temperature. A description of test apparatus, test procedures and complete test data are given by this report.

WADD TR 61-96

November 1961

SUBJECT: PROPERTIES OF INORGANIC ENERGY-CONVERSION AND HEAT-TRANSFER FLUIDS FOR SPACE APPLICATIONS

INVESTIGATOR: W. D. Weatherford, Jr., et al.

CONTRACT: AF 33(616)-7206, Southwest Research Institute

ABSTRACT: This report is a complete revision of WADC TR 59-598. It is intended to serve as a properties handbook for various inorganic fluids which may have potential value as energy-conversion or heat-transfer fluids for space applications. The fluids are presented as three distinct classes - namely, liquid metals, nonmetals, and gases. The liquid metals include mercury, cesium, rubidium, potassium, NaK(78), sodium, lithium, bismuth, and lead. The nonmetals include aluminum bromide, sulfur, and lithium hydride. The gases include argon, helium, and hydrogen.

Data are presented, where available, up to temperatures ranging from 2300° F for mercury to 4500° F for lead, and for pressures ranging from less than one atmosphere to greater than twenty atmospheres. The enumerated properties include vapor pressure, density, viscosity, surface tension, electrical resistivity, thermal conductivity, specific heat, latent heats, enthalpy-entropy relationships, melting point, critical properties, dielectric constant, ionization potential, magnetic susceptibility, thermal neutron cross sections, and corrosion characteristics.

The characteristics of the various fluids are discussed, and the recommended values for the fluid properties are presented in either tabular or graphical form, or both, with detailed documentation as to basis and source. In addition, background material, including thermodynamic-cycle, heat-transfer, compatibility, and working fluid considerations is discussed. A summary of current research activities in this field is presented.

WADD TR 61-181, OTS Release

August 1961

SUBJECT: SUBSTRUCTURE AND MECHANICAL PROPERTIES OF REFRACTORY METALS

INVESTIGATOR: B. S. Lement, et al

CONTRACT: AF 33(616)-6838, Manufacturing Labs., Inc.

WADC TR 53-373 Sup 10

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WADD TR 61-181 (Continued)

ABSTRACT: Coordinated program on substructural characteristics of W, Mo, Ta and Cb was carried out by Man. Labs., M.I. T., Rutgers, U. Liverpool (England) and U. Cambridge (England) by means of advanced microscopic, x-ray diffraction, and mechanical testing techniques. Changes in subboundary spacing, particle size, and r.m.s. strain have been measured after annealing. Various orders of substructure in W were identified in terms of size, disorientation and dislocation arrays. Alterations of substructure in annealed W, Ta and Cb were traced by thin-film electron microscopy. Stacking faults and twins were found in high-temperature annealed W and Cb. Fiberling in b.c.c. wires was found to involve complex interfolding of distorted grains and deformation bands, resulting in a reduction in transverse boundary sites for initiation of brittle fracture. In Ta and Cb, the lattice friction stress was shown to be strongly dependent on test temperature, impurity content, and substructure. The latter continues to vary on annealing above the recrystallization temperature, which can account for the anomalous values of the Cottrell locking force reported for Cb.

WADD TR 61-199

August 1962

SUBJECT: CREEP-RUPTURE PROPERTIES OF SIX
ELEVATED TEMPERATURE ALLOYS

INVESTIGATOR: J. G. McBride, B. Mulhern, R. Widmer

CONTRACT: AF 33(616)-6200, New England Materials
Laboratory, Inc.

ABSTRACT: Room temperature tensile properties, short time elevated temperature properties, and elevated temperature creep-rupture properties were determined for six widely used elevated temperature alloys. Representative commercial lots of Udimet 700 (Bar), R-235 (Bar), GMR-235 (Cast Bar), Rene '41 (Sheet), R-235 (Sheet), and Nicrotung (Cast Bar) were tested at each of three typical application temperatures. Tabulated tensile and creep-rupture data, stress versus rupture-life curves, creep versus time curves, and stress versus time to 0.2 and 1.0% total creep curves are presented.

June 1962

SUBJECT: DETERMINATION OF THE EFFECTS OF ELEVATED TEMPERATURE MATERIALS PROPERTIES OF SEVERAL HIGH TEMPERATURE ALLOYS

INVESTIGATOR: M. M. Lemcoe, A. Trevino

CONTRACT: AF 33(616)-7056, Southwest Research Institute

ABSTRACT: This investigation was conducted to determine mechanical properties of several high performance alloys at room and elevated temperatures. The effects of temperature (up to 1900°F) and exposure (up to 1000 hours) at temperature on the tensile, compressive, bearing and shear properties were determined from measured stress-strain information in both the elastic and plastic range. The following five materials were considered in the program:

1. 301 extra hard stainless steel
2. Ph15-7Mo (TH 1050)
3. AM 355
4. Rene 41
5. N-155

All material was from 0.050-inch sheets, except the material for the 1/8-inch diameter shear pins, which were fabricated from 1/4-inch plate. Heat treatment was in accordance with existing specifications for the materials, or other procedures approved by ASD to develop the optimum strength properties.

Descriptions of the test specimens, equipment, and procedures used are included. Test results are reported in tables and in curves showing the effects of temperature and time on the various mechanical properties.

June 1962

SUBJECT: PROPERTIES OF PLASMA SPRAYED MATERIALS

INVESTIGATOR: M. A. Leinstein

CONTRACT: AF 33(616)-6376, General Electric Co.

ABSTRACT: A detailed investigation of the plasma spraying of tungsten and several refractory carbides was conducted in a controlled environment tank. Optimum parameters for the spraying of these

ASD TDR 62-201 (Continued)

materials were established. The refractory carbides included CbC, ZrC, HfC, TaC, and several binary carbides. The spraying of mixed oxides was studied both in the controlled environment tank and in air. Various properties for these materials were determined as applicable, tensile strength, yield strength, elongation, transverse rupture strength, bending strength, bending ductility, melting point, density, crystallographic structure, chemistry, coefficient of expansion, specific heat, oxidation resistance, and microstructure. The feasibility of spraying in a controlled environment is evaluated.

ASD TDR 62-215

July 1962

SUBJECT: THERMOPHYSICAL PROPERTIES OF THERMAL INSULATING MATERIALS
INVESTIGATOR: C. E. Moeller, et al
CONTRACT: AF 33(616)-7875, Midwest Research Institute
ABSTRACT: This report is a compilation of thermophysical property data for insulating materials, for both cryogenic and high temperature applications. Thermal conductivity, linear thermal expansion, specific heat, total emissivity, thermal diffusivity, and compressive strengths are plotted with respect to temperature. Density, melting point, continuous service temperature, typical available form, and modulus of elasticity are given in tabular form. Material composition and methods by which the data were obtained are also included.

ASD TDR 62-230, OTS Release

August 1962

SUBJECT: DETERMINATION OF ENGINEERING PROPERTIES OF MAR-STRAINED STEELS
INVESTIGATOR: R. E. Yount
CONTRACT: AF 33(616)-7655, General Electric Co.
ABSTRACT: The Mar-Strain response (the addition of strain and aging to quenched and tempered steels) was determined for eight alloys representing four classes of steel: low alloy martensitics, secondary hardening martensitics (hot work die steels), semi-austenitic and martensitic stainless. All four alloy classes responded to the process by demonstrating yield strength increases of 10 - 20%. It was found that the tempered structure and strain hardening characteristics were the most significant factors controlling the Mar-Strain response.

ASD TDR 62-230 (Continued)

Two alloys were selected for determination of their engineering properties including uniaxial tensile, fatigue and center notch properties and biaxial performance in sub-scale cylinder tests. One alloy, Ladish D6AC (.46%C, 1%Cr, 1%Mo), was capable of being MarStrained to a 275,000 psi .2% yield strength. A second alloy, Modified S-5 (.48%C, 2%Si, .5%Mo, .25%V), was capable of being Mar-Strained to a 300,000 psi .2% yield strength. Mar-Straining was found to increase the fatigue strength of both alloys. The process was adequately demonstrated in the sub-scale pressure vessel tests. Burst strengths (greater than 350,000 psi hoop) achieved with the Mar-Strained S-5 cylinders were higher than any previously reported for a homogeneous material.

ASD TDR 62-315, OTS Release

May 1962

SUBJECT: AN AUTOMATIC RECORDING DILATOMETER
FOR THERMAL EXPANSION MEASUREMENTS
TO 2000°F

INVESTIGATOR: G. L. Denman

ABSTRACT: The design, construction, and calibration of an automatic recording dilatometer capable of accurately measuring thermal expansion properties of solid materials from room temperature to 2000°F is discussed.

The dilatometer was of the quartz tube differential expansion type with auxiliary equipment capable of continuous and automatic recording of thermal expansion data. A statistical error analysis is included which indicates that an accuracy of $\pm 1\%$ can be obtained with the instrument.

ASD TDR 62-335, Vol I, OTS Release

December 1962

SUBJECT: DETERMINATION OF DESIGN DATA FOR HEAT
TREATED TITANIUM ALLOY SHEET VOL I.
Summary of Mechanical and Physical Property
Data Collected, Including Tensile Creep and
Fatigue

INVESTIGATOR: P. J. Hughes

ASD TDR 62-335, (Continued)

CONTRACT: AF 33(616)-6346, Lockheed-Georgia Co.
ABSTRACT: Mechanical and physical property data, necessary to fulfill the requirements of Phase II of the Department of Defense Titanium Alloy Sheet Rolling Program, were obtained for selected solution treated and aged titanium alloys in sheet form.

Four alloys were investigated: B120VCA (Ti-13V-11Cr-3Al), Ti-6Al-4V, Ti-2.5Al-16V and Ti-4Al-3Mo-1V. They were supplied by the producers in the heat treated condition from three or more heats and three thicknesses of each alloy. Static mechanical property data for tension, compression, bearing, shear and crippling; creep and rupture data for tension, compression, bearing and shear; and axial-load fatigue data were obtained at room and elevated temperatures. Fastener and weld joint data from -320°F to 80°F and physical properties from -420°F to 1200°F were obtained.

Volume I summarizes mechanical and physical properties in a form consistent with those given in MIL-HDBK-5.

ASD TDR 62-335, Vol IIa, OTS Release

December 1962

SUBJECT: DETERMINATION OF DESIGN DATA FOR HEAT TREATED TITANIUM ALLOY SHEET VOL 2a
Details of Data Collection Program. Test Techniques and Results for Tension, Compression, Bearing, Shear, Crippling, Joints, and Physical Properties

INVESTIGATOR: W. M. McGee, B. R. Matthews
CONTRACT: AF 33(616)-6346, Lockheed-Georgia Co.
ABSTRACT: Mechanical and physical property data, necessary to fulfill the requirements of Phase II of the Department of Defense Titanium Alloy Sheet Rolling Program, were obtained for selected solution treated and aged titanium alloys in sheet form.

Four alloys were investigated: B120VCA(Ti-13V-11Cr-3Al), Ti-6Al-4V, Ti-2.5Al-16V and Ti-4Al-3Mo-1V. They were supplied by the producers in the heat treated condition from three or more heats and three thicknesses of each alloy. Static mechanical property data for tension, compression, bearing, shear and crippling;

ASD TDR 62-335 (Continued)

creep and rupture data for tension, compression, bearing and shear; and axial-load fatigue data were obtained at room and elevated temperatures. Fastener and weld joint data from -320°F to 80°F and physical properties from -420°F to 1200°F were obtained.

Volume 1 summarizes mechanical and physical properties in a form consistent with those given in MIL-HDBK-5. Experimental procedures and test results for static mechanical properties and physical properties are reported in Volume 2a. Volume 2b contains procedures and results for creep and fatigue tests and Volume 3 is a tabular compilation of all data obtained in the program.

ASD TDR 62-335, Vol IIb, OTS Release

December 1962

SUBJECT: DETERMINATION OF DESIGN DATA FOR
HEAT TREATED TITANIUM ALLOY SHEET
VOL IIb. Test Techniques and Results for
Creep and Fatigue

INVESTIGATOR: D. L. White, H. T. Watson

CONTRACT: AF 33(616)-6346, Lockheed-Georgia Co.

ABSTRACT: Mechanical and physical property data, necessary to fulfill the requirements of Phase II of the Department of Defense Titanium Alloy Sheet Rolling Program, were obtained for selected solution treated and aged titanium alloys in sheet form.

Four alloys were investigated: B120VCA (Ti-13V-11Cr-3Al), Ti-6Al-4V, Ti-2.5Al-16V and Ti-4Al-3Mo-1V. They were supplied by the producers in the heat treated condition from three or more heats and three thicknesses of each alloy. Static mechanical property data for tension, compression, bearing, shear and crippling; creep and rupture data for tension, compression, bearing and shear; and axial-load fatigue data were obtained at room and elevated temperatures. Fastener and weld joint data from -320°F to 80°F and physical properties from -420°F to 1200°F were obtained.

Volume 1 summarizes mechanical and physical properties in a form consistent with those given in MIL-HDBK-5. Experimental procedures and test results for static mechanical properties and physical properties are reported in Volume 2a. Volume 2b contains procedures and results for creep and fatigue tests and Volume 3 is a tabular compilation of all data obtained in the program.

December 1962

SUBJECT: DETERMINATION OF DESIGN DATA FOR
HEAT TREATED TITANIUM ALLOY SHEET
VOL III Tables of Data Collected

INVESTIGATOR: D. G. Cumro

CONTRACT: AF 33(616)-6346, Lockheed-Georgia Co.

ABSTRACT: Mechanical and physical property data,
necessary to fulfill the requirements of Phase II of the Department
of Defense Titanium Alloy Sheet Rolling Program, were obtained for
selected solution treated and aged titanium alloys in sheet form.

Four alloys were investigated: B120VCA (Ti-13V-11Cr-3Al), Ti-6Al-4V, Ti-2.5Al-1.6V and Ti-4Al-3Mo-1V. They were supplied by the producers in the heat treated condition from three or more heats and three thicknesses of each alloy. Static mechanical property data for tension, compression, bearing, shear and crippling; creep and rupture data for tension, compression, bearing and shear; and axial-load fatigue data were obtained at room and elevated temperatures. Fastener and weld joint data from -320°F to 80°F and physical properties from -420°F to 1200°F were obtained.

Volume 1 summarizes mechanical and physical properties in a form consistent with those given in MIL-HDBK-5. Experimental procedures and test results for static mechanical properties and physical properties are reported in Volume 2a. Volume 2b contains procedures and results for creep and fatigue tests and Volume 3 is a tabular compilation of all data obtained in the program.

May 1962

SUBJECT: ELEVATED TEMPERATURE CREEP
PROPERTIES OF FOUR MARTENSITIC
HIGH STRENGTH STEELS IN SHEET FORM

INVESTIGATOR: V. F. Lardenoit

CONTRACT: AF 33(616)-6202, Metcut Research Assoc.

ABSTRACT: This program was conducted to determine the
creep properties of four martensitic steels in sheet form. Materials
tested included Unimach 2 (Thermold J) and Potomac M, both of which
are Hot Work Tool Steels, USS 12MoV, a Martensitic Stainless Steel
and 17-22A (S), a Low Alloy Martensitic Steel. Data includes ultimate

ASD TDR 62-358 (Continued)

tensile strength, tensile yield strength and creep rupture properties. The data are presented in tabular and graphical form depicting creep deformations of 0.05%, 0.10%, 0.30%, 0.50% and 1.0% in times ranging from 10 to 1000 hours. Where available, rupture data is also presented.

ASD TDR 62-394, OTS Release

July 1962

SUBJECT: ELEVATED TEMPERATURE CREEP
PROPERTIES OF 17-7PH AND A-286
STAINLESS STEEL

INVESTIGATOR: M. Knight

ABSTRACT: The creep properties for 17-7PH stainless steel were investigated at room temperature, 600°, 800°, and 900°F. The same properties were investigated for A-286 stainless steel at 1200°, 1350°, and 1500°F. The data includes ultimate tensile strength, tensile yield strength, elongation, creep deformation, and creep rupture properties..

ASD TDR 62-401, OTS Release

July 1962

SUBJECT: BIAXIAL STRESS AND STRAIN DATA ON
HIGH STRENGTH ALLOYS FOR DESIGN OF
PRESSURIZED COMPONENTS

INVESTIGATOR: E. L. Terry, S. W. McClaren

CONTRACT: AF 33(616)-7720, Chance Vought Corp.

ABSTRACT: A cross shaped specimen was developed for generating complete biaxial stress-strain curves under 1:1 and 2:1 biaxial tension stress ratio loading. Tests on several materials have shown that the specimen has good reliability.

The influence of strength level on the behavior of the 5CrMoV steel under biaxial loading was investigated. These tests showed that by lowering the uniaxial strength level from 280 to 260 ksi, the shattering type failure observed at the 280 ksi level ceased to exist. However, the biaxial failure strains did not increase as the strength level was decreased.

ASD TDR 62-401 (Continued)

Good correlation was obtained between the failure stresses from the pressure vessels and the biaxial specimens.

The data is presented in a form which can be used directly in the design of biaxially loaded components. The test materials are ranked according to the efficiency parameters "biaxially ductility rating", "resistance to crack-like flaws", and "biaxial strength weight".

ASD TDR 62-480, OTS Release

August 1962

SUBJECT: FATIGUE AND DYNAMIC CREEP OF HIGH-STRENGTH STEELS
INVESTIGATOR: R. F. Brodrick
CONTRACT: AF 33(616)-6946, Lessells and Associates, Inc.
ABSTRACT: A program was conducted to obtain detailed tensile, stress rupture and fatigue data on a series of high-strength steels. Data were obtained from D6AC, LaBelle HT, Thermold J, Vascojet 1000 and Peerless 56, heat treated to nominal ultimate strength of 280,000 psi.

Tests were conducted at room temperature and at elevated temperatures, the particular temperatures being selected according to the material. Maximum test temperature was 1000°F.

Dynamic creep data were obtained in conjunction with the fatigue tests.

ASD TDR 62-524, OTS Release

July 1962

SUBJECT: TENSILE AND CREEP PROPERTIES OF Al10-AT Titanium Sheet Material at Elevated Temperatures
INVESTIGATOR: J. O. Hachet, E. L. Horne
ABSTRACT: This investigation was made to determine creep properties at elevated temperatures of hot-rolled annealed sheet Al10-AT titanium alloy.

ASD TDR 62-524(Continued)

Tests were conducted at 800°, 1000°, and 1100°F on sheet specimens cut parallel to the direction of rolling. Differences in properties with respect to chemical composition of the material from two sources were small. The data includes ultimate tensile, tensile yield strength, and creep properties at each temperature.

Ultimate tensile strengths varied from 132,000 psi at room temperature to 62,300 psi at 1100°F. Creep deformation becomes more rapid at the two higher temperatures indicating that 1000°F is close to the upper useful temperature limit of this alloy.

There was marked scatter of the creep properties of the alloy at 1000° and 1100°F, but the average strength properties are good.

ASD TR 62-656

July 1962

SUBJECT: THERMAL PROTECTION SYSTEMS
INVESTIGATOR: H. Hurwicz, R. Mascola
CONTRACT: AF 33(616)-7483, Avco Corp.
ABSTRACT: Pertinent factors affecting the function of thermal protection systems are reviewed with emphasis on glide re-entry applications. Review is made and new information is generated in the areas of: (1) system, material, and design performance criteria needed for evaluation, (2) methods and techniques necessary for experimental and theoretical acquisition of the necessary parameters, (3) correlation of data to present information for the material selection and development effort, and protection system design use, (4) design and development techniques for built-up shield - structure components (vehicle sizing), and (5) basic mechanisms of heat and mass transfer associated with the mode of heat absorption or dissipation and of load carrying and transmission capacity.

Selection of significant parameters for materials and overall thermal protection systems is made. Parametric studies are conducted to provide information for the designer and materials developer for several systems of interest, while correlation methods and techniques are developed. Effort is devoted to the examination of Q* concept for radiation and radiation ablation systems. Experimental

techniques and facilities are improved and modified and experiments are conducted to permit meaningful determination of performance criteria needed for materials ranking, and to extend the knowledge of material behavior. A rapid inexpensive and accurate thermal design method is developed for sizing and parametric studies. Environmental factors and structural effects in the aero-thermo-structural interface are studied. Design and development techniques are established or clarified for initial sizing of the vehicle and methods provided for final design. The dependence of the thermal protection system design on aerodynamic environment for a flight corridor and a range of load carrying substructures is shown, while desirable material characteristics to strive for in material development effort may be deduced from the parametric studies.

Systematic means for material and system selection are provided for radiative system, radiative systems with subsidiary mass transfer (ablation), and for combinations of above systems with forced backface cooling. Forced transpiration system (mass injection) and plastic impregnated matrix are investigated.

ASD TDR 62-830

February 1963

SUBJECT: MECHANICAL PROPERTIES OF MATERIALS
FABRICATED BY SHEAR FORMING

INVESTIGATOR: F. Jacobs

CONTRACT: AF 33(616)-7874, Temco Electronics & Missiles
Co.

ABSTRACT: The effect of shear forming on the mechanical properties was determined for thirty-two alloys. The eight different classes of alloys included: (a) austenitic, ferritic, martensitic and precipitation hardening types of stainless steels, (b) super alloys, (c) titanium alloys, (d) alloy steels, and (e) aluminum alloys. All materials were shear formed into cones with room temperature reductions of 20, 30, 40, 50, 60 and 70 percent. Some alloys did not withstand the higher percent reductions. As received material properties, properties after shear forming plus heat treatment were determined. Tensile, shear and hardness tests were performed. The typical microstructures of the various materials and conditions were recorded.

December 1962

SUBJECT: LITERATURE SURVEY ON SYNTHESIS,
PROPERTIES, and APPLICATIONS of SELECTED
BORIDE COMPOUNDS

INVESTIGATOR: B. R. Emrich

ABSTRACT: A comprehensive review of the literature was made to assist in providing background information needed for future work concerning boride materials. The materials reviewed included solid bodies of TiB_2 , ZrB_2 , HfB_2 , VB_2 , NbB_2 , TaB_2 , CrB_2 , and ThB_4 . This compilation presents information on synthesis, properties, and applications of the selected boride compounds, including selected abstracts and articles.

This documentary search, by showing many gaps and wide scatter where information is available, demonstrates the need for the development of authoritative scientific information on borides applicable to future technological requirements.

ASD TDR 62-906

February 1963

SUBJECT: TABLES OF THERMOPHYSICAL PROPERTIES
OF MATERIALS

INVESTIGATOR: Y. S. Touloukian

CONTRACT: AF 33(616)-7617

ABSTRACT: This final report covers work on a continuing systematic program involving the search, collection, extraction, evaluation, correlation, interpolation, extrapolation, and the final preparation of tables of properties referred to as "most probable" values. The report does not contain the completed data sheets (11" x 17" in size) but does reproduce in the Appendix up-to-date Tables of Contents of Volumes I, II, and III of the Data Books in which the loose-leaf data sheets are organized. The works reported on consist of the thermal conductivity and viscosity of gases, and the thermal conductivity of metallic and nonmetallic elements, their alloys and their compounds. The data sheets are released twice annually, in June and in December of each year. Current sheets are obtainable through ASRCM-1, and complete sets are available from TPRC only.

ASD TDR 62-1003

March 1963

SUBJECT: FATIGUE PROPERTIES OF SOME ALUMINUM AND STAINLESS STEEL SANDWICH CONSTRUCTIONS

INVESTIGATOR: P. M. Jenkinson, E. W. Kuenzi

CONTRACT: AF 33(616)-61-06, Forest Products Laboratory

ABSTRACT: A few specimens from four aluminum and three stainless steel sandwich constructions were evaluated in fatigue. All constructions were subjected to edgewise compression fatigue, three to flatwise shear fatigue, and one aluminum construction to flatwise tension fatigue.

At 30 million cycles, most of the constructions had a fatigue strength in edgewise compression based on maximum specimen stress above 70 percent of the static strength. One stainless steel (17-7PH(TH1050) sandwich construction was about 50 percent. The fatigue strength at 30 million cycles in shear was found to be about 25 percent of the control strength for two constructions and about 10 percent for the 17-7PH(TH1050) stainless steel construction. The fatigue strength at 30 million cycles in flatwise tension for one aluminum sandwich construction was about 25 percent of control strength.

ASD TDR 62-1004

December 1962

SUBJECT: FURTHER INVESTIGATION OF NOTCH SENSITIVITY OF REFRACTORY METALS

CONTRACT: AF 33(616)-7604, Battelle Memorial Institute

ABSTRACT: Molybdenum and Mo-0.5Ti alloy with various degrees of cold work and different recrystallized grain sizes were tested to determine their low-temperature properties. Sufficient cold work (25 per cent; about 68 per cent is sufficient) markedly reduced the ductile-to-brittle transition temperature of both materials, whereas increasing the recrystallized grain size increased this parameter. Mechanical notches resulted in pronounced degradation of properties in fibered structures, but were much less detrimental to recrystallized structure. This behavior was attributed to the controlling (detrimental) effect of grain boundaries in the fracture of recrystallized molybdenum or Mo-0.5Ti, regardless of material geometry. The fracture toughness of unalloyed molybdenum, tungsten, tantalum, and columbium was studied over a range of temperatures chosen to include the ductile-

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brittle transition. Sheet specimens, 2 x 8 x 0.050 inc, containing sharp central notches were employed. Both wrought and recrystallized conditions were examined for each material. The effect of specimen orientation relative to the rolling direction was studied for molybdenum. Where possible, results were reported in terms of the fracture toughness parameters K_{Ic} and K_{Ic} . In an effort to provide some basic rationale for the behavior of a brittle material in the presence of notches, a model of an idealized partially relaxed crack was adopted. Using theoretical values of the stress at the tip of such a crack, the resultant macroscopic strain was calculated by means of parameters relating to dislocation density and velocity.

ASD TDR 62-1109, OTS Release

January 1963

SUBJECT: STUDIES OF DESIGN CRITERIA FOR WELDED STRUCTURES SUBJECTED TO A BIAXIAL STRESS FIELD

INVESTIGATOR: B. L. Baird

CONTRACT: AF 33(657)-8595, Bruce L. Baird, Inc.

ABSTRACT: Parent and weld metal biaxial stress-strain data were obtained in a 2:1 tension-tension stress field from 4 steels and 1 ti. alloy at 3 test temperatures. Steels were oil hardening, air hardening, and precipitation hardening types. Ti. was all-beta alloy. Honed and ground tubular specimens containing 2 longitudinal seam welds were tested, using strain gage rosettes attached in both weld and parent metal zones to develop bi-axial stress-strain relationships. Values of Fabrication Stress Concentration Factors were obtained from burst testing welded vessels made from the test materials. Use of the bi-axial stress-strain data in combination with Fabrication Stress Concentration Factor values in the design of high strength pressure vessels is illustrated at the end of the text.

INFORMATION PROCESSING

ASD TDR 62-539

February 1963

SUBJECT: ELECTRICAL AND ELECTRONIC PROPERTIES
OF MATERIALS INFORMATION RETRIEVAL
PROGRAM

INVESTIGATOR: H. T. Johnson, E. Schafer, E. M. Wallace

CONTRACT: AF 33(616)-8438, Hughes Aircraft Co.

ABSTRACT: A documentation system has been established for abstracting, indexing and retrieving data on the electrical and electronic properties of insulator and semi-conductor materials. The system is a coordinate index type immediately usable as a manual system and adaptable to machine methods of data storage and retrieval. The data acquired and indexed is published in the form of property tables, data sheets, and summary reviews. Methods used in the documentation, compilation, and evaluation of the data are described and examples of format illustrated. Appended are an index to semiconductor and insulation material names and a glossary of selected properties and symbols.

ASD TDR 62-630

April 1963

SUBJECT: INFORMATION PROCESSING FOR MATERIALS
ENVIRONMENTAL CAPABILITIES - CHEMICAL
PROPERTIES

INVESTIGATOR: J. Gasser, W. E. Few

CONTRACT: AF 33(616)-6288, Chemical & Metallurgical
Research, Inc.

ABSTRACT: A survey and study has determined the scope and direction of needed information processing in the field of chemical properties of materials required for aerospace structures. Seventy-four priority environmental situations, encompassing nine important reaction types, were identified. Needed and timely information processing of specific chemical property data for leading material class candidates were resolved.

In addition, the survey and study revealed deficiencies in data available, data being generated, test methods, and information processing.

SUBJECT: DEVELOPMENT OF A MATERIALS PROPERTY
DATA PROCESSING SYSTEM
INVESTIGATOR: R. C. Braden, C. S. Wright
CONTRACT: AF 33(616)-7238, Belfour Engineering Co.
ABSTRACT: This report discusses a mechanical properties
information system including the operation of a fatigue of metals sub-
system and the design-development and initial operation of other
mechanical properties sub-systems. These sub-systems, employing
punched card equipment and techniques, actively provide mechanical
properties data and associated descriptive information of metals and
reinforced plastics.

The System, sponsored by the USAF, is intended
primarily for the use of Defense Agencies and their contractors.

Formats, codes and procedures utilized to store,
retrieve and display mechanical properties of these materials are out-
lined.

CHEMICAL ENGINEERING

ASD TR 62-7-654, Vol I

March 1962

SUBJECT: HIGH TEMPERATURE AIRCRAFT WIND-SHIELD DEVELOPMENT PROGRAM

INVESTIGATOR: A. F. Shoemaker

CONTRACT: AF 33(600)-36852, Corning Glass Works

ABSTRACT: Glasses having high thermal shock resistance can be made, processed and assembled such that they perform with great reliability as the transparencies in high performance manned flight vehicles. Use of glass as a structural material is feasible through use of bonded edge members.

Specific glasses studied, i. e. Code 1723 Alumino-Silicate, Code 7900 Vycor Brand and Code 7940 Fused Silica, are readily manufactured by standard rolling and/or casting procedures.

The rolled or cast glass can be reduced to required thicknesses and contours having tolerances within $\pm .005$. Surface finish on sizes up to 50 x 30 inches will meet or better 2N deviation.

Contours are reproduced automatically from cams. Various edge shapes can be accomplished by the same process.

Tempering of the alumino-silicate glass produces strength values as high as 25,000 psi MOR abraded. Warp on both thick and thin panels in sizes 50 x 30 inches is less than .002.

Electrical conductive coatings can be applied to a variety of unsymmetrical shapes having coverage over the entire panel and resulting in heat distribution which is defined by "K" factor measurements. Representative values from a large triangular shape are $K_H = 1.3$, $K_A = .9$, $K_M = .75$.

Laminating of Pyrex Brand glasses including Code 1723 alumino-silicate type can be done using both P. V. B. and Silastic-K interlayer material. Parting medium designs have been accomplished which practically eliminate failure due to cold temperature soak.

Press and sag forming of polished flat panels has been accomplished. Precision contours are obtained because of intimate contact with a full mold. Optical characteristics need not be impaired due to the use of glass cover plates.

ASD TR 62-7-654, Vol II

March 1962

SUBJECT: HIGH TEMPERATURE AIRCRAFT WIND-SHIELD DEVELOPMENT PROGRAM
INVESTIGATOR: A. F. Shoemaker
CONTRACT: AF 33(600)-36852, Corning Glass Works
ABSTRACT: Glasses having high thermal shock resistance can be made, processed and assembled such that they perform with great reliability as the transparencies in high performance manned flight vehicles. Use of glass as a structural material is feasible through use of bonded edge members.

Three types of mounting systems were developed and studied. The same panel shape was used for each system, i. e. similar to that of the F-102 windshield. Requirements and design criteria stipulated for the development were different from those used on the actual windshield.

Type I system involved a floating flush mount for use on the outer most panel of a multi panel unit. Temperatures up to 900° F with pressure differentials of 10 psi were specified. Major emphasis was placed on flushness of surfaces.

Type II system was concerned with edge clamping such that no edge fixity was attained. This was to be adopted as the attachment for an inner laminated panel of a multi panel unit. Temperatures of 500° F and pressure differentials of 15 psi were specified.

Type III system was based on the bonding of metal edge member to the glass such that tensile loads could be transmitted through the panel and not have to be directed around it. Temperatures of 500° F with pressure differential of 15 psi and tensile loading of 300 pounds per inch or better were specified.

ASD TDR 62-7-665, OTS Release

March 1962

SUBJECT: TOXICITY OF BERYLLIUM
INVESTIGATOR: J. Cholak, et al.
CONTRACT: AF 33(600)-37211, University of Cincinnati
ABSTRACT: Report on toxicity of beryllium and a guide for those responsible for protecting the health of personnel engaged in operations in which beryllium is used.

ASD TR 7-880b(IV)

September 1962

SUBJECT: SOLID PROPELLANT GRAIN CORE
MANDRELS
INVESTIGATOR: A. E. Hornsey
CONTRACT: AF 33(657)-7178, Thiokol Chemical Corp.
ABSTRACT: A propellant cutting machine and three small scale segmented mandrels (one each of three different designs) have been used to form PBAA propellant burning surface configurations. The burning surface was formed through case openings that were smaller in diameter than the required perforation.

ASD TDR 62-967

October 1962

SUBJECT: LARGE CERAMIC RADOME MANUFACTURE BY
DRY-ISOSTATIC PRESSING TECHNIQUES
INVESTIGATOR: A. Pedigo
CONTRACT: AF 33(600)-37879, Coors Porcelain Co.
ABSTRACT: Large ceramic (99% + alumina) radomes for future hypersonic aerospace vehicles may possibly be fabricated using dry isostatic pressing techniques. Seven scale model (12-1/2" dia. x 39" long) radomes made by the dry isostatic pressing techniques were successfully completed. This report covers Phase I of the large ceramic radome program.

Coors Porcelain Company's standard 99% alumina body (NR AD-99) and 99-1/2% alumina body (NR B-1170) was encased in a preformed rubber bag around a shaped metal mandrel. After the final vacuum was drawn the entire assembly, radome blank and mandrel was placed in a hydraulic pressure vessel and pressed to 6000 psi.

During the pressing operation the inside geometry has been accurately shaped by compaction of the powder against the arbor. But the external geometry is rough and must be shaped by turning on a vertical lathe.

The shaped blank was removed from the mandrel and fired in a bell-type kiln using a special shrinkage setter plate. The radome blanks were fired in both upright and inverted positions.

A large number of test specimens were also fabricated for physical and electrical testing to determine the properties of high alumina bodies. The results of electrical and mechanical tests performed by Melpar Inc. are shown in "Appendix I" and results of attachment and testing performed by the Boeing Co. are shown in "Appendix III". The results of the thermal shock testing of two (2) thin wall radomes are shown in "Appendix II".

ELECTRONICS

ASD TDR 62-7-444, Vol III

April 1962

SUBJECT: WAFFER RIBBON COIL MACHINE
INVESTIGATOR: D. McCarthy
CONTRACT: AF 33(600)-30839, American Machine &
Foundry Co.

ABSTRACT: This report describes the prototype Ribbon Coil Machine which was developed and fabricated during the final phase of this program. Volume I described all the development work associated with the wafer ribbon coil and its adaptation to transformer use. Volume II described the experimental equipment developed for making the wafer coil and assembling the transformer.

The prototype ribbon coil machine will automatically produce multiple section coils at a rate of up to 350 coils per hour, this rate depending on ribbon size, number of coil sections and number of turns. Either aluminum or copper ribbon may be wound with kraft tissue interwound as interturn insulation. The number of coil sections is variable from one to six, ribbon width from 1/8 to 1/2 inch, ribbon thickness from 1 to 10 mils, coil length from 1/8 to 1-1/2 inches and coil diameter from 5/8 to 4 inches. The machine is readily readjusted for a change-over to produce different size coils.

The most significant technical feature of the machine is its ability to produce multiple section wafer coils from a single unbroken ribbon conductor. This permits the most efficient use of insulation in the coil, allows the use of stronger conductors, and increases coil reliability because of increased insulation and lack of interwafer electrical connections.

ASD TR 62-7-625A

May 1962

SUBJECT: DEVELOPMENT AND PRODUCTION REFINEMENT OF MIL-C-26518(USAF) CONNECTORS, ELECTRICAL, MINIATURE RACK & PANEL ENVIRONMENTAL RESISTING, 200°C AMBIENT TEMPERATURE
INVESTIGATOR: C. H. Stuart

ASD TR 62-7-625A (Continued)

CONTRACT: AF 33(600)-35971, Amphenol-Borg Electronics Corp.

ABSTRACT: As a result of this contract, a new family of connectors has been developed, and improved production refinement techniques established for their manufacture. A significant upgrading in connector reliability, operational performance, and serviceability is achieved. This manual presents physical, electrical, and mechanical performance data as well as servicing procedures.

In order to acquaint the user with the connector, its construction and design features are described. Detailed procedures for preparing the wire, crimping wire to contacts, inserting contacts into connectors, removing contacts from connectors, and mating of connectors are also covered in this manual.

The appendix contains descriptions of additional available connector sizes, shell styles, and insert arrangements covered by Specification MIL-C-26518(USAF).

ASD TR 62-7-737

April 1962

SUBJECT: CERMET ELEMENT RESISTOR REFINEMENT PROGRAM

INVESTIGATOR: J. DiGirolamo, R. J. Masten

CONTRACT: AF 33(600)-40125, CTS Corporation

ABSTRACT: A line of cermet element variable resistors meeting the size requirements of MIL-R-94B Styles RV4, RV5, and RV6, but with higher performance specifications including full load rating at 125°C and no load rating at 275°C, was designed and produced.

A proprietary cermet resistance element, originally developed by CTS for its high stability, 125°C, 1/2 watt, 1/2" diameter variable resistor, and subsequently for its complete line of cermet fixed and variable resistors was used for all three styles.

A carbon-graphite contact button in conjunction with an elgiloy contact spring was used. Terminations from silver end terminals to the resistance element were made by means of a

high temperature conductive epoxy. The collector ring or center terminal was made from Premaloy. Low temperature melting glass was used as an inorganic adhesive to secure the terminals in the ceramic base. A glass-filled teflon sleeve between the shaft and bushing was used as a bearing and also to control the torque. This was supplemented with a high temperature lubricant at the metal-to-metal bearing points. The housing or cover was made from impact extruded aluminum. The substrate is steatite ceramic. Audio tapers were satisfactorily produced utilizing two different resistance materials for the cermet element.

Preproduction environmental tests were run with generally satisfactory results. Corrective measures on areas requiring improvement were made and a smaller sample lot was re-tested. After approval of these results, a pilot run of 2200 pieces encompassing various resistance values of all three styles was made, and acceptance inspection tests were conducted.

ASD TR 62-7-838

September 1962

SUBJECT: REPRODUCIBLE THERMISTOR REFINEMENT PROGRAM
INVESTIGATOR: M. C. Vanik, W. T. Barrett, J. E. Herrera et al.
CONTRACT: AF 33(657)-7104, W. R. Grace & Co.
ABSTRACT: Contact improvement has yielded stable thermistors of gold-doped P-type silicon. Similar techniques are being used on gold-doped N-type silicon. The unbalanced pilot line has been operated according to a twenty-four step manufacturing process.

ASD TR 62-7-850

July 1962

SUBJECT: INTERDIGITATED SILICON TRANSISTOR PROGRAM
INVESTIGATOR: B. Rappaport, F. J. Steinebrey
CONTRACT: AF 33(600)-43029, Pacific Semiconductors, Inc.
ABSTRACT: The 10, 50, and 100 ampere device designs have been completed. Some 10 and 20 ampere planar transistors have been

ASD TR 62-7-850 (Continued)

fabricated, and indications are that by the elimination of all thermal oxidations of the collector surface, the collector-base reverse leakage can be reduced. An electrochemical polishing technique has been tried and shows promise. A decision was made to employ a horizontal RF heated reactor for epitaxial collector deposition, and construction is under way. Some progress has been made in the development of a vapor technique for predepositing the base dopant on the wafer. Three package sizes will be required to house all five types of transistors, each utilizing ceramic insulators. Packages will be sealed by cold welding.

ASD TDR 62-7-988

February 1963

SUBJECT: THE GROWTH OF LARGE ZINC OXIDE CRYSTALS
INVESTIGATOR: R. W. Lambdin
CONTRACT: AF 33(657)-8795, Airtron
ABSTRACT: The installation of the Pilot Line equipment, molten salt furnaces, hydrothermal furnaces, and material handling equipment has been completed. Temperature gradients are monitored on all runs. Hydrothermal equipment is being checked and P-T data taken.

ASD TDR 63-308

April 1963

SUBJECT: DEVELOPMENT OF METALLIZED TEFLON CAPACITORS
INVESTIGATOR: D. H. Smith, R. J. Simpson, E. D. A. Geoghegan
CONTRACT: AF 33(600)-38431, Dearborn Electronic Labs.
ABSTRACT: Production of metal encased tubular hermetically sealed metallized teflon capacitors reduces the volume per μf up to one-fourth that of conventional teflon dielectric foil capacitors. The subminiature metallized teflon capacitors are designed to operate over a temperature range of $-65^{\circ}C$ to $+200^{\circ}C$.

SUBJECT: REPRODUCIBLE THERMISTOR REFINEMENT PROGRAM
INVESTIGATOR: M. C. Vanik
CONTRACT: AF 33(657)-7104, W. R. Grace & Co.
ABSTRACT: Gold-doped monocrystalline silicon exhibits temperature-resistivity behavior suitable for making highly reproducible, predictable and sensitive thermistors. Two types of thermistors were developed with reproducibilities of $\pm 2\%$ & operable ranges incl. -85 to $+200^{\circ}\text{C}$. Manufacturing methods were developed and demonstrated on an unbalanced pilot line.

SUBJECT: MANUFACTURING METHODS FOR INDIUM ANTIMONIDE INFRARED DETECTORS
INVESTIGATOR: C. H. Sutcliffe
CONTRACT: AF 33(600)-40090, Philco Corporation
ABSTRACT: Methods of manufacturing photovoltaic indium antimonide infrared detectors with detectivities within a factor of two of the theoretical limit have been developed. Detectors fabricated by these methods have demonstrated good electrical characteristics, thermal stability, and mechanical ruggedness.

Studies of junction formation techniques proved diffused junctions superior to alloyed junctions. Four diffusants were tried and cadmium was found to be best, considering the combination of conversion efficiency, responsivity, uniformity, and stability. The depth of diffused junctions was found to be too great for maximum sensitivity, consequently a controlled surface etch was developed to obtain optimum junction depth.

Of the sensitive area delineation techniques tried, mechanical masking proved best for this program. For higher production rates or more complex shapes, evaporation masking is recommended.

A glass-metal-sapphire dewar was designed for the detector package. It met the vibration requirements of MIL-E-5400 and the temperature requirements of MIL-E-202. Glass encapsulated platinum leads integral with the package were used to eliminate lead generated microphonics.

FABRICATION

ASD TDR 62-7-937 (III)

September 1962

SUBJECT: MANUFACTURING METHODS AND DESIGN
PROCEDURES FOR BRAZED REFRACTORY
METAL HONEYCOMB PANELS

INVESTIGATOR: J. W. McCown

CONTRACT: AF 33(657)-7276, Martin Marietta Corp.

ABSTRACT: Brazed honeycomb sandwich panels using molybdenum and columbium core and facings provide lightweight structural coverings for high temperature applications on aerospace vehicles. Panel configurations selected for fabrication simulate a hot structural and a radiant heat shield application. Manufacturing processes and procedures developed in the program will be used to fabricate test panels. Elevated temperature tests will determine thermal and structural capabilities of the test panels.

ASD TDR 7-938

October 1962

SUBJECT: DEVELOPMENT OF LIGHT WEIGHT HIGH-
TEMPERATURE STRUCTURES PHASE I.
DESIGN CRITERIA

INVESTIGATOR: E. H. Nickell, A. B. Burns

CONTRACT: AF 33(657)-9145, Lockheed Missiles & Space Co.

ABSTRACT: The purpose of this phase of the investigation is to establish a design criterion for high-temperature foil-gage sandwich structures and to design such a structure for fabrication.

ASD TDR 62-999

December 1962

SUBJECT: MOLECULAR FORMING TECHNIQUES

INVESTIGATOR: J. Glasser, W. E. Few

CONTRACT: AF 33(616)-6288, Chemical & Metallurgical Research, Inc.

ABSTRACT: This molecular forming study suggests that certain facets of R&D should be accelerated, since the technique does offer some advantages over conventional metal forming techniques.

ASD TDR 62-999 (Continued)

As used in this report, "molecular forming" is defined as the production of fabricated or semi-fabricated shapes directly from molecular species or compounds, wherein chemical or electrochemical reactions take place to form the shape and to define properties. The initial compounds may be in either condensed or gaseous states, and the subsequent reactions may be either liquid phase or gas phase reactions on a solid state surface. By molecular forming techniques, shapes can be finalized with little or no additional fabrication or machining.

ASD TDR 62-1035

October 1962

SUBJECT: DIRECT CONTACT EXPLOSIVES METAL-
WORKING
INVESTIGATOR: J. Savitt
CONTRACT: AF 33(657)-9631, Explosiform, Inc.
ABSTRACT: The technical foundation of employing high explosives in direct contact with metal workpieces for non-destructive metal forming was investigated. Quantitative effects of explosives weight and density upon direct contact plate and tube bulging were determined for NITROGUANIDINE explosives charges and mild steel workpieces.

ASD TDR 63-125

January 1963

SUBJECT: HIGH TEMPERATURE MACHINING METHODS
INVESTIGATOR: I. A. Dickter, C. L. Mehl, R. F. Henke
CONTRACT: AF 33(600)-40066, Cincinnati Milling Machine Co.
ABSTRACT: Hot machining can reduce the cost of manufacturing parts from many high strength steels, precipitation hardening stainless steels, and nickel and cobalt based alloys. Selection of parts with suitable materials, and proper planning of set-up and sequence of operations, materially influences the final cost. Manufacturing costs of aerospace parts when hot machined from heat treated AISI 4340 and Ph 15-7 Mo steels were reduced up to 36% when compared to conventional machining.

ASD TDR 63-125 (Continued)

Radio-frequency induction and resistance heating techniques were developed and proved practical for heating magnetic materials, while the inert tungsten arc torch was more satisfactory for non-magnetic materials.

Most high strength metals will show a significant increase in tool life and permissible metal removal rates at elevated temperatures. Hot machining of 6 Al 4 V, 13 V 11 Cr 3 Al titanium alloys and refractory metals and alloys, achieved only limited success.

While some metallurgical changes were observed on the hot machined nickel and cobalt based alloys, these changes were not severe or extensive.

No excessive safety or health hazards are involved.

ASD TDR 63-171

March 1963

SUBJECT: DEVELOPMENT OF THE PROCESS OF FLAME MACHINING WITH CHLORINE

INVESTIGATOR: H. E. Pattee, R. E. Monroe, D. C. Martin

CONTRACT: AF 33(657)-8615, Battelle Memorial Institute

ABSTRACT: It has been possible to simulate metal-turning operations using a stream of chlorine gas directed on a metal surface which has been heated by plasma-arc procedures. Copper was used to prove the capability of this process because chlorine reacts with copper at low temperatures and the reaction rate is rapid; by adjusting the flow of chlorine and the angle at which the stream of gas struck the rotating workpiece, it was possible to remove sizeable amounts of metal and to volatilize the metal chlorides which were formed. Rotary machining was enhanced because the same area passed repeatedly through the chlorine stream and metal was gradually removed. It has not been possible to make linear cuts in sheet stock because the reaction rate is slow compared to the desired cutting speed. This process should be extended to include the machining of the refractory metals, since their reaction temperatures and rates with chlorine are comparable to those of copper.

Studies indicated that it is possible to cut sheet metal with the constricted-arc plasma torch using chlorine as a substitute for the conventional plasma gases; however, the quality of cutting is poor, and the process does not appear to be superior to more common flame-cutting methods in terms of accuracy or metal removal rates. Linear cutting occurred as the result of melting the base metal, rather than from metal-chlorine reactions. While such reactions could be initiated and maintained, their rate was too slow to affect the cutting mechanism significantly. Linear cuts of the type associated with plasma-arc cutting were obtained only with the cutting mode of operation when chlorine was used; however, comparable cuts could be made with nitrogen under almost the same operating conditions. The corrosive effects of the chlorine caused extensive equipment difficulties; the useful life of the torch electrodes and nozzle inserts was measured in minutes. Further research with this type of plasma-arc equipment is not recommended.

ASD TR 63-7-926

February 1963

SUBJECT: DEVELOPMENT OF NON-VACUUM ELECTRON BEAM WELDING
INVESTIGATOR: L. H. Leonard, J. R. Morley, L. Dunn
CONTRACT: AF 33(657)-7237, Alloyd Electronics Corp.
ABSTRACT: Electron beam welding of aero-space metal in a non-vacuum environment is feasible if the electron beam has sufficient initial power density that after being degraded by scattering, the residual power density is adequate to accomplish joining. This report discusses a system designed to accomplish such welding.

The minimum power-density required for the joining is approximately 0.5 Megawatt per square centimeter. To obtain these power densities in the non-vacuum environment, the beam, brought out from a region of high vacuum through a series of three differentially pumped apertures, must have an initial power density slightly in excess of 0.5 Mw/cm^2 when using helium gas as the controlled environment.

A system has been designed and constructed in accordance with these requirements. Some of its initial operating characteristics are discussed.

BASIC INDUSTRY

ASD TR 61-7-876

May 1962

SUBJECT: A STUDY FOR FORGING VARIABLES
INVESTIGATOR: H. J. Henning, et al
CONTRACT: AF 33(600)-42963, Battelle Memorial Institute
ABSTRACT: The major objective of this program is the preparation of an engineering report relating fundamental metal properties and deformation characteristics to the forging process. This report presents (1) some of the sections it will contain, (2) the results of supplementary experimental forging studies conducted on the three program alloys: AISI 4340, A-286, and Mo-0.5Ti-0.08Zr.

Forging data are presented for alloys of aluminum, magnesium, steel, titanium, and molybdenum. Forging design practices are described.

ASD TDR 62-334

March 1962

SUBJECT: TITANIUM COLD ROLLED STRIP AND SHEET DEVELOPMENT
INVESTIGATOR: B. L. Shakely
CONTRACT: AF 33(600)-37939, Crucible Steel Co.
ABSTRACT: Three Department of Defense Titanium alloys, Ti-16V-2.5Al, Ti-4Al-3Mo-IV, and Ti-6Al-4V, were investigated and processes developed with reference to producing wide, thin sheets for aircraft and missile applications. Laboratory and production-scale investigations of the variables affecting the rollability of these alloys revealed:

a. Production mill design requirements and strip rolling pass schedules can be predicted from laboratory measurements.

b. Modern rolling mills can cold roll wide, thin Ti-16V-2.5Al strip now at reductions of five to ten percent per pass. At light reductions they can roll to gages thinner than one five-hundredth of the roll diameter.

ASD TDR 62-334 (Continued)

c. Edge cracks and striations, not mill capacity prevent the cold rolling of Ti-4Al-3Mo-1V and Ti-6Al-4V to wide, thin strip.

ASD TR 62-390

March 1962

SUBJECT: BERYLLIUM CASTING
INVESTIGATOR: J. B. Denny, B. H. Hessler
CONTRACT: AF 33(600)-37902, The Beryllium Corporation
ABSTRACT: A manufacturing process has been developed for the production of sound three inch diameter vacuum cast beryllium billets suitable for fabrication. The casting technique relies on a thermal gradient within the mold (hot top, cold bottom) to control directional solidification of the metal and eliminate the center-line shrinkage and cracking encountered in conventionally cast billets. Ingots produced by this process were successfully converted on a laboratory scale into rolled sheet, extruded rod and tubing, and forged cup.

ASD TR 62-7-644

June 1962

SUBJECT: PROGRAM FOR THE DEVELOPMENT OF
EXTRUDED BERYLLIUM SHAPES
INVESTIGATOR: L. M. Christensen, R. R. Wells
CONTRACT: AF 33(600)-36931, Northrop Corp.
ABSTRACT: Successful completion of this extruding process development has demonstrated techniques by which aircraft structural shapes can be extruded from unalloyed beryllium. Pilot production of five 20 foot lengths of a structural channel 1.50 wide by 1.00 high with a nominal wall thickness of .060 inches proved acceptability and reproducibility of the developed process.

ASD TR 62-7-647

June 1962

SUBJECT: BERYLLIUM FORGING PROGRAM
INVESTIGATOR: A. F. Hayes, J. A. Yoblin
CONTRACT: AF 33(600)-36795, Ladish Co.

ASD TR 62-7-647 (Continued)

ABSTRACT: Unclad vacuum hot pressed beryllium was successfully forged in closed dies to provide an improved aerospace quality structural material for Air Force requirements. A positive method for keeping beryllium in compression is necessary for forging operations, which normally develop stresses of high magnitude. The expendable, hot steel support technique developed by Ladish Co. has been successfully used to drastically deform unclad beryllium in trial production runs. A mild amount of unrestrained deformation can be imposed on billets of relatively small size. Forged beryllium shows a significant increase in mechanical properties over those reported for hot pressed beryllium.

ASD TR 62-7-648(II)

July 1962

SUBJECT: CHEMICAL COMPOUNDS FOR METAL SHAPING
INVESTIGATOR: J. R. Cady, J. L. Jamieson
CONTRACT: AF 33(600)-43027, Douglas Aircraft Co.
ABSTRACT: Further development of gel systems has improved physical, chemical and mechanical properties. Milling metal plate stock is limited by the amount of chemical energy presently available in the gel. Stainless steel honeycomb was chemically contoured with good dimensional control. Porous solids carrying liquid etchants formed profile cuts but tolerance and surface finish need improvement. Comparative tests with liquid and non-liquid etchants determined probable limits of performance.

ASD TR 62-7-671

July 1962

SUBJECT: PROCESS DEVELOPMENT FOR THE RADIAL EXTRUSION OF TURBOJET DISCS
INVESTIGATOR: R. W. Krohn, E. C. Gilewicz, F. H. Lang
CONTRACT: AF 33(600)-37191, Curtiss-Wright Corp.
ABSTRACT: A manufacturing process has been developed for the precision forging by radial extrusion techniques for AMS 6432 steel compressor discs for current and future Air Force applications. Full-scale AMS 4928 titanium discs can be semi-precision forged using similar methods. Major advantages of this process are:

a. Both steel and titanium compressor discs can be produced with press tonnage only 50 to 60% of that needed for conventional forging methods.

b. Steel discs have metal volume overages of 29.6% as compared to 230% for conventionally forged discs.

This report details the entire development history of radial extrusion as applied to the J-65 Third Stage Compressor Disc. Included in the discussion are the results of the one-fifth scale laboratory work and one-half scale extrusion of AMS 6432 steel and AMS 4928 titanium discs. This work was conducted by the Syracuse University Research Institute. Also presented are the details and results of full scale extrusion of AMS 6432 steel and AMS 4928 titanium discs. Forming variables such as preform and die temperature, die lubricants, preform transfer, locating equipment and instrumentation are thoroughly reviewed. Heat treatment, inspection and machining of full scale discs are described in detail. Finally, the successful 150 hour engine test regime used to evaluate and qualify a radially extruded AMS 6432 disc is reported.

ASD TR 62-7-689

May 1962

SUBJECT: FLATTENING TITANIUM SHEET BY ROLLER LEVELING TECHNIQUES

INVESTIGATOR: K. W. Curry, W. H. Heil, G. M. Richmond
CONTRACT: AF 33(600)-37498, Titanium Metals Corp.
of America

ABSTRACT: Two Department of Defense sheet alloys of titanium, Ti-4Al-3Mo-1V, Ti-6Al-4V and an all beta alloy, Ti-13V-11Cr-3Al, were investigated in developing roller leveling techniques to produce wide flat sheets for Air Force requirements. Roller leveling improves flatness 25 to 50 percent but, except for the Ti-13V-11Cr-3Al alloy, less than 1% flatness was not realized. Data on wide sheets indicate that an average flatness of approximately 2 percent can be realized in normal production.

ASD 7-780

June 1962

SUBJECT: FLAT HIGH STRENGTH STEEL SHEET
APPLICABLE FOR USE IN THE AIRCRAFT
AND MISSILE INDUSTRY

INVESTIGATOR: T. H. McCunn, T. H. Sack

CONTRACT: AF 33(600)-40312, Allegheny Ludlum Steel Corp.

ABSTRACT: Description of work directed toward producing 36" and 48" wide sheet of air frame structural materials to 1/4 of AISI thickness tolerances. Coils of A-286, AM-350, Potomac A, Type 422 and Altemp R-41 were processed to finish thicknesses of 0.010", 0.030", 0.080" and 0.100". Coils were gaged at hot roll, intermediate and finish thicknesses. It has been demonstrated that all materials with possible exception of Altemp R-41 can be produced to 1/4 AISI thickness tolerance on available equipment. The most important factor affecting crown at finish gage is the percent crown at hot roll thickness.

ASD TR 62-7-828

March 1962

SUBJECT: DEVELOPMENT OF TECHNIQUES FOR
PRODUCING BERYLLIUM STRUCTURAL
SHAPES

INVESTIGATOR: E. E. Weismantel, K. C. Taber

CONTRACT: AF 33(600)-41959, Beryllium Corp.

ABSTRACT: A manufacturing process has been developed demonstrating the feasibility of forming structural configurations from high strength beryllium sheet and extruded pre-forms.

ASD TR 62-7-884

August 1962

SUBJECT: DEVELOPMENT OF SUBMICRON POWDER
MANUFACTURING METHOD

INVESTIGATOR: J. O. Gibson, J. D. Holmgren, R. Weidman

CONTRACT: AF 33(600)-42916, Vitro Laboratories

ABSTRACT: A manufacturing process, utilizing the high intensity arc vaporization techniques, has been developed for producing submicron size powders of refractory and non-refractory metals, oxides, and carbides. Powders having average particle sizes less than $1000\text{\AA}$ (0.1 micron) and purities exceeding 99% have been demonstrated as reproducible.

ASD TDR 7-911(I)

September 1962

SUBJECT: IMPROVED PRODUCTION OF POWDER
METALLURGY ITEMS

INVESTIGATOR: K. Farrell, N. M. Parikh

CONTRACT: AF 33(657)-9140, Armour Research Foundation

ABSTRACT: This report describes the initial studies concerned with the production of superalloy powders by inert gas atomization of liquid metal 713C. Argon gas was employed for atomization. Relations between atomization pressures, melt temperature and powder characteristics have been established. The properties of the powders produced to date are summarized and discussed. Important features of the equipment used in this program are also discussed. The powders produced are fine, have good flow characteristics and low interstitials.

ASD TR 62-7-924(I)

February 1962

SUBJECT: FIBER REINFORCEMENT OF METALLIC AND
NONMETALLIC COMPOSITES

INVESTIGATOR: R. H. Baskey

CONTRACT: AF 33(657)-7139, Clevite Corp.

ABSTRACT: This survey summarizes the progress in fiber metallurgy. Whiskers possess the maximum strength but are not available commercially. The survey confirms that of the three types of fibers available, i. e., glass, ceramic, or metal, only glass fibers are used extensively to reinforce material. Metal fibers have a limited commercial application as a reinforcing agent. In the instances where metal fibers are used to reinforce metals, the processes are still laboratory or pilot plant size.

ASD TR 7-936 (I)

October 1962

SUBJECT: DESIGN AND MANUFACTURING DEVELOPMENT
OF LIGHTWEIGHT HEAT EXCHANGERS

INVESTIGATOR: K. O. Parker, et al.

CONTRACT: AF 33(657)-9340, The Garrett Corp.

ABSTRACT: Cb-1Zr refractory alloy, Waspalloy, superalloy, and Ti-3 Al-2.5V alloy are the available materials in sheet and tube which best meet lightweight heat exchanger requirements for

ASD TR 7-936 (I) (Continued)

their respective temperature ranges. Columbium alloys are superior to tantalum alloys in strength to weight ratio, and to molybdenum alloys in ductile brittle transition temperature. A Ti-(cr-Ti)-Si multicycle pack cementation coating process is expected to be the most suitable for a nitrogen-air Cb-12r heat exchanger.

The purpose of this program is to develop new design concepts and new manufacturing techniques and processes to be used in the fabrication of extremely lightweight heat exchangers for advanced aerospace vehicles. The program covers fixed-boundary tubular construction and includes consideration of all contending materials, except stainless steel, through the temperature range of 40°R to 3000°R. The program is divided into three phases: Phase I consists of material evaluations and design analyses; Phase II consists of the fabrication and experimental testing of heat exchanger sections; and Phase III consists of the evaluation and reporting of results.

ASD TR 7-945 (I)

September 1962

SUBJECT: LINER FOR EXTRUSION BILLET CONTAINERS
INVESTIGATOR: S. A. Spachner
CONTRACT: AF 33(657)-8784, Armour Research Foundation
ABSTRACT: A container-sleeve-liner assembly was designed which will provide adequate support for ceramic, ceramic coated metal, or metal liners. The design minimizes mechanical property requirements of linear materials, and permits rapid removal of worn or damaged liners. A high-strength stem was designed and fabricated. Technical literature on high-strength materials has been reviewed, and high-strength materials producers have been contacted to locate sources and assess applicability of existing materials for refractory metal extrusion liner use.

ASD TDR 63-110

December 1962

SUBJECT: FINAL REPORT ON THE DEVELOPMENT OF
OPTIMUM MANUFACTURING METHODS FOR
COLUMBIUM ALLOY FORGINGS
INVESTIGATOR: P. F. Darby

WADC TR 53-373 Sup 10

262

ASD TDR 63-110 (Continued)

CONTRACT: AF 33(600)-39944, Crucible Steel Co. of America
ABSTRACT: A manufacturing process has been developed to produce closed die forgings of high strength columbium base alloys. Four candidate alloys, F48, D31, D41, and Cb74 were investigated and evaluated. A melting practice was determined to provide the best starting material, and primary breakdown and forgeability processing were used for the selection of Cb74 type alloy as having the best combination of forgeability and high hot strength characteristics for a useable structural forging.

ASD TDR 63-201

January 1963

SUBJECT: DEVELOPMENT OF METHODS TO PRODUCE
COLUMBIUM ALLOY Cb-74 (Renumbered
HAYNES ALLOY Cb-752) SHEET
INVESTIGATOR: J. G. Bewley
CONTRACT: AF 33(657)-7210, Haynes Stellite Co.
ABSTRACT: Methods for successfully producing columbium alloy Cb-752 sheet and bar have been developed. Approximately 125 pounds of sheet and 60 pounds of bar have been produced and evaluated for mechanical and physical properties.

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