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FLIGHT VEHICLE POWER and MATERIALS

Volume II (Part I)
Abstracts 2-208 through 2-316

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Prepared for COMMANDER SPACE SYSTEMS DIVISION
UNITED STATES AIR FORCE
Inglewood, California

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ABSTRACT

This Bulletin contains abstracts of the unclassified literature on the subject of Flight Vehicle Power (USAF Applied Research Area 750F). Abstracts of selected references in the field of materials are also included. All abstracts are prepared from the original reports and are limited to those available within Aerospace Corporation.

Approved by: K. B. Andrews
Supervisor
Literature Research Group

AEROSPACE CORPORATION
El Segundo, California
PREFACE

This Bulletin contains abstracts of the unclassified literature on the subject of Flight Vehicle Power (USAF Applied Research Area 750F). Abstracts of selected references in the field of materials are also included. All abstracts are prepared from the original reports and are limited to those available within Aerospace Corporation. Aerospace Corporation is not able to furnish copies of the abstracted reports to requestors outside the corporation. It is suggested that persons wishing to obtain such reports contact the originator. The preparation of this Bulletin began under Contract No. AF 04(647)-930 and is continuing under Contract No. AF 04(695)-169. The first 12 numbers of Report No. TDR-930(2701-01)TN-1 were published under the title "Applied Research Management Abstract Bulletin."

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I. FLIGHT VEHICLE POWER

SECTION A - GENERAL

2-208. FEASIBILITY INVESTIGATION OF CHEMICALLY SPRAYED THIN FILM PHOTOVOLTAIC CONVERTERS.

The objective of this work is to conduct the necessary applied research to obtain experimental data which would demonstrate the feasibility of a chemical spray process for the fabrication of thin film photovoltaic converters, using either CdS or CdSe as the semi-conducting layer. Quarterly Progress Report No. 1 covering the period of 1 February 1962 through 30 April 1962 dealt with: background; instrumentation; initial work in deposition of thin films of CdS, CdO, and SnOx; development of a new method for the deposition of copper films; and general considerations involved in fabricating a total thin film system such as one incorporating SnOx-CdS-Cu. This report covers in detail the evaluation of the semiconducting layer (CdS) and the initial work in fabricating a photovoltaic converter utilizing a barrier formed at the interface between a thin (0.5μm) film of CdS and a thin film (0.05μm) of Cu9-xS5 (digenite). (Author)

2-209. FEASIBILITY INVESTIGATION OF CHEMICALLY SPRAYED THIN FILM PHOTOVOLTAIC CONVERTERS.

The objective of this work is to conduct the necessary applied research to obtain experimental data which will demonstrate whether it is feasible to fabricate a thin film photovoltaic converter of either CdS or CdSe, using a chemical spray process as the means of depositing the semiconducting layer. Quarterly Progress Report No. 1 covering the period of 1 February 1962 through 30 April 1962 dealt with background, instrumentation, general considerations involved in fabricating a total thin-film system, etc. Quarterly Progress Report No. 2 covering the period of 1 May 1962 through 31 July 1962 dealt with the evaluation of a differently prepared and doped

(continued)
2-209. (Continued)

semiconducting layer (CdS), the initial work carried out in fabricating a
thin-film photovoltaic converter, etc. This report covers: (1) work done
in determining possible variations in the physical structure (crystallinity
and crystallite orientation) of the CdS and CdSe semiconducting layers due to
changes in deposition parameters; (2) effect of heat treatment on different
orientations; (3) changes in resistivity due to heat treatment and doping;
(4) improvement of the deposition of the barrier layer; (5) investigation of a
barrier layer using copper selenide; (6) search for a flexible substrate
(metal foil) compatible with the film deposition conditions; (7) investigation
of the possible correlation between crystallite orientation and crystallinity
to photovoltaic response; (8) spectral characteristics of CdSe, CdS, and
CdSe-CdS photovoltaic cells.

2-210. MERCURY-ARC OSCILLATOR. Ta-Kuan Chiang.
Massachusetts Inst. of Tech., Cambridge, Research Lab. of
Contract: AF 33(616)-7624, Proj. 3145, Task 61098. 69 refs.
A62-10850.

A new device, the mercury-arc oscillator, is proposed for conversion of
low-voltage, high-current dc to ac at the voltage level of conventional ac
systems. Operation depends on the interruption of a constricted mercury
column and subsequent arc formation when a low dc voltage (=10 volts) is
applied. The phenomena involved are discussed in relation to arc discharge
in mercury-arc rectifiers, circuit breakers, and EWP. Theoretical and
experimental studies show that the magnetic pinch effect initiates circuit
interruption and that high-field emission is responsible for arc maintenance.
An expression for the oscillation frequency is obtained and compared with
results obtained by using a 0.031-inch diameter capillary constriction over
the pressure range from 106 mm Hg to 1 atmosphere. Methods of frequency
control are discussed, and an experimental device, operating on a dc supply
of 4-8 volts at a current of 100-160 amps and with a frequency in the range
of 60-250 cps, is described. The elements affecting duty factor are con-
sidered, and typical device geometry and operating conditions for this factor
to be 50 per cent are given. There are brief discussions of frequency
stability and of parallel and push-pull operating characteristics. (Author)
2-211. PIEZOELECTRIC CONVERTER INVESTIGATION.

This report presents an extension of an effort to develop a piezoelectric energy converter with major emphasis on acoustic-to-electric transduction. Both thickness and flexural mode devices were chosen for evaluation. Three ceramic transducer materials were manufactured in a variety of shapes and sizes that would allow investigation of these operational modes. Theoretical response of a thickness mode receiver for plane waves is discussed, and power output ratios are found for three ceramic materials. Initial low field test data are presented for a thickness mode device and for three flexural mode devices. The flexural elements tested are edge-supported, bilaminar discs, each of a different ceramic material. The transducers are being designed with the capability of operating in a 173 db sound field. (Author)


The purpose of this study is to investigate the possibilities of using the Nernst effect in solids as the basis of a generator and, further, to attempt to find those materials which will give optimum performance. The main effort during the first quarter has been a theoretical analysis of the factors that enter the figure of merit so that an experimental program could be limited to those materials which show the most promise. The results and conclusions based on these results form the basis of this report. Calculation of efficiency and a preliminary study of the figure of merit are extended somewhat, and numerical calculation of the efficiency that could be obtained for various materials is calculated and discussed. The possibility of obtaining the optimum material parameters is explored. It is concluded that probably a large phonon thermal conductivity will limit the ultimate efficiency of a thermomagnetic generator so that the search for an optimum band structure may have to be subordinated to a search for low thermal conductivity material. Furthermore, the efficiency is rather slowly varying with band gap so that reasonable performance can be expected even if the optimum is not obtained. When the possibility of finding an optimum band structure is considered, however, prospects are promising, since a variety of materials, ranging from graphite through bismuth and its alloys to metals (continued)
such as tin and indium, may be considered. The manner in which phonon drag and coherent electron-phonon interaction might influence the figure of merit is briefly considered.


The objective of this study has been the development of new concepts of flight-vehicle power utilization to perform required functions in specific missions. The particular problem on which the effort has been focused is the simplification of the power conversion and transmission system interface between the energy source and the utilization equipment. A secondary objective has been the formulation and evaluation of the method itself; that is, the process by which novel utilization methods can be suggested and the framework within which they can be evaluated. An unconventional approach toward utilization of vehicle power is presented. Brief mission profiles are given for six manned and unmanned air and space craft to give the schedule of operation of systems which utilize power. Greater detail is presented on the power utilization of two of these, the unmanned reconnaissance satellite and the manned lunar-landing and return vehicle. A general discussion is presented on power sources intrinsic and extrinsic to vehicles, and a forecast is shown for chemical, solar, and nuclear sources. The application of the morphological method to the development of new concepts is discussed and examples of several "morphological boxes" are given in an appendix. Some of the concepts that developed during the course of the study are also given in appendices. In addition, the appendices contain general reviews of EWASERS, radio space communications, and electrical propulsion. It was concluded that a modified morphological approach offers a better framework for conceiving advanced concepts. Recommendations are made for further study and development of the method and new concepts.


This report presents the results of a study of three competitive flight vehicle power units: (1) a battery powered, solid state converter; (2) a turbine driven, solid propellant powered converter; and (3) a turbine driven,
solid propellant powered, gas pressurized converter. Each unit produces seven electrical power outputs (2 low voltage ac and 5 high voltage dc ranging from 150-3000 volts dc) for the electronic equipment of a reentry package. The study covers power requirements for from 100-2000 watts and 2-20 minutes duration. The units are analyzed in terms of the following parameters, in decreasing order of importance: reliability, size (volume), weight, environmental capabilities, availability, development cost, production cost, and growth potential. It is concluded that no one selection is best for all applications; therefore, data are presented to allow the selection of the best unit for a specific application. Sperry, in an acknowledgement, credits the data largely to the efforts of AiResearch Division of Garrett Corporation.
SECTION B - CHEMICAL SOURCES OF ENERGY


This report covers the first two periods of an alkaline battery applied research and failure analysis program. The purpose of this program is to establish a broad base of battery test data for use in the design of the electrical system of future space vehicles and to determine the actual failure mechanism of all new battery systems under varying environmental and cycle-life conditions so that improved space batteries can be developed. Another objective is to determine techniques and/or materials to prevent these failures while at the same time to increase the usable watt-hours-per-pound capability and cycle-life of the battery. To date, cycle-life tests have been conducted on one type of 12 ampere-hour, sealed, nickel-cadmium cell in four temperature environments and four depths of discharge, in groups of 10 cells and in batteries of 20 cells. Initial results in the program show that: (1) cell cycle-life with shallow discharges is considerably longer than cycle-life at deep discharges; and (2) cycle-life is reduced by high and low ambient temperatures. The program will include as future work an evaluation of silver-cadmium and silver-zinc type cells. (Author)


The cells evaluated were of the sealed, secondary, silver-zinc type manufactured by the Missile Battery Division of the Electric Storage Battery Company. These cells were off-the-shelf items intended for aerospace applications requiring a short cycle life and a relatively high rate discharge capability. The procedure for cycling is described. The silver-zinc cells tested do not appear to be suited for high cycle-life applications even when cycled at shallow depths of discharge. The apparent cell unbalance and early cycle-life failure may possibly be attributed to the fact that the cells were activated in the latter part of February and not cycled until the (continued)
following June. While the silver-zinc couple offers an attractive energy-to-weight ratio, a considerable amount of work must be accomplished toward improving cycle life before this battery system can be used for long-life aerospace applications.


This report contains an orbital fuel cell experiment design discussion, a discussion of fuel cell requirements, an orbital fuel cell package design concept, and a detailed discussion of fuel cell orbital package system control and instrumentation. Two preliminary models of the orbital package have been assembled and are being subjected to various tests. The tests include: (1) fuel cell capacity tests with and without cooler; and (2) shock, acceleration and vibration tests of a fuel cell operated before and after motion tests. Functionally, the two packages are identical except for the methods of applying the evaporative cooling. A theoretical preliminary heat loss study was conducted and is reported in detail. A detailed description of design and operation of the fuel cell voltage controller is presented. Development tests carried out on the preliminary models of the package are described and results presented. These tests included: evaporative cooling tests, humidifier tests, and operational tests of fuel cell package model No. 1. The evaporative cooling tests were divided into two parts: thermal actuated valve and flash evaporative cooler tests, and pressure relief valve tests. The package tests included package operation, flow restrictions, secondary gas pressure regulation, and solenoid valve tests. No overall conclusions could be drawn from the tests for this period.


An investigation was carried out to determine methods of improving the performance of a solid columbium diffusion diaphragm when used as the hydrogen electrode in a lithium-hydrogen fuel cell. Treatments were devised for purification of the process media and preparation of the electrode, and a (continued)
confirmatory run was conducted in an all-columbium fuel cell. In the present program three purification studies were carried out. To test the results of the various treatments, samples of columbium of known, low, oxygen content were exposed to the test media at a temperature of 1150°F for four hours. Subsequent oxygen analysis could then indicate the effectiveness of the treatments. Methods of cleaning the columbium were studied, and it was found that a simple soap and water wash, followed by an acetone rinse, was effective. Starting oxygen contents of the standard specimens were found to be approximately 0.020%. Various procedures involving vacuum-drying, vacuum-melting, ball milling, and "gettering" with columbium turnings were investigated. It was found that the oxygen content of test samples exposed to salt which had been vacuum-dried, but with no other treatment, increased from 0.012 to 0.61%, a factor of fifty. A purification study of process gases (argon and hydrogen) was then undertaken. Both hydrogen and argon were purified by passage over titanium sponge at a temperature of 1550°F. Test samples of columbium were then exposed to the gases. The oxygen content of the sample exposed to unpurified tank hydrogen was 0.41%, twenty times the original value. A brief study of lithium purification was undertaken. It was found that both purified and untreated lithium reduced the oxygen content of the test samples from 0.020% to 0.007%. A fuel cell was built of columbium, 1.8 inches in diameter by 13-1/2 inches long. A columbium foil cathode of 0.685 inch diameter by 0.005 inch thick was employed. It was necessary to develop and utilize special welding and brazing procedures in the assembly of the unit. Cell output was recorded on a sine wave, pulse-type battery tester of conventional design, where true IR-free characteristics of the electrodes could be determined. A maximum short-circuit current density of 3,500 amps per square foot was obtained. The optimum power density of 400 watts per square foot was obtained at 68% of open circuit voltage. Following the run, there was no apparent deterioration of the equipment.

FEASIBILITY STUDY OF PALLADIUM AS A HYDROGEN DIFFUSION ELECTRODE MATERIAL FOR FUEL CELLS.

Palladium films as hydrogen diffusion electrodes in fuel cells have been investigated and found to possess very interesting possibilities when quite thin (of the order of 100 Å) or at temperatures above 200°C. An evaluation of (continued)
Above 400° C, hydrogen transport through palladium films is found to follow a semi-logarithmic relationship, with an activation energy for diffusion of about 5.5 kcal/g-mole. At approximately 200° C a low temperature phase having the approximate composition Pd₂H forms with a heat of dissociation of about 8.5 kcal/g-mole. Extrapolation of a few low temperature points yields an energy of activation for diffusion of about 17.8 kcal/g-mole below 150° C. Thus, at temperatures below 150° C, the current density supportable by mass transport is too low to make palladium films suitable for gas diffusion electrodes in fuel cells. Additional experimental work is suggested to clarify the possibilities of using palladium as a fuel cell electrode under conditions other than those indicated above. (Author)

An essential feature of a redox fuel cell employing a cheap hydrocarbon fuel is that the fuel must reduce the metallic ion (produced from the cell) at a reasonable rate. A large number of tests on the titanyl-titanous system and the stannic-stannous system, at temperatures up to 200° C, indicate that formic acid, formaldehyde, and methanol will not reduce the higher valent ion to the lower at a feasible rate. Consideration of thermodynamic equilibrium, solubility, and mass transport predict that regeneration by hydrocarbons will be even less feasible. This report is divided into two main sections; a theoretical discussion, and a report of experimental procedures. The theoretical discussion is concerned with the general aspects of the titanous-titanyl couple and the stannous-stannic couple. Experimental investigations were carried out on the following reaction cells: titanium couples in HCl - formic acid at low temperature; titanium in HCl - formaldehyde at low temperature; platinum coil - formaldehyde at higher temperature experiments; titanium couple at higher temperature; hydrolysis rate of titanium solutions; titanium couple - sulphuric acid; titanium couple - oxalic acid or glyoxal; titanium couple - zinc; stannic-stannous couple; and methanol as a fuel. Experimental procedures and results are presented for each of these systems. Although the investigation described in this report was performed using relatively crude and simple techniques, the work indicated fairly conclusively that the homogenous regeneration of the titanium or tin couples by means of hydrocarbons is not feasible. It also demonstrated that the addition of a simple catalyst, consisting of platinum dispersed on high area active carbon, does not give a satisfactory system. It seems unlikely that the use of better catalysts would improve this part of the system to a feasible point. In addition, the discouraging results on the oxidant regenerator reported by Walker, et al (Special Report No. 1, Contract No. DAI-49-186-502-ORD(P)-860, College of Engineering, University of Florida, March 1960; ibid., (continued)
Summary Report No. 3, April 1962) would indicate that this type of redox fuel cell system is not feasible. It is concluded, therefore, that further work along the lines reported here is not to be recommended.

This project was devoted to an investigation of metallic oxygen electrodes for low temperature fuel cell systems. The electrodes were examined both as physical entities and as components of electrochemical half-cells when used with oxygen and suitable electrolytes. Emphasis was placed upon the identification and characterization of significant physical and chemical parameters. Ultimately the study was to investigate surface structure, electrical conductivity, and reactions in an oxygen half-cell with high activity metal electrodes. In the study of half-cells, the applicability of carbonate-bicarbonate electrolytes was considered and compared with the results obtained with both alkaline and acidic electrolytes commonly employed in fuel cell research studies. To initiate this study, smooth non-porous electrodes of the metals (nickel and silver) that are contained in the Justi DSK electrodes were investigated. Since these metals are not stable in acid media, this work was done in basic solution. To extend the study to acid media, it was necessary to employ platinum group metals and their alloys because of their resistance to corrosion. During the year of the contract, considerable progress was made in relating the surface characteristics of electrodes to their electrochemical behavior. With two varieties of commercial high purity and one variety of ultra-high purity nickel cathodes, a correlation was found to exist between the concentration (small) of interstitial impurities and the polarization behavior. This result may correlate with the enhanced behavior of a nickel base with Raney silver, such as is used in a Justi-type electrode. Astropower developed very satisfactory techniques for measuring surface areas of about 50 cm$^2$ by the BET adsorption method, using a thermistor pressure-sensing device. Uniform films of nickel oxide were prepared on nickel electrode surfaces, and the electrochemical nature of these films was shown to be highly superior to platinum surfaces. Definite evidence was obtained that hydrogen peroxide exists as an intermediate, and that the reduction of hydrogen peroxide is the rate-determining step in the reduction of O$_2$ to OH$^-$ on nickel electrodes in the 12 to 14+ pH range. It was also clearly indicated that a low concentration of H$_2$O$_2$ is responsible for the zero current potential of nickel in the 12 to 14+ pH range. In contrast, no evidence was obtained to show that the reduction of hydrogen peroxide was rate-determining in the reduction of O$_2$ to H$_2$O on platinum in the 0 to 4 pH range.
The objectives of the work were to study fundamentals of fuel cell anode processes, particularly with the use of carbonaceous fuel gases, and to select suitable electrode structures to carry out such studies. The particular fuel cells investigated were low temperature, low pressure cells with aqueous alkaline electrolytes. A summary of experimental methods is presented. The hydrogen anode in alkaline electrolytes was investigated in more detail than electrodes employing other fuel gases. The electrochemical potential of electrodes employing 1b metal catalysts was not observed to be theoretical, while those electrodes employing group VIII metals were observed to produce theoretical or near theoretical potentials. The metals of the iron triad: iron, nickel, and cobalt are more subject to electrochemical oxidation than the noble metals of the platinum group in group VIII. Considering this fact, and the results of the investigations on other metals, the platinum group metals appear to be the best catalysts for a hydrogen anode. Two electrolytes, aqueous solutions of sodium hydroxide and potassium carbonate, were studied with the hydrogen electrode under open circuit conditions. The effect of various cell parameters on the potential-current characteristics of the hydrogen electrode was investigated. The results of these investigations are presented. The catalytic activity of the group VIII and 1b metals at electrodes employing carbon monoxide, ethylene, and acetylene was observed. As in the case of hydrogen, the group VIII noble metals appear to be the most advantageous catalysts for these electrodes.

The objective of the work performed under this contract is to demonstrate the technical feasibility of the solid electrolyte fuel cell for aerospace applications. The development of a series connected, fuel cell test model having long life, low volume, high power-to-weight ratio, and reproducible performance is sought in order that realistic evaluations of its characteristics can be made. In the development of solid electrolyte materials it was found that (ZrO₂)₀.₈₅(Y₂O₃)₀.₁₅ has less than one-fifth the resistivity of (ZrO₂)₀.₉(Y₂O₃)₀.₁. Discs fabricated from this material showed good performance in life tests. Electrode performance is being studied by: measurements of electrode resistivity; performance of flat plate fuel cells; and performance of instrumented tube cells having potential probes placed
to enable separation of cell resistance components. These tests have indicated the necessity of controlling the electrode application process. A description of the electrode application process is presented. Sheet resistivity measurements of various electrode surfaces were made using two methods; each employed four graphite probes and measured the potential differences between them. A detailed description of each method, including circuit diagrams is presented along with experimental data for some of the electrodes being tested. The performance of the solid electrolyte fuel cell is critically dependent upon the effectiveness of certain structural seals. Three types of seal were investigated during the report period. These included platinum-platinum pressure bonding, ceramic-to-metal seal, and ceramic-glass-platinum seals. Various tests, including photomicrographs, gas tightness, visual inspection, etc., were applied to seals, and the results of these tests are presented. A four-element, stacked tube, fuel cell system was assembled and tested. This assembly was used to investigate the electrical properties of such a fuel cell system and to determine the adequacy of a seal obtained with the given overlap and joining techniques. After assembly, the device was found to leak gases through the joints. The test was continued in order to determine: the electrical resistance of the cell and of the joints, the magnitude of the leak under operating conditions, and the effect of the leak on cell performance. Methods of testing and results obtained are described. In an effort to establish the effect of electrode porosity on cell performance and to produce an electrode which adheres more tightly to the electrolyte, platinum electrodes were applied to \((\text{ZrO}_2)_{0.85}(\text{CaO})_{0.15}\) discs by evaporation. Experiments were conducted on two disc cells having evaporated metal electrodes. Resistivity/thickness values for each electrode were determined, along with the over-all resistance of the cell. Load curves are presented. In each case it was found that the total cell resistance was much larger than could be accounted for from the resistivities of electrodes and electrolyte. In order to study electrode and cell performance under carefully controlled conditions of \(H_2\) fuel flow and temperature, three single-stage tubular cells were fabricated and tested. Overall resistivity and load curves were determined. A detailed description of results is presented. A series of small systems studies have been carried out to estimate the volume, weight, and ruggedness of solid electrolyte fuel cell systems based on the stacked tube segment concept.

2-224. OXYGEN ADSORPTION BY THE SILVER ELECTRODE.

A study was made of the coulombic efficiency of the silver electrode in KOH solutions. The coulomb input during anodization is greater than the coulomb output during cathodization plus the coulomb equivalent of the oxygen evolved.
This is interpreted as due to adsorption of oxygen by the electrode during the process in which AgO is formed. Both constant current and constant voltage conditions were used. (Author)

STATE OF CHARGE INDICATORS FOR ALKALINE BATTERIES:

Based on work done so far, there are several conclusions which can be drawn about phase shift. There is a definite relationship between residual capacity of a nickel-cadmium cell and the phase shift it produces. The shift increases, algebraically, with increasing residual capacity. The phase shift is a monotonic function of residual capacity, with temperature as a parameter which can be accounted for. Temperature affects the phase shift at a given capacity in a predictable, reproducible way so changing the ambient temperature presents no difficulties. The change in phase shift with residual capacity is large enough to provide a means of determining residual capacity. Having established these points, tests have been run, and are continuing, to evaluate the phase shift technique as a useful measuring tool. These tests are being carried out using various sizes and types of cells and batteries: a sealed 4 A-h cell in steel case, a 6 cell battery of VO-4 cells, a 6 cell battery of sealed 9 A-h cells, and a vented 35 A-h cell in nylon case. The tests were carried out under a variety of conditions; room temperature, 19°C and 30°C.
SECTION C - MAGNETOHYDRODYNAMIC SYSTEMS

No entries are made in this issue.
SECTION D - MECHANICAL DEVICES


The TAPCO Division of Thompson Ramo Wooldridge is conducting an applied research program on a hermetically-sealed coupling for space power transmission. The objective of the program is to develop and demonstrate a one-to-one speed ratio, hermetically-sealed coupling for a 15 KW, 24,000 rpm unit and to provide a technical evaluation of the problems encountered in scaling the unit from 15 KW to 3 KW and 3000 KW. The design requirements provide for power transmission through a continuous boundary with a device having a 90% overall efficiency and 10,000 hours of maintenance-free design life. Operating conditions will be 20 to 40 psi pressure differential, 1000°F potassium vapor on one side of the boundary, and 10⁻⁵ mm Hg vacuum on the opposite side. The following has been accomplished during the past three months of the program: (1) The magnetic coupling parametric analysis has been completed, and the results indicate that a radial gap coupling with stationary electromagnetic sleeve is the most promising magnetic coupling concept. (2) The detailed design of the selected electromagnetic coupling has been started and is 50% complete. (3) Design of the high temperature potassium vapor and vacuum environment test rig has been started and is 50% complete. (4) The deformable membrane coupling detailed design is 90% complete. (5) A test rig for the high frequency evaluation of various bellows configurations has been fabricated and is ready for the start of testing.


An operation of the system over the entire range of loading, i.e., from zero to full load, overload, and short-circuited output terminals, was attained by two different methods. The system performed without damaging results under these conditions and would recycle after any faulty condition was removed. A choice between one technique that introduces passive components into the power circuit and another that requires active components (transistors) in the control system arises. Another problem, that of unsatisfactory regulation, was solved after extensive investigation by replacing the physically speed-limited ferromagnetic integrators by new type electronic
circuits. The latter permitted a satisfactory open-loop regulation of the system over its entire range of input voltage regulation and loading. A detailed description of the study for the tap switch modulation technique as carried out under the provisions of this contract is included. (Author)
SECTION E - NUCLEAR SOURCES OF ENERGY

2-228. BURNUP ENHANCEMENT FOR REENTERING NAP SYSTEMS.
Sixth Monthly Progress Letter, through 31 Aug. 1962. Battelle
Memorial Institute, Columbus, 5p. Contract:
AF 29(601)-4939, Proj. 1831, Task 183101. No accession no.

This letter summarizes progress during August 1962 in the study to determine
the feasibility of changing the properties of hydrided uranium-zirconium alloy
so as to enhance the atmospheric burnup of nuclear fuel rods during reentry.
Prior work is reviewed. During August, pressure-bonding of powder disper-
sions was used as a technique for introducing large percentages of O2N and C
into the fuel alloy. Adjustments and modifications to rocket test equipment
were completed. Test runs with ZrH alloy (no uranium) were made and the
results were being analyzed.

2-229. BURNUP ENHANCEMENT FOR REENTERING NAP SYSTEMS:
Battelle Memorial Institute, Columbus, 3p. Contract:
AF 29(601)-4939, Proj. 1831, Task 183101. No accession no.

This letter summarizes progress during September and October, 1962 in the
study to determine the feasibility of changing the properties of hydrided
uranium-zirconium alloy so as to enhance atmospheric burnup of nuclear
fuel rods during reentry. Prior work is reviewed. During the fifth test run
of the rocket test equipment with ZrH alloy, explosions damaged the system.
The critical factor was the need for conducting ablation tests at reduced
pressure. No repairs were made or planned. The study effort appears to
be terminated.

2-230. FLIGHT TEST CRITERIA STUDY FOR REENTERING NAP
North American Aviation, Inc., Downey, Calif., Space and
Information Systems Div. 6p. Contract: AF 29(601)-5104,
Proj. 1831, Task 79512. No accession no.

Progress during August-October, 1962 in the Nuclear Auxiliary Power
System Reentry Phenomena study is described briefly. The only technical
work done was calculation of additional trajectories and aerodynamic heating
data. This information will supplement data used in the Interim Report,
TDR 62-83, covering work done through August, 1962.
This report summarizes the progress made during the month of October in the determination of high temperature properties of unhydrided and hydrided zirconium-uranium alloy. Measurement of the heat of combustion of hydrided zirconium-uranium was repeated to determine reproducibility, and the value previously reported was found to be in error. Oxidation experiments on hydrided zirconium-uranium alloy and unhydrided zirconium-uranium alloy at 892°F and 1472°F were completed. Effect of the concentration of air on the rate of oxidation for the hydrided alloy at constant temperature was studied.

ERRATUM - one page and two graphs for insertion into the cited APL are presented.

This letter briefly summarizes work done during October, 1962 on the study of Nuclear Auxiliary Power Systems Reentry Phenomena. Equipment modification is discussed. No data.

The general objective of this effort is to determine the mechanisms and the extent of removal of material from models representing nuclear fuel rods.
under simulated reentry conditions. No evidence found to date supports the concept of removal by liquid-layer formation and droplet removal by aerodynamic forces. This indicates that the original study title, liquid layer instability and droplet formation is a misnomer. Hydrided zirconium-uranium alloy, comparable to the nuclear fuel material used in the SNAP-2 and SNAP-10A reactors, was used. Model fuel-rod sections were tested under conditions simulating reentry from an altitude of 400,000 ft at a velocity of 25,680 ft per sec, and a flight-path angle of 0°. This is close to the assumed situation for a SNAP reactor in a vehicle which reenters in a trajectory decaying naturally from an operational orbit of up to 1-year duration. The results of trajectory computations, reentry heating and pressure history calculations, calculation of the altitude at which fuel rod exposure is to be expected, the value of the ballistic coefficient $W/C_{D,A}$ (300 lb/sq ft max) and experimental results to date in arc plasma generator tests are summarized. The particles of nuclear material recovered corresponded roughly to the appearance expected for brittle fracture of metal, into spherical particles covered with gray oxide. Preliminary measurements of particle sizes indicated that about 5% were less than 100 microns in diameter. A preliminary discussion of the results is presented. The results indicate that fuel rod ablation occurs intermittently in periods of intense surface activity, during which particles literally explode from the surface (resembling a Fourth of July sparkler). It is postulated that buildup of internal hydrogen pressure causes this effect. The assumptions and simplifications invoked are severe, but it is shown that the mechanism is plausible. The arc-plasma generator tests are expected to be completed during the next period.


This letter briefly summarizes work done during August, 1962 on the Particle Disintegration Study for Re-entering Nuclear Systems. Improvements to the experimental equipment are described and a short note on runs of U-Zr alloy tests at various oxygen partial pressure is included. Results of the runs are to be included in the next quarterly report.
This letter briefly describes work done during October, 1962 on the Particle Disintegration Study for Re-entering Nuclear Power Systems. Thirty test runs were made using U-Zr alloy particles. Summary data are tabulated. Equipment problems influencing the results are discussed. The runs are judged significant, but interpretation must take into account the partial pressure of reactive gas in the plenum chamber.

Experimental investigations of particle disintegration of U-Zr and U-ZrH alloys are reported for the period July-Sept. 1962. Improvements made in the plasma apparatus used are summarized, preliminary experiments with Mo particles in the 100 to 200 micron size range are reported briefly, and the results of the powder preparation and test runs with nuclear fuel materials are given. Particles of hydrided U-Zr alloy tested in an argon atmosphere were reduced from 125 to 185 micron initial size to 0.02 to 40 micron final size; they were frequently spherical, indicating a molten stage. When tested in an atmosphere of argon plus oxygen, transformation in particles to transparent, rounded forms without changes in size was observed. Results with unhydrided U-Zr alloy were less clearcut but apparently similar to the above. Photomicrographs are presented, and the photographic techniques are discussed. The results cannot be considered as conclusive. Experimental work is to continue, and efforts to obtain more detailed information and correlation of all the experimental data with theoretical work done earlier in the project will be undertaken during the next period. (Note difference in tentative results as compared with the contemporaneous Vidya report on this project.)

During this period, work was pursued in scaling, thermodynamic analysis of fuel elements, and evaluation of heat transfer to a full rod. The scaling (continued)
parameters were determined for tumbling bodies without ablation, thermal stress to solid bodies and shell structures due to aerodynamic heating, and nose cap dissociation. Some radiation effects and the exact dynamic pressure variations of realistic reentry trajectories cannot be scaled. Thermo-dynamic analysis work consisted of programming reentry trajectory information for use in a thermodynamic program code for the IBM 7090, entitled Variable Boundary Transient Heat Conduction Program. Samples of the results are given but not complete output runs. The heat transfer evaluation work comprised examination of possible methods of evaluating the total heat flux imparted to a fuel rod. The results presented are only tentative.


Technical progress is reported on an analytical study directed toward optimization of space power systems of the SPUR type. Computer programming utilizing the existing LEADER procedure, with modifications, is being used. The initial phase included two illustrative problems which showed up the difficulties involved. The problems were not solved satisfactorily, but it is judged that the difficulties will be surmounted within the scheduled time. This report is quite fragmentary.


The final progress letter in a study of beryllium burnup for reentering nuclear auxiliary power systems reports on work done during August 1962. It was concluded that no toxic hazard from Be will be produced at ground level from complete burnup of a SNAP 10A reflector at the lowest possible altitudes, contamination will be 3 or 4 orders of magnitude below permissible levels and that complete burnup of the reflector to particles in the toxic size range (the worst case) is by no means certain.

A brief summary of work done during August, 1962 is presented on the re-entry phenomena of nuclear auxiliary power systems. Several runs were made with both hydrided and unhydrided U-Zr alloy fuel rod material. No data is presented. See the quarterly report on this task for July-September, 1962 for results.


A brief summary is presented of work done during October, 1962 on the reentry phenomena of nuclear auxiliary power systems. Simulation test runs for various trajectories were essentially repeats of earlier runs using equipment modified to achieve higher flux levels. No data is presented.
SECTION F - SOLAR SOURCES OF ENERGY


The objectives of work being performed are to determine the interrelationship between weight, surface accuracy, and structural integrity of a large, all-metal, solar reflector petal. In the selected approach the production of the parabolic petal is accomplished by utilizing an electroforming process. This report identifies the optical and structural test equipment to be used in connection with the production of a full set of solar reflector petals for assembly into an ultralightweight, high performance, unfurlable, solar concentrator suitable for use in space. Subjects treated in this report consist of structural testing of electroformed petal assemblies for determination of spring rate; optical testing of the same petal assemblies for definition of their geometrical accuracy; and a summary of available structural shake test facilities. As an essential addition, a survey of the vacuum depositing reflective coating facilities has been made and is reported herein.


The results of various temperature, radiation, and array configuration tests on 195 gallium arsenide cells are reported. The investigation of methods for interconnecting cells by means of gold-plated nickel tabs was continued. Work on cell standardization was continued. Measurements of the current-voltage characteristics of twenty cells selected as standards has suggested some changes in the experimental apparatus. Evaluation of cell tabbing techniques included measurement of shear strength, solder areas, and failure loads. Eight GaAs and twelve Si solar cells were irradiated with 17.6 Mev protons. The purpose of this experiment was to compare the radiation properties of GaAs and Si cells in filtered incandescent light and sunlight, as well as when extrapolated to outer space. One GaAs cell was irradiated at an elevated temperature to discover whether such operation substantially affects the radiation properties of GaAs. The cell measurements consisted of the spectral response and sunlight conversion efficiency, before and after bombardment. As expected, the major change in cell performance as a result of irradiation is the drop in short-circuit current due to a decrease in minority carrier lifetimes. In Si cells,
this decrease in lifetime occurs primarily in the base region, and it is manifested by a loss in infrared response. A consequence is the gradual decrease in output power as a function of bombardment. The response of GaAs cells, on the other hand, is due primarily to photons absorbed near the surface.

This report summarizes the more important points of the work performed by RCA Somerville, under contract AF33(616)-6615, during the past three years in developing improved solar energy converters from gallium arsenide. The report also contains a discussion of fabrication techniques including: (1) a table of the crystal used in this project, and wherever possible, the crystal properties; (2) a discussion of the five diffusion techniques used in this project. It is concluded that carrier gas systems are superior to closed systems and that the radiant furnace using carrier gas gives the highest efficiency cells, although the control on this type system is inherently poor; (3) a discussion of the various surface treatments used; (4) a comparison of polystyrene and silicon oxide anti-reflecting coatings, from which it is concluded that first-order 1/4-wavelength silicon oxide coatings are superior to polystyrene coatings; (5) a histogram showing roughly the distribution of conversion efficiency for each of the three years of the contract, and indicating that the yield of high efficiency cells is improving; (6) data for n on p cells, from which definite conclusions are not drawn because the work performed was not extensive enough; (7) an x-ray diffractometer trace showing that gallium phosphide can be formed on a gallium arsenide substrate by diffusion techniques; (8) a discussion of the methods for growing large area gallium arsenide single crystals; (9) a discussion of methods for epitaxial growth of gallium arsenide layers from the vapor phase. The experimental procedures employed are outlined in this report. Measurements for conversion efficiency, high temperature, and collection efficiency are described. Also described are the results of radiation damage experiments, investigations of the junction forward current characteristic, injection luminescence studies of gallium arsenide junctions, and a surface study of the short-circuit current pattern for silicon and gallium arsenide cells. Gallium arsenide cells are shown to be superior to silicon cells with respect to radiation damage and temperature degradation. The critical flux for radiation damage is approximately one order of magnitude higher for gallium arsenide. The temperature coefficient for fall-off of conversion efficiency for gallium arsenide is about 2/3 that of silicon. Several analytic investigations are presented, namely: (1) determination of the optimum grid structure, by incident energy, contact
2-245. (Continued)

and sheet resistances, and stripe thickness; (2) an investigation of solar cells with cascaded energy gaps, which shows that the short-circuit current of all the cells must be equal and that this is the limiting factor in improving the overall conversion efficiency; (3) the effect of a "built in" drift field; (4) a method of determining surface recombination velocity from the spectral response measurements; (5) a method of determining the short-circuit current for any energy spectrum from the spectral response curve; (6) an analysis of the optimum antireflecting coating. It is shown that silicon oxide has very nearly the optimum index of refraction and that the coating must be a zero order 1/4-wavelength coating for the peak of the solar spectrum. (Author)


The characteristics of a photoemissive solar energy converter were studied. The practical form of this device is a perforated sheet of a dielectric such as polyethylene terephthalate resin, coated on one side with a photoemitter and on the other with a low work function conductor. Photoelectrons emitted from the front surface by solar radiation fall back through the holes and are collected on the back surface. The resulting charge transfer builds up a potential difference which may be used to perform useful work. Models were constructed and tested in evacuated glass envelopes of ultraviolet transmitting glass, using a xenon arc as a solar simulator. The observed power conversion efficiency was of the order of 10^-4%. There was no correlation between converter efficiency and geometrical parameters. Measurements of spectral response showed good agreement with published values for Cs3Sb. Life tests did not show any deleterious effects due to the evaporation which would be expected to occur in space. Calculation of rates of evaporation indicated a life of 0.8 year at 27°C and 70 years at 0°C. The effect of oblique incidence was found by experiment to be a decrease in output at a lower rate with respect to angle of incidence than would be predicted by the cosine law. It was found that Cs-Sb surfaces can be formed at temperatures considerably below 120°C by co-deposition of cesium and antimony.

The objectives of this program are to demonstrate operation of at least 3 thermionic converters in series array using heat from a liquid metal loop to heat the cathodes and to provide a 1000 hour demonstration of thermionic operation. Cylindrical converters are to be used. During this period, the development of liquid-metal-tubing to converter bonding proceeded with (1) fabrication of plasma-sprayed alumina coatings for electrical insulation of the liquid metal tubing (Nb alloy) with thermal-conducting material and with (2) development of brazing techniques. Thermal transport analysis of the thermionic radiator system was initiated. The test loop design was modified to accept the cylindrical converter configuration. Analytical work is reported. Fabrication is scheduled to start during the next quarter.


This report summarizes the analyses made during the past six months for both photochemical energy conversion and thermal energy conversion by means of thermogalvanic converters. The objective has been to evaluate the photochemical and thermal reactions solely on the basis of their inherent limitations. A survey has been made of all endothermic photochemical reactions which have been studied and which show promise of converting solar energy in a practical manner. Two sets of analyses were made for the reactions. For the first set, values of $E_{\text{max}}$ are calculated on the basis of experimental values of wavelengths of light and of quantum yields. $E_{\text{max}}$ is the maximum efficiency obtainable for a given reaction and given wavelength when using solar energy, and is the product of two factors. One factor, $e_{\text{max}}$, refers to the maximum efficiency for any reaction when using sunlight. The second set of analyses summarizes data in part from a few of the better reactions in the first set of analyses, and in part from some other promising reactions for which insufficient experimental data were available for their inclusion in the first set. The basis on which the latter analyses were made differs from that of the earlier analyses in that values of $\lambda_{\text{max}}$, the maximum possible wavelength which can initiate the primary reaction, have replaced $\lambda$. Since the photolysis of water has long been of interest as a method of converting solar energy, in part because of its obvious application to hydrogen-oxygen fuel cells, a summary of the different methods for accomplishing this is given. The Hill reaction is very briefly discussed and analyzed. It is concluded that this reaction definitely can (continued)
represent energy conversion, but with very low efficiency. A short analysis is given concerning the maximum current densities which a certain class of photochemical converters would experience and qualitatively what effect this current density would have on efficiency. A survey of binary (two element) compounds was made under the previous contract. In this report an extension of that work has been made to include tertiary compounds. A number of organic hydrogenation reactions have also been included, together with a specific method for using them in a thermally regenerative fuel cell. It is pointed out that $dE/dT$, the rate of change of potential with temperature for a reaction, is an important criterion for selecting a reaction for a practical converter. Since this quantity is equal to $\Delta S/n$, where $n$ is the number of electrons per reaction and $F$ is the value of the Faraday, values of $-\Delta S^0/n$ were calculated for each compound. Melting points were also included, when available, for the compounds and their dissociation products. This property may also be very important in selecting a chemical system. A beginning has been made on the kinetics analysis, which will occupy the remainder of the program. Analysis has been restricted in this report to the kinetics of regeneration. It is pointed out that the kinetics analysis will be unable to rule out absolutely a given reaction; however, the analysis should show which systems have no kinetic problems, and which ones need more intensive investigation to determine the necessary catalytic conditions.


The objective of this contract was to further investigate the possibilities of solar energy conversion, utilizing the photovoltaic effect in thin polycrystalline silicon layers, and to apply the results by devising methods and techniques applicable to the fabrication of usable solar cells. Silicon deposition apparatus was designed and constructed to overcome some of the inadequate features of the apparatus being used at the outset of this program. This apparatus was used during the major part of this contract year. The deposition of silicon is accomplished by the thermal decomposition of silicon tetrachloride in a hydrogen atmosphere. A detailed description of the apparatus and procedure is presented in this report. Other equipment briefly described includes the lifetime apparatus, Hall measuring equipment, solar cell checker, resistivity measuring apparatus, diode characteristic measurement equipment, and infrared monochrometer equipment. Experiments were performed with this equipment, and the results are presented in detail. The results of these experiments lead to certain conclusions: The low temperature conductivity measurements indicate that conduction through the bulk of the individual crystallites is dominant. The tremendous decrease in conduction is precisely what one expects qualitatively for single crystal
were the grain boundaries to provide important conduction paths, like fine metallic wires encircling each crystallite, no such drastic conductivity decrease could be expected when the samples were immersed in liquid helium. In analyzing the resistivity and Hall data on the polycrystalline layers, the following results were found. The Hall coefficient measured on a polycrystalline layer indicates a carrier concentration which agrees well, in all cases so far, with the value deduced from resistivity measurements on the accompanying single crystal layer. However, the resistivity of the polycrystalline layer is always higher, to a greater or lesser degree, than that of the single crystal layer. Therefore, the two types of layers have about the same carrier concentrations, but the mobility is lower in the polycrystalline layer, presumably due to grain boundary scattering. An analysis of the data shows that a rather unusual relationship exists between grain boundary scattering and impurity concentration. At the lower impurity concentrations, the dependence of the grain boundary scattering strength on impurity concentration is qualitatively reasonable; the grain boundaries can probably be treated as impenetrable regions in the crystal due to space charge layers at the boundaries, in which case the scattering cross-section should decrease with increasing impurity concentration, as was observed. However, the peculiar behavior of the grain boundary scattering at the higher impurity concentrations is not understood. It has been suggested that a grain boundary in silicon acts as a donor level similarly to the manner in which it would act as an acceptor level in germanium. With the many combinations of dangling bonds that could exist in polycrystalline silicon, many energy levels might be expected, ranging from those easily thermally ionized, to extremely deep levels. Certainly, the optical absorption and photoconductance data support this suggestion. On the other hand, since the resistivity of small crystallite layers is always higher, this scatter of points may be directly related to varying degrees of grain boundary scattering. Heat treatment of these samples went in the P-type direction. As previously stated, x-ray back-reflection photos made on two of these samples revealed a definite increase in crystallite size. Such a process would anneal out many of these donor levels. The fact that the N-type samples increased in resistivity precludes the possibility of increased mobility. In silicon P-N junctions, the reverse saturation current due to generation of carriers within the space charge layer region is many times larger than the "ordinary" diffusion current which the classical P-N junction theory of Shockley describes. It is possible to show that the forward current at low bias voltages is dominated by recombination within the space charge region, which is just the inverse of the generation mechanism. As predicted, the reverse saturation current should become smaller with an increase in majority carrier concentration of the base layer. The exact opposite is shown by the experimental curves. Here again, as in the optical transmission measurements, the impurity atoms play a part. A detailed discussion of many electrical properties of the cells is presented in this concluding section of the report. Three distinct advantages of the N- or P-type configurations for polycrystalline solar cells are discussed: (1) They tend to have better reverse current characteristics and thus a smaller (continued)
reverse saturation current. (2) The minority carrier diffusion length in the base P-type layer is greater for a given lifetime. This should improve the "far side" collection efficiency, particularly in the larger crystallite layers. (3) The polycrystalline resistivity of P-type layers is lower for a given impurity concentration than is N-type. This permits the use of smaller base doping levels without adding to the series resistance of the device.

PHOTOEMISSION SOLAR ENERGY CONVERTERS.

The use of photoemission to convert solar photon flux into electrical power and the development of a high power-per-unit-weight photoemissive solar power converter suitable for use in space were considered in this study. Several interim WX-3964 experimental photogenerators were constructed, the latter displaying an increase in efficiency with a decrease in anode-to-cathode spacing, and an efficiency of 0.01% at the relatively large anode-cathode spacing of 70 mils. Analytical and technological studies which were carried out show that a 2% photoemissive solar converter should be potentially competitive with photovoltaic converters on a dollars per watt and watts per pound basis. Task A was concerned with all the necessary analyses, processes, and techniques required to make the component parts of the photoemissive solar power converter. Over the two year period this task was broken down as follows: electron optics study, electrode surface studies, bell jar processing mechanism, glass tubing processing system, WX-4220 photoemission control tube, and measurement procedures. Several conclusions are presented on the basis of the results of this work. Electron optical studies carried out on both the rubber membrane potential analog and the resistance paper analog showed that a close-spaced configuration was desirable not only from the standpoint of minimizing the effect of space charge, but also desirable for increasing the collection efficiency and decreasing the electron optic barrier. The most information was obtained from the resistance paper analog, but the rubber membrane analog was useful in its own right in presenting the approximate trajectories within the device and giving an intuitive appreciation of the effect of the variation of electrode potentials. While the WX-3964 experimental photogenerator was not successful in determining the proper electrode surface as originally intended, it gave valuable data relating to processing and enabled measurements to be made of the effect of anode-to-cathode spacing upon the efficiency of the photogenerator. Furthermore, measurements taken with this tube led to the establishment of an equivalent circuit representation of the photogenerator, making it possible to explain certain phenomena observed during field tests performed upon this device. The WX-4220 photoemission control tube helped materially in determining the degree of processing of the (continued)
component parts of the photogenerator. Work with this tube led to the
development of plated antimony and manganese evaporators and the proper
procedures to be used in making cesium generators, using cesium chromate-
silicon powder. Experiments carried out with the bell jar processing sys-
tem also showed that the degree of cleanliness and vacuum necessary to
make a satisfactory photoemissive surface was high. In a negative fashion,
the fact that none of the photosurfaces within the bell jar processing lasted
a satisfactory length of time, indicated the necessity for keeping the number
of parts within the processing system to an absolute minimum, restricting
the volume during cesiation, outgassing, and heating not only the substrate
but all other surfaces within the vacuum chamber during cesiation. It
underlined the fact that although a particular vacuum system was entirely
suitable for thin-film evaporations, this did not mean that it could be used
for photosurface deposition unless proper and particular precautions were
taken. From a mechanical point of view, the bell jar was also unsatisfac-
tory due to its limited provision for access within the vacuum enclosure.
These considerations led to the glass tubing processing system, the design
of which was intended to override the disadvantages inherent in the bell jar
processing system. Unfortunately, this design was still under construction
at the time of termination of the contract, so was not proven. Its primary
features were the capability for cesiation within a small volume, and the
provision for an adequate sealing method that could be carried on while the
device was under vacuum. The system of measurements established during
this program not only involved measurement of photogenerator character-
istics, but also included measurement of the processes involved. The auto-
matic readout unit constructed for this program enabled a constant check to
be maintained upon the vacuum system without the need of an attendant
operator. During processing, various parameters could be recorded while
observing progress of the process visually. Design of the WX-4209 repre-
sented the first step in the construction of a thin, sealed-off photogenerator.
This design, was chosen as the best structure that could test sealing
methods, deposition procedures, and compatibility of materials. At the
time of termination, the majority of the processing necessary was deter-
mined and the sequence of assembly established. Many of the parts had
been fabricated and provisions were being made to start assembly of tubes.
The exact nature of the final seal had not been determined and was to have
been determined with the use of these tubes. Theoretical conclusions con-
cerning the complete project are presented along with recommendations.

2-251. PHOTOGENERATOR POWER CONVERSION STUDY.
no. 3, for June, July, Aug. 1962. Westinghouse Electric Corp.,

Westinghouse Electric Corporation is investigating ways and means of con-
structing developmental models of a close-spaced, thin, solar power

(continued)
converter using the photoemissive principle. This report notes the completion of the processing system, its shakedown, and preparation for processing. It notes minor changes in fabrication techniques and reports on electrical tests made on a converter delivered by Westinghouse Astrotechnics. This device had an adequate photoemission but showed no converter action, due either to the large spacing between photocathode and anode, or to a high work-function anode. Three major designs of the photogenerator are being worked on. These have been chosen on the basis of a structure having the minimum number of component parts and processing techniques. Difficulty experienced in shorts between the photocathode and anode has been traced to rupture of the insulating film on the anode by the bulge at the cathode contact made by the silver paint. Work on the design of a thin-glass small aperture converter is reported. The construction of the tube delivered by Westinghouse Astrotechnics Department was changed to side-step the difficulties in sealing the glass-sandwich design. Improvements in the procedure for assembling the anode subassembly are described. These include: (1) chemically depositing chromium on the edges of the silver anode to eliminate excessive alloying of the indium on the anode; and (2) careful machining of the indium after deposition of calcium fluoride on one surface of the anode mesh, providing the correct sealing characteristics to the glass disc. Tests of a sample photogenerator, by exposure to sunlight, gave no measurable results. Measurement of this device as a photodiode (positive voltage applied to the anode) gave a current of 390 microamps with 10 volts applied. Evidently the anode-to-photocathode distance is much too great, so that excessive space charge exists, and the tube cannot operate as a converter. A description of work on the vacuum processing system is included.

RESEARCH ON IMPROVED SOLAR GENERATOR.

Theoretical studies directed toward the achievement of very high efficiency solar cells were undertaken. An entirely new method of P-N device analysis, referred to as the "flux method," was evolved. The flux method is characterized by analysis of the carrier fluxes rather than the carrier concentrations. This yields solutions to problems which cannot be treated simply by diffusion calculations. It is expected that shallow junction cells will yield higher efficiencies if suitable technology can be developed. High surface recombination in these cells will be less significant in reducing overall efficiency than will be the case in relatively deep junctions. Extensive studies were undertaken for material evaluation and the determination of optimum material parameters such as resistivity, orientation, grade, etc.

(continued)
Since the overall efficiency of a solar cell is inversely proportional to the reverse saturation current, extremely doped regions are desirable on either side of the junction, resulting in a high built-in electric field. This resulted in the selection of low resistivity (0.1 to 0.2 ohm-cm) silicon as a starting material. Solar grade material apparently yields better efficiencies than Grade 1 silicon. Gallium arsenide solar cells of both N+P and P+N type were prepared. Efficiencies in the order of 15% and 14% were obtained in the case of boron diffused Czochralski crystals and silicon dendrites, respectively. This indicated conclusively that dendrites can yield as efficient solar cells as Czochralski crystals. N+P solar cells using crystal and phosphorus diffusion were prepared with efficiencies better than 10%. It is expected that better efficiencies can be attained with further work. 

A heat-exchanger has been assembled and tested to determine the performance of the waste heat radiator and to determine the heat losses from the thermoelectric power converter. A thermoelectric module efficiency tester was built and a typical section of thermopile was tested. The test results show that the power required by the thermocouples is 30% greater than anticipated. The power input is greater than anticipated since the efficiency of the couple is 15% lower and the power output is 13.9% greater than calculated values. Delays are still being encountered in work with the thermal energy storage system.

An absolute theoretical upper limit of efficiency has been established for solar cells utilizing photoelectric generation in p-n junctions. This limit, which was derived for the first time in connection with this research, depends only on basic quantum mechanical and statistical mechanical concepts and not on empirical constants which may be determined chiefly by the current state of the technological art. It gives a limit of efficiency of about 30% whereas previous theories have given about 23%. The best actual efficiency achieved is about 15%. Important extra sinks of current have been found in internal parts of the photocell p-n junctions. These sinks are found (continued)
to change with temperature treatments that would be insufficient to produce
significant disturbances of ordinary donor and acceptor distributions. These
observations strongly suggest that in the commercial solar cells studied,
there are present chemical impurities which deteriorate performance. The
nature of the impurities cannot be determined from the results obtained;
however, similar sensitivity to heat treatment was deliberately developed
in diodes exposed to contaminated furnace atmospheres which might contain
gold as well as other metals. One of the unsolved problems concerning
silicon solar cells is that the current increases less rapidly with voltage
than predicted by established theories. The factor \( A \) by which the dependence
of current upon voltage is reduced is found experimentally to be fre-
quently larger than 3 or even higher than 4, whereas established theories
for interior effects do not predict values higher than 2. A further theoretical
process, known as the Auger process, which might have an important influence
on junction characteristics and limit solar cell efficiencies, has also
been investigated and found to have little probability of being important.
The most likely explanation of the high A-values appears to be the presence
of precipitated metal particles in the space charge layer of the p-n junction.
SECTION G - THERMIONIC DEVICES


The principal purpose of this investigation was to determine the feasibility of neutralizing the electron space charge in a cesium thermionic converter with ions formed by the collisions of electrons with neutral atoms. Equations were set up for the ion density and subsequent potential function for a low-pressure plasma in which the ions are formed by collisions of atoms with electrons emitted from the cathode with a Maxwellian velocity distribution. The special case of zero field at the emitter was taken to simplify the mathematics. Voltage versus current characteristics were determined experimentally for a cesium diode with a low work-function impregnated tungsten emitter operating at 1410 K and an electrode spacing of 0.254 cm. Cesium vapor pressures corresponding to condensation temperatures of 30°C, 100°C, and 155°C were used. The results indicated that higher cesium pressures, leading to more dense plasmas and successive collisions between electrons and excited atoms, are needed for complete space-charge neutralization. (Author)


The objective of this program is to define and minimize the life limiting and degradation factors affecting the usefulness of the low-temperature cesium vapor converter. In line with this objective, work during the second quarter has been directed toward completion of the life test equipment, initiation of life tests, and the acquisition of operational data. Two state-of-the-art cesium vapor thermionic converters have been started on life test at a cathode temperature of 1450°C. To date, tube No. 4 has accumulated 320 hours of life at a total power output level of 5.8 watts while tube No. 6 has accumulated 130 hours of life at steady state operation at a power level of 9.1 watts. A third tube has been mounted in a life test chamber and the system is presently undergoing exhaust prior to initiation of life test. Several figures illustrating various features of the life test chamber and the system are presented along with a detailed description of the experimental test procedure. Initial volt-ampere characteristic curves for tubes at cathode temperature of 1350°C, 1450°C and 1550°C are presented. The effect of both load and temperature variations on converter performance is an important factor from an overall system point of view. Instabilities in the converter performance as a result of temperature variations in either cathode,
anode, or reservoir, affect over-all system operation. Although transient characteristics will be determined by the thermal structure of any given system, the individual tube transients will be useful in understanding any unforeseen instabilities and as an indication of the operation of a practical device under these conditions. Therefore, transient data is accumulated for all life tests carried out in this program and is being presented for one tube under test during this quarter.


The objective of the program is to determine the feasibility of enhancing the performance of cesium vapor thermionic converters at low temperatures (1200° C) through photon processes. These photon processes are expected to increase the power output of a cesium converter by reducing the drop through the converter under load conditions. To date, the study and preliminary calculations have yielded the following results: (1) The most promising approach appears to be in the use of the 3888Å helium line to excite the cesium vapor 8521Å resonance line through resonance and cascade fluorescence phenomena. (2) A possible but less promising scheme is to use the band of near ultraviolet wavelengths between 2000Å and 3184Å in the sun's spectrum for photo-ionization and excitation and for initial breakdown effects. (3) Use of the resonance lines 8521Å and 9844Å of cesium will require a unique tube design in order to introduce the radiation to the plasma region, because the vapor is opaque at these wavelengths. It may, in fact, be impossible to introduce the radiation in a practical device. The planned experimental tube will be a cylindrical converter with a sapphire window on one end through which the annular interaction space can be irradiated with light of appropriate wavelengths. The light source will be a helium discharge tube.


A new static heat-to-electricity conversion principle is described which attempts to duplicate in the solid state at a lower temperature the high impedance performance of a vacuum thermionic converter. The basic arrangement is a multilayer structure of thin film semiconductors or
Thermionic
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2-258. (Continued)

insulators perpendicular to the heat flow, separated by thin layers of metals. The optimization of the transverse mean free path of the charge carriers, film thickness, contact potentials and other parameters is discussed. An outline of the exploratory experimental study of this new concept, initiated recently, and some practical details on film production and measurements are given. (Author)

2-259. FLAME HEATED THERMIONIC CONVERTER RESEARCH.

The nominal 150-w electrically heated thermionic converter was successfully assembled and tested. The operating information obtained, such as volt-ampere curves and thermal conductivity of cesium vapor, indicates that the large diode behaves essentially the same as do the smaller ones regularly tested at Atomics International. Improved aspirated burners were constructed and tested. Some of these operated silently, and attained new highs in temperature at the simulated emitter surface. From all aspirated burners so far tested, it appears that the desired quiet mode of operation can only take place when the air is preheated to below ignition temperature for the air-fuel mixture. This limits the heating efficiency of propane fuel to less than 40%. If higher heating efficiencies are required, it will be necessary to use a fan to push the air through the heat exchanger and combustion chamber assembly. Practical means have been found to control the temperature of the emitter, collector, cesium reservoir, and fuel injector. The use of capsules, containing liquid at its critical point, as a means of controlling temperature is explained. Evaluation of Durak-B coated molybdenum continues. A wire, coated with 2.5 mils of Durak-B, was heated to 1730°C in air for 650 hr before failure occurred inside one of the quartz end protectors. Also, another 2.5 mil Durak-B coated wire was heated in air to 1840°C for 422 hr before failure occurred at the center.


Detailed post-test analysis of a test cell (E) has been completed. The results emphasize the necessity of improving the cleanliness of future cell (continued)
assemblies and especially the need for techniques to determine the emitter condition throughout final assembly. The next test cell (F) contains a 10 UC-90ZrC emitter. Thermocouple installation and calibration and high temperature vacuum emission tests are being conducted prior to final closure of the cell. Recent vacuum emission studies carried out under other contracts on three uranium carbide compositions (10 UC-90ZrC, 30 UC-70 ZrC, and 90 UC-10 UC) demonstrate that emission behavior is essentially independent of bulk uranium content. The presence of some uranium on the emitting surface is of the utmost importance. Intentional "poisoning" of the surface of sample emitters has been produced by exposure to air at room temperature. It has been concluded that the emitter surface must be activated in addition to providing a clean cell during operation.
SECTION H - THERMOELECTRIC DEVICES

No entries are made in this issue.
II. MATERIALS

SECTION A - GENERAL


The high temperature thermodynamic properties of argon in ionization equilibrium were obtained by the methods of statistical thermodynamics. Property values at the stagnation point and across traveling, standing and reflected normal shockwaves were obtained by employing the basic laws of fluid mechanics. A shock tube choked flow analysis is presented. In addition, ideal, ideal-choked, and real gas stagnation point heat transfer calculations are performed. Transport properties based on prior Boeing work and perfect gas relations are included for completeness. The numerical computations were executed on the IBM 7090 digital computer, and the results are presented in both graphical and tabular form.


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. 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. The most efficient operating voltage and the power capabilities of x-ray targets are evaluated. The basic concepts necessary for the development of a uniformly well-focused x-ray tube electron beam are described. Construction details of the x-ray tube are given, together with a brief description of the high voltage supply and an evaluation of the overall x-ray tube power supply system. 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. (Author)
2-263. BASIC STUDIES IN RADIATION EFFECTS ON MATERIALS.

The effects of radiation on any given material depend on the physics of the primary interaction with the material and the complex chemistry initiated. In the present studies, the combination of a coincidence mass spectrometer (patented) and a special x-ray tube, described in Part I, has made possible direct measurement of the ion fragmentation patterns produced by x-ray impact on simple gas phase molecules. Mass spectra are reported for nitrous oxide, and for propane under bombardment by low energy x-rays (predominantly aluminum Kα). For comparison purposes, spectra of these materials under bombardment by 1200 ev primary electrons were also obtained. The present studies also concern measurement of the secondary electron energy distributions for a number of molecules of importance in radiation chemistry, at a fixed angle, using primary ionizing electrons in the energy range of 100 to 1000 ev. Measurement of the electron energy spectrum is accomplished in the coincidence mass spectrometer. Theoretical interpretation of the data in terms of radiation chemical effects is included. Design, construction, and operation of the instrumentation which has made these measurements possible, are described. (Author)


The concept and early work done in the field of expandable space structures is covered herein, together with a justification for an auxiliary flight experiment. The problems involved in the design and fabrication of these structures are analyzed along with the solutions used in the manufacture of experimental models. The support work required to integrate this experiment into the design of the Mark 4, Mod 4 reentry vehicle, involving such factors as packaging and storage, instrumentation, and modification of the reentry vehicle, is discussed. Work on the flight test experiment for expandable space structures has progressed to the extent where fabrication of a full-scale model could be executed with a reasonable probability of success. Full-scale masts have been fabricated and erected satisfactorily. The support work involving the redesign of the Mark 4, Mod 4 access door and construction of a fiberglass mockup has been completed. Work with the 4-foot model has determined valuable techniques for folding, ejection, and
expanding lightweight aluminum foil structures. An ultrasonic welding machine was acquired for use with aluminum foils. Work is continuing on the study of physical properties of foil materials, mechanical characteristics of thin foil structural elements, and other related light-weight structure projects.


Small pyrex glass spheres, representative of stoney meteoroids, were fired into double-sheet structures at velocities to 24,000 feet per second to determine the effects of a number of target variables upon penetration resistance. It was observed that the impacts could be classified into various categories as a function of the impact velocity: a low-speed impact region, a transition region, and a high-speed impact region. The regions are discussed and illustrated by individual frames from high-speed motion pictures of the impact process. It is noted that results obtained in one region of impact are not applicable to the others. The effects of combined sheet thickness and sheet spacing upon penetration resistance were investigated for the low-speed and the high-speed regions of impact. It was determined that the required total sheet thickness varies with the first power of the velocity in the low-speed impact region and appears to vary with the $2/3$ power of the velocity in the high-speed impact region. It was also observed that increases in spacing between sheets resulted in only slight increases in penetration resistance at low speeds. However, in the transition and high-speed impact regions, spacing became very much more effective in controlling performance of the structure. It was concluded that structures having front sheets with equal mass per unit area and identical rear sheets have the same penetration resistance for several front-sheet materials. Also, for maximum penetration resistance per unit weight, the available structural weight should be concentrated in the rear sheet with the front sheet only thick enough to shatter the projectile completely. Discussion and illustrations of oblique impact on double-sheet structures are also presented. (Author)

An investigation of chemical reactions of ablating species from low-observable heat shield materials is being conducted. An objective is the prediction of the radiation signatures which result from injection of these species into the boundary layer and wake. Diffusion and premixed flames of pyrolysis products with oxygen or air are established and analyzed chemically, spectroscopically, and electrically. Flames of tetrafluoroethylene, the major pyrolysis product from Teflon, and oxygen have been established and chemically sampled. Theoretical calculations of reaction products at the adiabatic flame temperature have been made for this system at several stoichiometries. A program for the investigation of effects of exposing materials to extreme thermal stresses, using the arc-imaging furnace, is described. (Author)


Cerium sulfide was prepared, and some x-ray diffraction studies were performed on it after various heat treatments. Previously derived equations relating equilibrium pressure of the more volatile component in a nonstoichiometric compound to the degree of deviation from stoichiometry were applied to some additional metal hydrides. It was demonstrated that the interaction energy between defects and the range of homogeneity of a nonstoichiometric compound are related. From available pressure-composition-temperature data on some rare earth dihydrides, the intrinsic disorder, or concentration of defects in the stoichiometric compound, and the defect formation energies were computed. (Author)

2-268. MATERIALS AND STRUCTURES FOR SPACE STATIONS. Robert S. Osborne, Clarence O. Keffer, and George Look (Langley Research Center, NASA, Hampton, Va.). Astronautics 7(9)36-9 (Sept. 1962).

A major concern in the development of manned orbiting space stations will be materials and structures, especially for the crew's quarters or cabin. Living quarters might be constructed of rigid modules, or of a combination of rigid and inflatable sections, or inflatable sections made entirely of flexible materials. The state of the art of flexible materials and inflatable structures being many years behind that of rigid materials and structures, the
author concentrated effort on inflatables. Early configuration studies indicated that the cabin might be toroidal. Several ways of constructing the load-carrying portion of inflatable toroids have been investigated. One of these employs a filament cage and bladder. Design considerations including pressure loads, weight of the structure, and materials used, their strengths and other characteristics are presented. A model having an over-all diameter of 24 ft. and a cross-sectional diameter of 8 ft., has been built. The cage is constructed of dacron cords arranged meridionally around the torus. The bladder is made of butyl-impregnated nylon. Results of various tests on this model are described. An isotensoid concept for structural skin has also been investigated. This filament wound structure is so constructed that all primary loads are carried by filaments or cords having equal tension, and no load is carried by the flexible binder or elastomer. A 45 in. OD torus of this construction was studied. A third method of construction was studied. This method employs the pattern-layup method, gores of special three-ply fabric being joined to form the torus. Each ply is made of a flexible elastomer and flexible cord reinforcements with all the filaments in a given ply being parallel to one another. A description of a laboratory to test properties of materials in conditions comparable to a space environment includes facilities for hard vacuum, UV radiation, and large temperature extremes over extended periods of time. Also described is an apparatus for accurately determining gases emitted by the exposed materials. Results of tests on composite materials carried out at this lab are presented.


Gamma ray induced changes in the conductivity of CdS crystals are studied as a possible mechanism for monitoring gamma dose rates. Data is presented on the change in conductivity due to gamma exposure over a range of $1.4 \times 10^5$ ergs/g hr (C) to $5 \times 10^7$ ergs/g hr (C). The change in photocurrent as a function of voltage at two dose rates is also reported. The rise time of this photocurrent is investigated for photons in the visible light range as well as for Co 60 gamma rays. (Author)

This progress report contains 57 survey articles relating to: (1) high-dielectric-constant materials; (2) high-temperature dielectrics; (3) dielectric and elastic spectroscopy; (4) crystal physics; (5) magnetics; (6) mathematical crystallography; (7) magnetic spectroscopy; and (8) optical modulation.


The exposure of organic materials to ultraviolet light and ionizing radiation results in absorption of energy which produces excited molecules. Among other paths, either this excitation energy may be transferred to neighboring molecules that lose their energy by emission of a photon, or the excited molecules may initiate photochemical reactions of various kinds. These two processes are intimately related to the behavior of organic materials in a space environment and both are being studied from a fundamental point of view. The energy transfer process is being studied by using molecules that are excited to a triplet state by ultraviolet light. These molecules are mixed in solution with other molecules (acceptors) which can accept energy from the first kind being excited (donors) to their triplet states. The energy transfer between these molecules is being investigated by using electron paramagnetic resonance methods since this technique allows the direct observation of molecules in the triplet state. The experiments are carried out at about 77°K in suitable organic gases. Measurements of half-lives of the triplet state of several molecules have been made and theoretical studies of the decay process are in progress. Theoretical treatment of the decay problem is complicated by the fact that transition probability for the exchange process is a sensitive function of the distance between donor and acceptor molecules. At present, it appears that an approximate analytical solution to the problem can be obtained. The photolysis of phenyl isopropyl ketone was carried out using the 3130 Å line of a mercury arc. The reaction products were found to be those expected for a free radical degradation mechanism. It had been expected that this reaction would proceed by an internal rearrangement. Experiments are now in progress to establish the details of the mechanism. (Author)

This report virtually duplicates the format of Emerson Electric Mfg. Co. Report No. 1139. The subject is another member of the group of proprietary materials developed under the trade name "Thermo-Lag" subliming compounds. Extensive properties information is given but the composition is not reported.


This report presents the important thermodynamic, physical and mechanical properties of a new material, a "THERMO-LAG" subliming compound, which is offered for use in hypervelocity escape, reentry and ballistic vehicles. "THERMO-LAG" is the trade name for a family of sublimating, paint-like, moldable inorganic compounds developed on a proprietary basis by the Emerson Electric Mfg. Co. The composition of the material is not reported, but the mode of operation is described and extensive properties information is given. The report is company-sponsored and not contractual.


The report consists of evaluations of work done on radiation effects during 1961-1962 in each of the following fields: (1) electronic components and equipment, semiconductor devices and materials; (2) polymeric materials; (3) fuels, lubricants, and hydraulic fluids; (4) structural metals and alloys; (5) ceramics. Work being done on the space environment as it pertains to radiation effects is also summarized, and a section is devoted to progress in dosimeter hardware. Each section contains a brief description of work done in important areas of the subject, a short summary of important results, a listing of agencies and contractors working in the area, and conclusions and recommendations. A bibliography of 152 reports is included.
2-275. RESEARCH ON TRANSITION METAL-CARBON CHEMISTRY.

The interaction of tri-iron dodecacarbonyl, Fe₃(CO)₁₂, with octafluorocyclo-hexa-1, 3- or 1, 4-dienes forms a compound, C₆F₈Fe(CO)₃, which must be formulated as a π-bonded olefin complex. In contrast, the interaction of tetrafluoroethylene with Fe(CO)₁₂ gives a compound, C₄F₈Fe(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 CO₂(CO)₈ and (π-C₅H₅)₂Co and of the latter with trifluoromethyl iodide are described. (Author)


Electrons and holes were produced in mixed crystals of KCl-KBr and KCl-RbCl solid solution systems by irradiation with 3-Mev electrons at 190°C, -80°C, and 20°C with doses of from 10⁵ to 10⁶ rads. The optical absorption resulting from the trapping of electrons and holes was measured at -190°C (liquid-nitrogen temperature) to reduce bleaching. The mixed crystals of KCl-KBr and KCl-RbCl irradiated at liquid-nitrogen temperature contained F, F', K, and V₁ centers. The F and K centers appeared at -80°C (dry-ice temperature) while at 20°C (room temperature) irradiations the F, K, and M bands were observed. Some other bands have been also observed but not studied. The Mollwo relation (λ = const. d²) is not valid for the shift of the maximum of all bands observed, possibly because of lattice distortion. The center frequency of the F' band shifted with composition opposite to the F-band deviation. In pure crystals the center frequency of the F' band was found to be lower than previously observed. The half-width of the F band is greater in mixed crystals - its maximum approximately corresponding to the microhardness maximum and the maximum deviation from linear of the exciton band-edge shift. The V₁ band half-width in the KCl-RbCl system decreases contrary to that in KCl-KBr. Intensity of the F band-versus-composition curves for KCl-KBr exhibit both a relative maximum and a relative minimum, but in KCl-RbCl only a deep minimum. In no case did a mixed crystal color more than pure KBr, contrary to what might be expected from the large distortions present. Curves for the F' and V₁ bands follow the general pattern of those of the F band but with reduced intensity. (Author)

The transient ablation process for non-radiating glassy materials is discussed and numerical calculations are presented for quartz along an ICBM trajectory. It is shown that the transient effects do not appreciably affect the amount of ablated material along the trajectory, but that they affect very significantly the amount of insulating material required after ablation ceases. The transient ablation analysis is also applied to quartz under arc wind tunnel heating conditions and it is shown that the experimentally obtained ablation history can be used to deduce the high temperature thermal diffusivity of the ablating material.


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. (Author)
SECTION B - METALLURGY


Results from 17 investigations conducted on the tensile properties of tungsten are compiled and correlated and the data evaluated, taking into consideration factors such as the starting material, fabrication history, strain rate during testing, and surface condition of the samples which influence the tensile properties.


An increased hardness of grain boundary regions above that of the bulk material is found to exist generally in intermetallic compounds having a stoichiometric excess of active metal component. This hardening is shown to be associated with the anomalously high brittle-ductile transition temperature in these materials, and to be related to the "pest" phenomenon. The presence of adsorbed oxygen and/or nitrogen in grain boundary regions is found to be responsible for the increased hardness in these areas; the precise manner in which the resistance to plastic deformation is increased in this phenomenon is not clear. The effect can be modified, however, by ternary solute additions and to some extent by appropriate annealing treatments. (Author)


Electrochemical reactions at metal-crystalline salt interfaces were investigated. The cathodic reduction of silver ion, mercuric ion, cupric ion, and thallous ion gave characteristic current voltage patterns from which information about the electrode reactions could be deduced. The reduction of oxygen gas and water, and the oxidation of iodide were also observed. Applications of electrical methods to chemical analysis are discussed. (Author)

The purpose of this investigation is to select a material and to develop a process for fabricating an integral rocket motor case (with no weldments) by shear spinning the material while in the metastable austenitic condition. It is intended that such a case will develop hoop strength values in excess of 300,000 psi. During the first semiannual project period a literature review was completed. Three alloys; Type H-11 hot work steel, AM 355 semi-austenitic stainless steel, and 18 NiCoMo (300) mar-aging steel were selected for shear spinning evaluation. The selected materials were purchased, extruded and shear spinning preform blanks machined. Subscale pressure cylinders with 0, 25, 50 and 75% reductions were produced and final machining of test specimens started. One pressure vessel has been tested to failure. The available results of shear spinning and biaxial burst testing are presented in this report.


This publication sets forth specifications for the process of applying polytetrafluoroethylene (Teflon) coatings to aluminum alloys in work done for BUWEAPS.


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, MoC₀.₆₇, to the cubic phase, MoC₀.₆₇₊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 (continued)
2-284. (Continued)

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. (Author)

University of Cincinnati, Cincinnati. The Kettering Laboratory.

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Recommended Activities

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2-286. GAS-METAL REACTIONS IN ROCKET NOZZLES.
James D. Batchelor, Stewart W. McCormick,
R. Stephen Scheffee, and Eugene L. Olcott, Atlantic Research
June 1962. 84p. illus. Contract: AF 33(616)-7744,

Results of the first year of a program to study the chemical reactions be-
tween refractory metals and the hot combustion products of solid propellants
are summarized for tungsten, tantalum, and the 90 per cent tantalum-10
per cent tungsten alloy. Three of the propellants used represented state-of-
the-art and development systems with flame temperatures from 4700°F to
6500°F. A fluorocarbon propellant was also used to define specifically the
effect on the metals of a substantial HF concentration in the combustion
products. The refractory metals were exposed directly to the action of pro-
pellant combustion products in burning propellant strands. Hot filament tests
were also performed in gaseous atmospheres containing each of the major
gaseous ingredients of the propellant combustion products and selected mix-
tures of these gases. The laboratory studies proved conclusively that these
experimental procedures yield useful and accurate data on the reactions be-
tween refractory metals and propellant exhaust products. Conclusions drawn
from the laboratory tests agree with rocket nozzle tests. In particular, it
was shown that oxidation by CO₂ and H₂O represent the principal modes of
attack on each of the refractories studied. Tungsten is essentially unaffected
up to its melting point by other gas species, including HCl and HF. Tantalum
is attacked by both HCl and HF but these reactions are of secondary impor-
tance compared to oxidation. The alloy behaves similarly to tantalum but is
less reactive. Carbide formation, which has been noted in nozzles tested in
rocket motors, was clearly demonstrated in the laboratory tests. The ex-
perimental observations agree reasonably with predictions from thermody-
namic calculations. This program will continue and will include other
refractory metal alloys and other propellants selected to determine the effect
of various chemical environments which may exist for future propellant
systems. (Author)

2-287. IMPURITY DEPENDENCE OF CREEP ALUMINUM OXIDE.
of Utah, Salt Lake City, Utah, ASD TR 61-481. April 1962.
66p. illus. Contract: AF 33(616)-6832, Proj. 7350, Task
735000. 72 refs. A62-7102.

Polycrystalline sintered compacts of doped Gulton Alucer MC alumina have
been deformed in three point beam loading in the temperature range 1000°C
to 1350°C. Creep consisted of a transient deformation (εₜ) superposed on a
steady state deformation (εₘ = C₁·t). The steady state creep rate of
specimens doped with MgO or MnCO₃ from 50 to 5000 ppm by weight were fit

(continued)
to the expression $A \exp(-E/kT)$. The activation energy $E$ was found to be 130 Kcal/Mole independent of added impurity. The constant $A$ was independent of MnCO$_3$. 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 ($\varepsilon_{tr}$) occurring on application of stress approximated the relation $\varepsilon_{tr} = C_2 [1 - \exp(-t/\tau_2)] + C_3 [1 - \exp(-t/\tau_2)]$.

On removal of the load, recovery was observed which followed the relation $\varepsilon_{rec} = C_2 \{\exp(-t/\tau_1)\} + C_3 \{\exp(-t/\tau_3)\}$

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. (Author)


Studies on pyrolytic graphite have been made with respect to its possible use as a reentry heat shield material. Preliminary work is reported on changes in x-ray parameters and physical dimensions caused by heat treatment above the temperature of deposition. Data are presented which indicate that the pyrolytic structure becomes similar to that of natural graphite after treatment to 3000°C. Initial studies on structural changes as a function of time are reported. (Author)


This report summarizes work accomplished during the period Oct. 1960-Oct. 1961 in a continuing investigation of graphite technology aimed at development of premium quality, reproducible graphite-base materials for missile and aerospace applications. Raw materials, fabrication and material characterization and evaluation are covered. In each of these areas, basic research was undertaken to supply fundamental information. Applied research dealt with the characterization of materials and processes which cannot be
elucidated by reasonable efforts in the basic research, and development efforts were concerned with study of the materials and operations involved in fabrication of gross graphite bodies. Interim results of those mutually supporting efforts are given. In the raw materials area, extensive progress has been made on the delineating of the chemical reaction steps and physical processes involved in the pyrolysis of pure hydrocarbon compounds first to pitch-like materials, then to coke, and finally to graphite by use of differential thermal analysis, absorption spectroscopy electron spin resonance, and other selected techniques. Preparation of a synthetic pitch fromacenaphthylene has provided a reproducible material which can replace coal tar binders for process studies. Studies by Union Carbide European Research Associates on coal tar materials used as binders have made good progress on the characterization of molecular components and have tentatively indicated the presence of significant numbers of molecules consisting of clusters of small aromatic units in addition to the other components. Progress is reported on study at the Armour Research Foundation on: (1) interaction of specific binder-filler systems for carbon and graphite particles; and (2) chemistry of the furfuryl alcohol polymer system. Initial results are reported from a study of lamellar compounds of various materials with graphite. In the materials characterization area, basic understanding of the graphite crystal has been advanced through solid state studies on the magnetic susceptibility and on the electron spin resonance of graphite, both in single and polycrystalline forms. Findings are reported from studies of mechanical properties of graphite in single crystals and whisker form, including tensile behavior, dislocation analysis, and thermal expansion over a wide temperature range. Applied research in this area covered studies on high temperature creep to 3000°C as influenced by composition and fabrication variables. The results are interpreted in terms of a useful mechanical model. Data are presented on elastic moduli, shear strength, and high temperature tensile strength. Development work on fabrication covered a number of studies. The pressure curing technique has been developed and extended to the fabrication of fine grain graphite bodies as large as 30 inch diameter by 30 inch length which were superior in strength to ATJ grade graphite but exhibiting less than one-third as much variation in strength or bulk density. Substrate graphite bodies having a thermal expansion which matches that of silicon carbide oxidation-resistant coatings have been developed and found to impart improved stability and integrity to the coatings. Scale-up to desired sizes is in progress. Bulk graphite bodies, based on fibrous forms of carbon and graphite have been developed and found to provide unique combination of strength, density, and elasticity. High density, recrystallized graphite formed by a hot working process and possessing physical properties intermediate between premium quality commercial graphites and pyrolytic graphite has been scaled up as grade ZTA to sizes as large as 14 inch diameter by 10 inch length. This recrystallized graphite has been fully characterized, and has shown lower erosion rate as nozzle insert material in solid fuel rocket motors. Hot-pressed composites of graphite with refractory metal compounds have resulted in materials with promising resistance to oxidation at high temperatures. Progress in applications technology accompanied the research and development work. A
correlation has been obtained between the erosion rate of the throat of a rocket motor nozzle made from a superior graphite (grade ZTA) and the amounts of oxidants present in the propellant exhaust stream. A program to more clearly define this relationship is recommended. Monolithic graphite nozzle components of a size larger than heretofore used have been satisfactorily fired by other Contractors in static test motors. Preliminary studies have been made of processing, facilities, and time required for scaling up in size of suitable grades for applications requiring graphite in excess of 100 inches in outside diameter. Segmented graphite nozzle components have been studied in small sizes. Scale-up to very large sizes in segmented construction will require additional studies of configuration and joining techniques and grades. High density, recrystallized graphite (grade ZTA) has shown lowest erosion rates as nozzle throats in solid propellant rocket motors. Fibrous base composites are under study for insulation applications where erosion is not critical but where low thermal conductivity, high strength-to-weight ratio, high temperature strength, and compatibility with other carbon or graphite systems are essential.


Melting temperatures are being determined for ZrC and structurally related carbon-deficient members of this phase field. It has been observed that ZrC0.90 develops some liquid phase at approximately 3350°C and is completely molten at 3400°C. ZrC0.85 (nominal), on the other hand, shows no signs of melting at 3390°C. These data indicate that the melting point maximum is not associated with stoichiometric ZrC, but rather with a substantially carbon deficient member. A series of arc melted Ta-C compositions were quenched from 2850°C after prolonged annealing. Microstructures revealed compositional gradients from core to edge, attributable to carbon loss. It was anticipated that the Ta2C and TaC phase boundaries could be delineated by these experiments. At this time it can be stated that the Ta2C/Ta + TaC boundary occurs at less than 29 atomic % carbon, a value which differs from the earlier measurements of 31.5 atomic %. At this same temperature, 2850°C, the TaC/TaC + Ta2C boundary is located between 38 and 43 atomic % carbon. In addition, samples containing 60 atomic % carbon have been melted in the induction unit. Subsequent metallographic studies have verified earlier observations (1, 2, 3) of a eutectic reaction between TaC and C. The flux concentrator, as an efficiency aid in induction heating, is proving to be a very worthwhile tool for high temperature equilibrium studies. TaC crucibles are more effective than graphite in this operation because of reduced vapor pressure. Graphite felt insulation has substantially improved the thermal efficiency of the concentration system. The felt serves the additional important (continued)
function of adsorbing vapors which normally would migrate into the concentrator "donut" and induce arcing.

2-291. THERMAL STRESS TESTS OF PYROCERAM SHELLS.

The thermostructural problems associated with ballistic re-entry vehicles become more serious when an electromagnetic radome is substituted for the re-entry vehicle nose structure, as in a terminally guided vehicle. Materials with suitable dielectric properties have mechanical properties which, in the thermal flight environment associated with a nominal re-entry trajectory, cause severe thermal stresses in the radome. These stresses may exceed the strength of suitable materials. The method of approach therefore, is to protect the radome until exposure is necessary for vehicle guidance. Under these conditions, Pyroceram Code 9606 is considered to be the most suitable radome material. Results of an elasto-plastic stress analysis on such a radome are presented, along with results of tests of Pyroceram 9606 hemispherical shells subjected to typical thermal reentry environments. These tests were performed utilizing the Avco RAD 10-megawatt arc shroud facility.

Significant conclusions: There is sufficient ductility at high temperatures to relieve the high elastic compressive stresses resulting from severe thermal gradients. Although surface crazing accompanying high surface temperatures and stresses may be present, failure results from exceeding the tensile strength at the inner surface. There is little ductility in tension. Once the tensile strength is reached, cracks will propagate rapidly throughout the shell causing failure. Local end effects make a minor contribution to stress level, and are not considered to have induced failure in these tests, since at failure, the shells were intact at the mounting attachment. Meridional temperature gradients effect the stress level markedly wherever there is a temperature discontinuity.
SECTION D - POLYMERIC AND COMPOSITE MATERIALS


A new class of thermally protective materials which have outstanding resistance to dimensional erosion in simulated reentry environments was synthesized. The process for 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. The reinforced carbonized material concept provides a new class of materials for hyperthermal environments. The versatility of the synthesis process for these materials should permit the "tailor making" of specified systems for many uses. Of the substrate and ablative filler systems studied (a) Quartz fabric reinforced carbonized matrix gave the best over-all performance of the matrices evaluated. In addition to having desirable structural characteristics, it also has the best insulation properties and is particularly well suited for applications involving conditions of low heat flux and pressure. (b) Graphite fabric reinforced carbonized substrate plus oxidation resistant additive provides very low recession rates. It should perform well as a re-radiation material at relatively low heat fluxes for long periods of time. (c) Graphite fabric reinforced carbonized matrix plus insulative and burnout additives demonstrate excellent over-all performance, especially at high enthalpy levels. It is best suited for environments where insulation is important. (d) Ammonium chloride provides the best improvement in over-all composite performance of the fillers tested. A semi-empirical equation was derived which relates environmental conditions and backface specimen temperatures. The equation provides values which are in good agreement with experimental data.


Progress is reported on fundamental studies concerning force evaluation of compressed O-rings and thermal expansion phenomena of O-rings at cryogenic (continued)
temperatures. Force and sealability data obtained to date are given. Results for sixteen elastomers are tabulated and graphed. The temperature range covered is 76-300°K. Several observations have been made in the testing to date. If an O-ring leaks, there is very little difference in the leak temperature for the second and third cooldown, and the leak temperature is usually lower for the first cooldown cycle. The lower leak temperature for the first cycle is probably due to the higher force present at the start of the first cooldown. After the soldering procedure and one cooldown cycle, the O-rings tend to come to equilibrium and maintain a fairly constant force for the second and third cycles, hence, the constant leak temperature for the last two cooldown cycles. There appears to be strong correlation between the force on a given O-ring and the temperature at which it leaks. Another observation made during the testing was the relative unimportance of the cooldown pressure. It seems that if an O-ring will hold a seal at 76°K with 100 psig helium pressure, it has an excellent chance of maintaining this seal at 76°K and 500 psig helium pressure. In tests to date, there has been no case where a seal leaked at 500 psig and not at 100 psig. Apparently, O-rings that hold 100 psig helium pressure at 76°K have a good chance of maintaining a seal at 1,000 psig pressure and 76°K, or at least to a temperature near 76°K. It was observed that there is no change in force at the point where the leak occurs, which further enforces the theory that small leak passages may develop, even though there is considerable compressive force on most of the sealing surface. In all tests, it was observed that there was no significant change in the force-temperature curves as the elastomer passes through the glassy state transition temperature. Possibly the high compression, close confinement due to friction, and low final thickness combine to prevent the material from showing any sudden change as it passes through the transition temperature. Thermal expansion data are given for seven elastomers. Preliminary results of a study of volume compression and the glassy state transition in elastomers are given.


A series of arylurea grease thickeners was prepared under widely different conditions. Because of the complexity of the systems studied, the experimental techniques were not the ultimate in scientific precision. However, the resulting extensive data were utilized to determine the important parameters governing particle growth. Available theory was applied to arrive at recommendations to aid in the production of organic, non-soap, grease solids possessing desirable thickener properties. The recommendations are applicable generally to particles which form by nucleation followed by growth due to deposition of product molecules onto particle surfaces. This growth mechanism occurs frequently in practice.

This document presents information on Rocketdyne's efforts in developing glass-filament-wound structural components for use as lighter-weight, lower-cost thrust chamber materials in liquid propellant rocket engines. Extension of the areas of application to include solid-propellant rocket motor nozzles, solid-propellant rocket motor cases, fuel tanks, and pressure vessels are discussed. Prototype structural units are illustrated. Materials and fabrication processes are described and some properties data, quality control information and facilities information are included. This noncontractual report can be classed as a sales engineering document but is not a proposal for any specific project.


Thermal stabilities of polyphenylsilsesquioxane and poly-m-phenoxylene were studied and these polymers were preliminarily evaluated as high temperature surface coatings. Polyphenylsilsesquioxane shows no improvement in thermal stability over conventional high temperature silicone coatings, but it does offer the possibility of a lacquer-type air-drying high temperature coating. Poly-m-phenoxylene has excellent thermal stability but poor color. This polymer can be thermally cured and shows good adhesion and flexibility as a coating after ageing at 700°F for 20 hours.


Investigations on two classes of intermediates, the epoxides and urethanes, as candidate impregnants for rigidizing aerospace structures has been initiated. Included in this work are the evaluation of select commercially available materials as well as synthesis of novel urethane and epoxy type materials for special desirable characteristics. Data describing the curing characteristics and shelf stability with incorporated, deactivated, catalysts of epoxy resins and urethane modified epoxy resins are presented. Data on urethane systems consists of viscosity characteristics at 100% solids content, physical properties of cured impregnants in film form, moisture permeability and curing time profile of select systems. Also included are data on physical properties of laminates prepared from select urethane impregnants and glass fabric. (Author)

The objective of this program was the elucidation of the mechanism of the peroxide induced cross-linking of fluoroelastomers. 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 interaction products. The lowest curing temperature of the vinylidene-fluoride perfluoropropane (VF-PFP) copolymer-benzoyl peroxide magnesium oxide-system was found to be approximately 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.


Model branched polymers have been prepared by the reaction of relatively monodisperse polystyryl lithium with silicon tetrachloride. The branched molecules have been carefully characterized by light scattering and osmotic pressure measurements. 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. Additional studies have been carried out on the effect of aging active anionic polystyrene on the molecular weight of the polymer. In addition, the effect of polymerization temperature was also studied. The three possibilities anticipated were: chain scission, chain branching and monomer-polymer equilibration. 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

(continued)
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. A sharp dependence of the relaxation rate at long times on the degree of crosslinking is observed, similar in form and magnitude for both butyl rubber and natural rubber vulcanizates. The molecular mechanism is not yet clear. Although the process is qualitatively in accord with Bueche's concept of the delayed rearrangement of crosslink positions in the deformed network, serious quantitative discrepancies are revealed. (Author)

2-300. PROPERTIES OF GLASSES AT ELEVATED TEMPERATURES.

The ASD program to obtain useful, statistically sound, design criteria on optically transparent window materials of a brittle nature and suitable for military air vehicle applications, is summarized. Several factors associated with the determination of Young's modulus and the modulus of rupture for seven commercially available glasses are presented and interpreted. The practical strength of plate glass is dependent on several factors including surface finish, thermal conditioning, cutting techniques and composition. Effects of these variables together with long and short time elevated temperature strength capabilities are shown. (Author)


Dimethylacetamide and dimethylsulfoxide have been successfully employed as dry-spinning solvents for poly-2, 2'-[(m-phenylene)-5, 5'-bibenzimidazole. The physical properties of both fibers appear to be comparable. Since dimethylacetamide produces a lighter colored yarn and in general lends itself to easier solvent removal, it emerges as the more desirable solvent for dry-spinning operations. The fibers exhibit remarkably high resistance to hydrolysis and thermal ageing although under the more realistic condition of air ageing the thermal properties are not nearly as impressive as under nitrogen. The N-H bond in polybenzimidazoles is suspect as the weak link (continued)
leading to degradation in the hot oven. To test this hypothesis, N-substituted polymers were prepared via the methylation of N-sodio polybenzimidazoles. Oriented and crystalline polyhydrazide fibers can be converted by a unique cyclodehydration reaction into oriented and crystalline fibers of poly(1, 3, 4-oxadiazoles). Conversion of fibers (T/E/Mi = 5/24/94) of the polyhydrazide derived from equimolar amounts of isophthalic dihydrazide and terephthaloyl chloride (OIOT) gave fibers (T/E/Mi = 2.6/3.1/124) of poly(1, 3-/1, 4-phenylene-2, 5-(1, 3, 4-oxadiazole), (from author's abstract) PODZ-I/T, which have excellent retention of their fiber structure even when exposed to temperatures up to 400°C for prolonged periods of time. Such heat treatments afford a final polyoxadiazole fiber (T/E/Mi = 1.2/1.2/90). The chemical structure of model compounds containing up to 13 alternating rings consisting of 2, 5-(1, 3, 4-oxadiazole), m-, and p-phenylene moieties was found to be stable up to a range of from 440°C to 500°C. This is in agreement with observed stabilities of the corresponding polymer towards thermal degradation. (Author)

The new reagent, hexamethyldisilazyllithium, has been prepared in tetrahydrofuran solution, and its reactions with a variety of chloro compounds have been investigated. The compounds, tristrimethylisilamime and triphenylhexamethyldisilazyl silane, are reported. Other reaction products are unstable and decompose even in the absence of air and moisture. The previously reported reaction of trialkyl phosphites with cyanuric chloride to give 2, 4, 6-tris(dialkylphosphonato)-1, 3, 5-triazine has been investigated and extended to other tervalent phosphorus esters. The new compounds, 2, 4, 6-tris-(ethylphenylphosphinato)-s-triazine and 2, 4, 6-tris-(diphenylphosphinoxy)-s-triazine, are reported. Attempts to prepare partially-substituted chloro derivatives were unsuccessful. Diphenyl phosphine reacts with cyanuric chloride to give 2, 4, 6-tris(diphenylphosphino)-s-triazine, and its reactions are described. Melamine does not react with trimethylchlorosilane in the presence of pyridine. Cyanuric chloride reacts with hexamethyldisilazyl lithium in tetrahydrofuran to give a mixture of products including tristri-methylsilylamine. Cyanuric chloride reacts with diethylaniline to give two compounds believed to be 2-N-ethyl-N-phenylamino-4, 6-dichloro-1, 3, 5-triazine and 2-p-diethylanilino-4, 6-chloro-1, 3, 5-triazine. The reactions of cyanuric chloride with triphenylsilane, and with triphenylsilane in the presence of tertiary bases, are under investigation. (Author)
Cyanuric chloride was reacted with several negatively substituted primary alcohols with fluoro, bromo, nitro, or nitroxy groups as electronegative substituents. Completely substituted reaction products were obtained with trifluoroethanol, tribromoethanol, pentaerythritol trinitrate, and pentaerythritol dinitrate. Di- and tri-substituted products were obtained with 2-methyl-2-nitro-1-propanol which has a tertiary nitro group, whereas alcohols with primary and secondary nitro groups did not react. The reaction with the potassium salt of nitroform gave a product which gradually decomposed on standing. The monoperchlorate salt of trihydrazino-s-triazine was obtained, but attempts at the preparation of perchlorate derivatives of aminoazido-s-triazines were unsuccessful. Sensitivity and stability data are reported on some of the products. (Author)

This preliminary report presents the author's initial concepts in an investigation of thermal stability problems of organic polymers and inorganic polymers. The discussion seeks to establish a useful definition of thermal stability as applied to some physical or mechanical property and then to relate stability to the established concepts of chemical bonds and bond scission. Alternate approaches are described briefly.
SECTION E - ELECTRIC, ELECTRONIC, AND MAGNETIC MATERIALS


The objectives of this project are to study the physical and electrical properties of thin films. These studies may yield useful new devices and also furnish new insight into the basic nature of extremely thin layers of material. Some of the new devices utilizing thin films which have recently been discovered are the metal-interface amplifier, thin film solar cells, and various negative resistance devices. These devices are based on the utilization of hot electron emission or quantum-mechanical tunneling through thin metallic and dielectric films. The use of thin films in active devices may provide many advantages over the use of semiconductors. Some of the more important of these are increased resistance to high energy radiation and reduced temperature sensitivity. One phenomenon currently being studied is that of electron tunneling between metallic films through thin dielectric layers. Initial phases of this program have been concerned with the study of multi-layered tunneling configurations involving thin superconducting, ferromagnetic and dielectric films at low temperatures. This report presents research experiments in methods of forming structures suitable for the observation of tunneling phenomena. Metallic films of tin, indium, aluminum, gold, permalloy, lead, and silicon monoxide have been successfully deposited for several experiments. Dielectric films of aluminum oxide and barium stearate have been successfully fabricated.


The design of electronic equipment for operation at cryogenic temperatures (below 10°K) is rapidly becoming feasible from an engineering standpoint. This is due primarily to advances in storage of cryogenic fluids such as liquid helium, and to advances in design of small, closed-cycle, cryogenic refrigerators capable of maintaining temperatures in the range of 1° to 2°K. Also, some electronic functions can be performed only at liquid helium temperatures. For example, very high magnetic fields have been obtained in small and relatively cheap equipment. Some devices have become practical. The cryotron is a good example; this computer device is competitive with magnetic thin films and is superior in some respects. Research has consisted of the calculations and theoretical studies necessary in planning (continued)
2-306. (Continued)

experiments, and of the initial experiments on high field superconducting alloys. Two high-field superconducting solenoids have been designed and tested. Fields of 24 kilogauss were obtained in the last design in a coil with a working inner diameter of 0.5 inch. Instrumentation for two experiments, one to study the critical current in thin film rings, and the other to determine the effects of different types of defects, dislocations, and strains on high-field superconducting materials, is being assembled.

2-307. LOW-TEMPERATURE CONDENSATION PHENOMENA.
No contract no. 10 refs. A62-9989.

The broad objectives of this program are to study the character and origin of the low energy states of condensed Fermi systems. Electron tunneling between superconductors separated by very thin dielectric layers has become a powerful tool in the investigation of the properties of superconductors. Examples of the types of information obtained are a quantitative measurement of the magnitudes of the energy gaps and densities of states of the metals involved. Preliminary experiments were conducted with aluminum-lead strips on glass, tunneling curves are presented. Later experiments will investigate tunneling characteristics of ferromagnetic-superconductor strips. The design of the He\textsuperscript{3} refrigerator under construction is presented. This refrigerator will permit experiments and tests to be carried out at temperatures lower than 1 K. Initial calculations have been made and equipment has been assembled for investigation of the effects of microwave phonons on the properties of superconductors.

2-308. RESULTS OF SUPERCONDUCTING MAGNET SURVEY.

This report presents the results of a survey of some 1800 people in the United States known to be interested in superconducting magnets. 1297 responses were received, tabulated and analyzed. The authors conclude that there is much interest in superconducting magnets; the interest and needs are far ahead of current technology. Within the next year or two, most superconducting magnets will probably be made using NbZr wire. As NbSn technology advances, it will be possible to obtain even higher fields than the 50,000-60,000 gauss limits attainable with NbZr. It is anticipated that larger sizes of superconducting magnets will be built, and will be applied in plasma containment, bubble chambers, accelerators, MHD, and etc. Many laboratories are currently working on magnets with coil ID 5-10 inches. Although most plan to conduct their experiments in liquid helium, many will

(continued)
want the experimental field at room temperature. Some special applications will require high-temperature working volumes. Closed cycle refrigeration equipment will be necessary for most of the experimental work. Helium requirements will increase substantially as the use of superconducting magnets increases.


Thermoelectric instabilities of noble metal thermocouples and individual thermoelements (platinum, rhodium, iridium, iridium-50% rhodium, and several platinum-rhodium alloys) have been studied from 1000°C to 1700°C under oxidizing and neutral atmospheres. The principal source of instability was found to be a contamination of the thermoelements by impurities from ceramic protection tubes. Several grades of alumina sheathing were tested, and thermocouple errors associated with each grade were determined. With alumina sheathing the principal contaminant was iron, and the instability generated was at least an order of magnitude greater in a neutral atmosphere than in an oxidizing atmosphere. Increasing the wire size increases stability in a neutral atmosphere, although not in an oxidizing atmosphere. Instability increases rapidly with temperature between 1000°C and 1700°C, but the order of decreasing stability remains: Pt-6%Rh/Pt-30%Rh; Pt-1%Rh/Pt-13%Rh; and Pt/Pt-10%Rh, or Pt/Pt-13%Rh. The instability of pure rhodium thermoelements, and of Ir and Ir-50% Rh thermoelements, depends on internal changes, and not as much on iron contamination as the Pt-Rh series of thermoelements; hence the pure rhodium and the iridium thermoelements become preferable if gross iron contamination is expected. When compositional changes are expected in a thermoelement, immersion depth should be maintained constant or increased.
SECTION F - FLUIDS AND LUBRICANTS


The objective of this contract was to develop fire-resistant fluids suitable for use in aircraft hydraulic systems over the temperature ranges of -65°F to above 450°F, and 40°F to above 700°F. Apparatus to measure pour point, viscosity from -65°F 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 halocarbons, phosphine oxides, aromatic and heterocyclic were synthesized and/or purified for the above physical measurements. Correlations of these data with structure were made. (Author)


Experimental research has been carried out to study deterioration in JP-6 jet fuel thermal stability performance during storage and to develop a simple bench scale test procedure for measuring jet fuel thermal stability. JP-6 type fuels were shown to deteriorate seriously in thermal stability performance following storage for 52 weeks or more at ambient temperatures. Storage under a sealed nitrogen atmosphere in the absence of solids or water contamination showed little ability to offset such deterioration. However there was evidence that large concentrations of certain phenol type antioxidants might retard fuel deterioration in this respect, as might tighter fuel composition limits on aromatic (aromatic-olefin) hydrocarbons and possibly sulfur content. Potential deleterious effects were shown by trace copper contamination in JP-6 fuels, though not at concentrations at the low levels found by analysis in typical JP-6 fuel samples. Fuel sulfur contamination appeared to contribute to fuel thermal instability performance, but showed up adversely in storage performance only in the presence of an aromatic-olefin fuel constituent. Test fuels which deteriorated in performance during storage usually showed decreased ability to transmit UV light as (continued)
well, though the converse was not always true. A small scale thermal
stability test procedure based on losses in UV light transmission following
heating has shown promising ability to differentiate thermal stability
performance of JP-6 and poorer grades of jet fuel in the same order as
larger scale test procedures. However test results with special high-
temperature-fuels blended for 500°F-plus stability performance were less
promising. It is recommended that development work be continued on this
small scale test procedure. (Author)
SECTION G - FABRICATION TECHNIQUES AND PROCESSES


The purpose of this project is to develop new or improved manufacturing methods for large scale production of ferroelectric or piezoelectric ceramic materials such as titanates, zirconates, niobates, and tantalates. A major requirement is high purity (99.95 percent). The accomplishments during this third period of Phase II have been the installation of pilot plant equipment, initial start-up procedures and chemicals for reaction, chemical analysis and x-ray techniques, and the investigation of a compatible barium zirconate technique. The synthesis of BaTiO₃ in the pilot plant will be accomplished by the addition of triethanolamine titanate to a potassium hydroxide/barium chloride solution. Chelated esters of zirconium compounds are under investigation.


The purpose of this project is to develop new or improved manufacturing methods for large scale production of ferroelectric or piezoelectric ceramic materials such as titanates, zirconates, niobates, and tantalates. A major requirement is high purity (99.95%). The accomplishments during the fourth period of Phase II have been the operation of a pilot plant, the chemical and x-ray analyses of its product and the ceramic processing of the powders. The synthesis of BaTiO₃ in the pilot plant has proved to be a successful scale-up of the bench scale runs. Product analysis shows the synthesis to be within allowable impurity tolerances and stoichiometrically within analytical detection limits.
The objectives of this program are to acquire the techniques for sealing high speed rotating shafts operating in environments of high temperature liquid metals and vapors, and the near-vacuum environment of space, and to provide long seal life. Some of the requirements specified in the contract are: (1) The fluid to be sealed shall be potassium. (2) The seals shall be operative at fluid temperatures from the melting point of the fluid selected to 1400°F. (3) The seal, or seal combinations, shall be designed for 10,000 hours of maintenance-free life. (4) The working fluid, potassium, shall be used as the seal lubricant. (5) The seal, or seal combinations, shall be capable of maintaining zero leakage (in the technical sense) under all conditions of operation. (6) The seals shall be capable of operating in a zero "g" environment. The 20,000 rpm water seal test rig was designed, manufactured, and setup prior to the beginning date of this program. In this first reporting quarter, instrumentation and the test rig were checked out and first data were obtained. The data showed good agreement with previous calculations and no cavitation was experienced. Analytical investigation of screw seals has been started. The analysis is encouraging insofar as it indicates that turbulence (which will exist at the speeds considered) is likely to improve the pressure generating ability of the screw seal. Experimental results reported in the literature tend to confirm this prediction. There are presently three configurations of the stationary disk-rotating housing seal on hand at Evendale for testing in water. Design layouts have been completed on various configurations of the stationary housing-rotating disk and the squeeze seal. Layouts are presently being detailed for hardware, to be tested in September, 1962. Design work is in progress on the high speed test rig to be used for liquid metal testing and its auxiliaries required for liquid metal operation. Both should reach the procurement stage by the middle of September, 1962.

A variety of substrate materials was developed to substitute for heavy metal alloy. These materials included molybdenum TZM alloy, Zircaloy-2, and molybdenum-modified heavy metal. Coated lathe tools prepared on these substrates yielded four to eight times the life of solid carbide tools in turning 52 Rc steel at speeds to 250 rpm. Heavy metal alloy, unless adequately supported, is not a satisfactory substrate for carbide-coated rotary tools since it deforms during hot-pressing. With a change from the deformable heavy metal tap substrate to a non-deformable TZM tap (continued)
substrate the hardness of the carbide coating increased from 950 kg/mm\(^2\) to 1750 kg/mm\(^2\), and the porosity of the coating decreased almost to zero. Preliminary studies indicate that the following techniques are also promising for eliminating or minimizing deformation: ultrasonically broached molds, compression molded die inserts, isostatic hot-pressing, and the use of vibratory compaction techniques. A literature survey was made of the potential use of ultrasonic techniques in the coated tool program. Ultrasonic compaction and ultrasonic hot-pressing appear promising as means of improving the density of tool support materials and of the carbide coating. Application of these techniques will require close control of particle size distribution of the carbide dispersons. (Author)


A semi-continuous technique to hot-press beryllium oxide parts at production rates that can compete with those of sintering methods, and using much lower temperatures, has resulted from studies showing that, at a given maximum temperature and pressure, the end density is essentially independent of previous temperatures and pressures. By repressing a densified powder compact at a higher temperature or pressure, the sample becomes as dense as a ceramic sample pressed initially at that temperature and pressure. For this reason, a hot-pressing scheme was devised in which the ceramic samples themselves transmitted part of the pressure, permitting loaded dies to be cycled through the hot press.
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