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METALLURGY AND CERAMICS

SUMMARIES OF PHYSICAL RESEARCH IN METALLURGY, SOLID STATE PHYSICS, AND CERAMICS

Edited by
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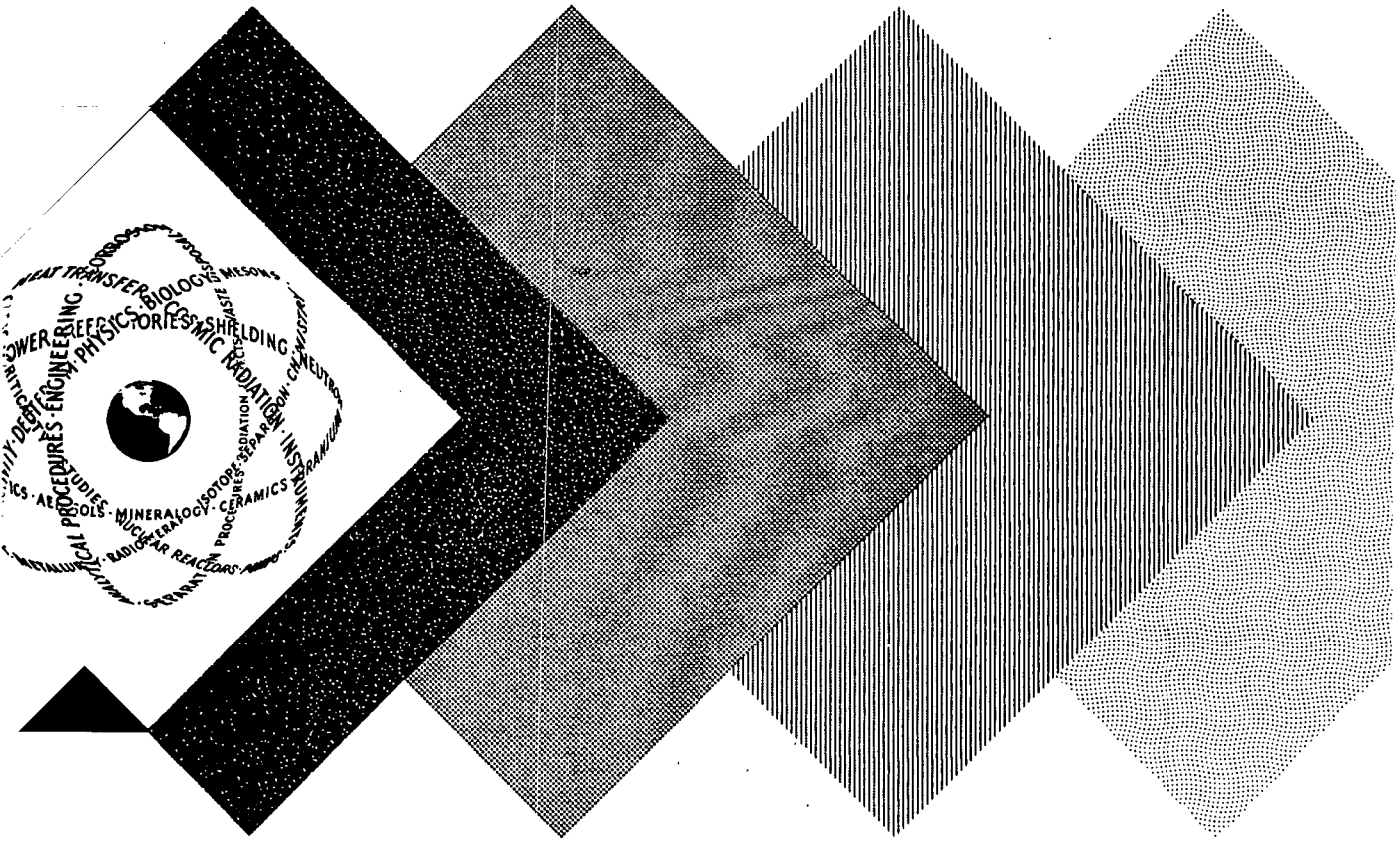

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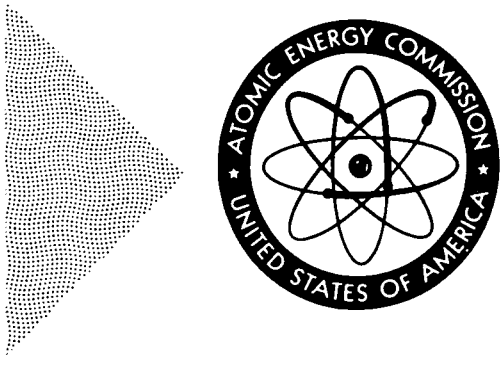

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MEMORANDUM TO: Recipients of TID-4005 (Pt. 1, 5th Edition)

FROM : Robert L. Shannon, Chief
Technical Information Service Extension

SUBJECT : CORRECTED INDEXES FOR TID-4005 (PT. 1, 5TH EDITION) SUMMARIES OF PHYSICAL RESEARCH IN METALLURGY, SOLID STATE PHYSICS, AND CERAMICS

The July, 1959 issuance of the subject publication contained a "Contractor Index" and a "Subject Index" with section rather than page references for the individual entries.

This memorandum transmits new versions of the indexes, using page references, which are intended to be inserted in those copies of TID-4005 (Pt. 1, 5th Edition) which were originally distributed.

Enclosure:
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FOREWORD

In addition to conducting scientific research in its own laboratories, the Atomic Energy Commission provides assistance to universities, research institutes, independent laboratories, industrial laboratories, and other government agencies who can contribute to the research program of the Commission. Such assistance takes the form of financial support for payment of salaries, purchase of equipment and other materials, and allowance for overhead costs and is formalized by direct contract between the Commission and the organization performing the work. Both basic and applied research studied are involved.

Under these contractual arrangements, physical research is being performed in metallurgy, chemistry, and physics. For each of the fields of science, summaries of the physical research projects under way at the various sites are published from time to time as directories. These directories serve to assist researchers in exchanging ideas and to acquaint them with related programs at other laboratories.

This document summarizes the objectives and current status of the individual research projects supported by the Metallurgy and Materials Branch, Division of Research, in the fields of physical metallurgy, solid state physics and physical ceramics. It includes the research conducted at the Commission's laboratories as well as that performed at university and other laboratories. These summaries were prepared primarily by the investigators whose names appear on the summary sheets.

The information has been organized into three broad categories:

I. Production Treatment and Properties of Materials
II. Physical Research in the Nature of Matter
III. Interaction of Radiation with Matter

Two indexes appear at the end of the directory, one arranged by subject and another arranged alphabetically by contractor.

All of the research presented in this document is unclassified. As a normal procedure, periodic progress reports of information resulting from these contracts are not prepared. Investigators are urged to report their results in the open literature at the earliest opportunity for wide dissemination of the information.

Section I

PRODUCTION, TREATMENT, AND PROPERTIES OF MATERIALS

I-A

METALLURGY OF REACTOR MATERIALS

Contractor: Ames Laboratory, Iowa State College, Ames, Iowa

Contract: W-7405-eng-82

Brief Title: METALLURGICAL AND OTHER INVESTIGATIONS ON SPECIAL METALS, OTHER MATERIALS AND PROCESSES

Investigators: G. Burnet, O. N. Carlson, P. Chiotti, A. H. Daane, R. W. Fisher, W. Larsen, R. E. McCarley, G. Murphy, E. Olson, D. T. Peterson, J. Powell, R. E. Rundle, J. F. Smith, M. Smutz, F. H. Spedding, A. F. Voigt, D. Wilder, and H. A. Wilhelm

Scope of Work

Much of the work in this area at the Ames Laboratory has some direct bearing on the search for special property materials and special processes of interest to the broad program of the U.S.A.E.C. The preparation and investigation of metals and alloys constitute a major part of these studies at Ames. Studies in ceramics, engineering processing, corrosion and mechanical testing and evaluation are associated with various phases of the metallurgy program.

Scope I - Extraction and Purification Studies

Many of the metals of special interest to the atomic energy program are not commercially available in satisfactory quality and/or quantity. Part of the program is, therefore, directed toward the extraction, separation and preparation of purified compounds of such metals from their ores with subsequent reduction to the metallic state.

Methods of opening such minerals as columbite, pyrochlor, tantalite, zircon, gadolinite, xenotime, monazite, fergusonite and bastnasite have been developed. Closely associated with this type of work are investigations of liquid-liquid extraction and ion-exchange techniques as they apply to the isolation of the elements of interest from the minerals.

Many of the less common metals, such as the rare earths, yttrium, scandium, columbium, thorium, vanadium and uranium have been prepared by new methods. Furthermore, decided improvements have been made in earlier methods of preparing some metals. In a number of cases improved quality and a lower cost have been realized from this effort. Work is continuing on various metals in an effort to reduce the cost of their preparation in satisfactory quality for use in the atomic energy program.

As a result of the increased availability of oxides of a number of less common metals, work in the field of ceramics is being extended to include new bodies and systems of interest in the field of atomic energy.

Scope II - Phase Diagrams

The general objectives of the laboratory in the study of alloy systems are to establish the equilibrium diagrams of systems, and to accumulate a body of

data which may serve as a basis for a better understanding of the nature of metals and alloys and the theory of the alloying behavior of metals.

The following systems are being investigated:

(1) the binary systems of yttrium with Ti, Cr, Fe, Co, Ni, Cu, Mg, Al, Sn, Nb, Th, La and Gd.

(2) the binary systems of lanthanum with Gd, B and Nd.

(3) the binary systems of thorium with H, Zr, Ta, V, Zn and Mg.

(4) the systems Al-Nb, Al-Ta, Cr-V, U-Hf, Ni-Zr, Hf-Ni, Re-U, Co-Zr, Ba-H and Ba-$CaCl_2$.

(5) the distribution of Ag between liquid Pb and Zn.

(6) the distribution of protactinium as well as fission products between U/Mg-Th and Mg-Th/fused salt phases are being investigated.

Scope III - Structure and Properties of Metals, Alloys and Intermediate Phases

Fundamental studies in the field of solid state metallurgy are in progress and include determinations of crystal structure and measurements of physical properties such as Hall coefficients, magnetic susceptibilities, thermal expantions, electrical and thermal conductivities, thermodynamic functions, elastic constants, other mechanical properties, and optical properties. These measurements provide information essential to the development of a better understanding of the solid state theory pertaining to metals.

Materials under investigation include elemental metals, alloys and intermetallic compounds of uranium, thorium, zirconium, the rare earth metals, and various other less common metals. The allotropy of the rare earth metals and of calcium have been found to be highly sensitive to purity. Layering of the crystallographic planes of the close-packed structures of these elements has been found to exhibit interesting variations. Somewhat analogous behavior has been observed in the layering of intermetallic compounds of uranium with zinc and of thorium with magnesium.

Correlations between crystal structure and physical properties are being sought in various ways. Elasticity of materials with the same and with related crystal structures are being compared. The elastic constants of single crystals of thorium have been determined, and measurements on single crystals of beryllium and yttrium are in progress. Anisotropic behavior of electronic properties are being studied in single crystals of non-cubic structures. Thermodynamic functions and equations of state are being determined from measurements of heat capacities, vapor pressure, compressibilities, and thermal coefficients of expansion.

Scope IV - Preparation of Pure Metals

The need for metals of special high purity for use in solid state investigations and alloy studies is constantly increasing. It is becoming increasingly

evident that many of the properties of a metal are sensitive to the presence of minute amounts of impurities and that the stability of many intermediate alloy phases is affected by trace elements.

Several methods for preparing metals of very high purity are being used in the various investigations underway in the Ames Laboratory. One such method is the preparation of high purity halides and oxides for use in subsequent reduction to the metal, a technique being applied to Th, U, Ba, V and several of the rare earth metals. A vacuum redistillation technique is being used to purify Ca, Ba, Li and the more volatile rare earth metals. Iodide crystal bar refining procedures have been applied successfully to Th and V so that small amounts of these metals are now available in high purity. Work is currently underway on the purification of Cb by the crystal bar process. Investigation of pyrometallurgical extraction techniques for purifying thorium is planned. The interaction of molten metals with crucible materials is being studied for many of these metals.

Scope V - Pyrometallurgical Research

The research in pyrometallurgy is concerned with the use of metallurgical techniques, for separation of the various components present in irradiated fuels and with the development and testing of materials to contain molten fuels at high temperatures.

The separation methods being studied most thoroughly are liquid metal extraction processes. Molten silver extraction of uranium has received the greatest attention. The success with molten silver in extracting 95% of the plutonium from U-Pu alloys has led to a detailed study of the behavior of plutonium and the fission products in the U-Ag system. Rare earth metals are being studied as extractants for removing fission products from uranium fuels; they remove much less plutonium from uranium than does silver.

Possible applications of pyrometallurgical techniques to the Th-U^{233} systems are being studied, in particular the use of Mg-Th alloys. Uranium is quite insoluble in these alloys; its solubility has been determined and the application of this behavior to a method of separating U^{233} and its parent Pa^{233} from Th are under investigation. Other metallurgical techniques involving selective oxidation and reduction with salts and Zn-Mg alloys have shown promise and are being investigated.

Static, dynamic and loop tests are in progress to study corrosion in various systems. Molten alloys such as Bi-U and Mg-Th have been circulated successfully in loops of tantalum metal for many hours at 1000°C and further testing is in progress. Yttrium has performed very well in small scale experiments and tubing is now on hand to be tested in hot loops against the U-Cr eutectic and other fuel alloys. Preparations are being made for in-pile loop tests.

Alloys which may find application in fuel systems, e.g. Th-Mg and Th-Zn, and as containers, e.g. Y-Ti, Y-Nb, Y-Mg, are receiving study from the points of view of their phase diagrams and their application to reactor problems.

Contractor: Anderson Physical Laboratory, Champaign, Illinois

Contract: AT(11-1)-544

Brief Title: PURIFICATION OF ALKALI HALIDES

Investigators: Scott Anderson

Scope of Work

The primary objectives of this research are: (1) to purify alkali halides to a factor of ten greater purity than is now readily available, (2) to develop adequate procedures for the quantitative chemical analysis of these salts and (3) to produce large single crystals of halides as free of imperfections as possible.

It has been decided to use the technique of zone refining as the initial means of purification. Zone refiners have been constructed and operated. Experimental difficulties have prevented the attainment of the maxium purification of the method. The best result obtained so far was an improvement of a factor of three.

The cathode layer technique of spectrochemical analysis has proved to be sensitive enough for the analysis of some metal impurities in these salts. Work is now in progress toward the quantitative calibration of the method. When this is complete, it will be determined whether or not this method will be entirely satisfactory. If not, then it is planned to investigate the efficacy of a hollow cathode source.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: PHYSICAL METALLURGY OF URANIUM

Investigators: L. Lloyd, E. S. Fisher, S. J. Rothman, M. Mueller and W. Yario

Scope of Work

The physical metallurgical studies of uranium are designated to accumulate fundamental data as well as to furnish information useful to development and production work in fuel element materials. The low temperature orthorhombic allotropic modification (alpha) is of particular interest from the viewpoint of fundamental studies because of its low lattice symmetry. The programs currently being investigated are briefly discussed below:

Scope I - Self-Diffusion in Uranium

The major objective of this program is to determine the dependence of the mobility in the uranium lattice on crystallographic direction. The technique is to observe the penetration of a layer of U^{235} which has been sputtered onto a sample of depleted uranium. A thin film of gaseous contamination is believed to be responsible for lack of penetration noted so far, and present efforts are directed toward improving the experimental conditions of producing the layer.

Scope II - Elastic Constants of Alpha Uranium Single Crystals

In cooperation with Bell Laboratories, six normal and three shear elastic constants have been measured for alpha uranium at 25°C. These nine constants completely describe the room temperature elastic behavior of this material. Arrangements are in progress to extend these measurements both above and below 25°C ambient. The room temperature data have been accepted for publication in the Journal of Applied Physics.

Scope III - Thermal Expansion Coefficients of Alpha Uranium Single Crystals

Measurements of thermal expansion of alpha uranium single crystals near room temperature as a function of crystallographic direction have indicated discrepancies with x-ray data, probably because of internal stressing in the latter. Plans are to extend the work to lower temperatures.

Scope IV - Plastic Deformation of Alpha Uranium Single Crystals

A systematic investigation of the plastic deformation mechanisms operative in alpha uranium single crystals upon compression at room temperature and as a function of crystallographic direction has recently been completed. This work has now been extended to include investigation of the deformation mechanisms occurring in alpha

uranium at elevated and subzero temperatures.

Four operative slip mechanisms have been identified in compressive loading at 600°C. It has been determined that the critical resolved shear stress on the (001) planes is an order of magnitude smaller than on the (010) planes, which may account for the observance of (001) texture in rolled plates at high temperatures. The work is being extended to include intermediate temperatures and below room temperature.

Scope V - Recrystallization, Recovery and Grain Growth of High Purity

Dilatometric studies have now shown that heavily cold rolled high purity sheet can be recrystallized without appreciable texture at as low as 320°C. Subsequent annealing at 400°C causes a new recrystallization which does involve texture change. This indicates the existence of two separate mechanisms of recrystallization. The assistance of x-ray data will be used to explain the nature of these two mechanisms.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: URANIUM ALLOYS

Investigators: A. E. Dwight, S. T. Zegler, J. Darby and M. V. Nevitt

Scope of Work

The primary effort in the area of uranium alloy research is directed to the determination of uranium constitution diagrams for which published data are still lacking and to those which require further work to resolve conflicting published versions. Specific objectives of this program can be summarized as follows:

Binaries with carbon: liquid-solid equilibrium relations at low carbon concentration.

Binaries with zirconium: solid phase relations from 600°C - 800°C for alloys containing up to 32 w/o zirconium.

Binaries with niobium: uranium-rich portion of the diagram below the solidus.

Zirconium-niobium ternaries: phase relationships below the solidus for the uranium-rich corner of the ternary diagram.

Ruthenium, rhodium, and palladium binaries, and ruthenium-molybdenum ternaries: entire diagram below the solidus.

For the U-Nb and U-Zr-Nb systems, the determination of constitution diagrams is being supplemented by a study of the kinetics of solid phase transformations.

Phase diagrams of the U-C, U-Zr and U-Zr-Nb systems havebeen completed. Current work concerns itself with uranium base alloy systems with the elements constituting the "fissium" spectrum, e.g., Ru, Pd, Rh, Tc and Mo. These data are intended to fill notable gaps or to resolve conflicts in existing information. A program is being initiated to study the thermodynamic properties of selected intermetallic uranium compounds. Work on sub-critical transformation kinetics is continued.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: PHYSICAL METALLURGY OF PLUTONIUM AND ITS ALLOYS

Investigators: L. Kelman, B. Blumenthal, C. Biver, and E. Benson

Scope of Work

A major objective of this program on the physical metallurgy on Pu and its alloys is to develop a reproducible source of plutonium of sufficiently high purity that individual impurities do not influence significantly the physical constants and transformation kinetics of the base material. A high temperature salt electrolysis apparatus has been completed, and runs are in progress. A liquation furnace has been constructed and is being tested. The metal will be evaluated by metallographic, chemical and density techniques. Analyses for hydrogen, carbon, oxygen and americium have been developed by the ANL Chemistry Division. Spectro-chemical methods compare favorably with those for uranium, except for iron and silicon, for which special techniques are being developed.

The alloy systems under investigation include U-Pu, Zr-Pu, and systems based on Pu and the fissium elements.

Contractor: Armour Research Foundation, Chicago, Illinois

Contract: AT(11-1)-515

Brief Title: NIOBIUM PHASE DIAGRAMS

Investigators: D. J. McPherson

Scope of Work

An important aspect of the development of refractory alloys is the effect of the interstitial elements, oxygen, carbon, nitrogen, and hydrogen on the mechanical properties. Determination of the niobium-interstitial binary phase equilibria is in progress.

At the present time, two phase diagrams are under investigation, niobium-oxygen and niobium-carbon. The scope of investigation is a complete determination of the composition-temperature dependent equilibrium under one atmosphere pressure up to the compound richest in the interstitial component. Particular emphasis is being placed on the terminal solubility limit of the interstitial element in niobium as this is probably the most important factor affecting mechanical properties. Three-phase equilibria whereby the interstitial compounds are generated or whereby the alloys freeze are likewise being determined.

The form of the niobium-oxygen system has been established and will be completed by the end of the present contract period. Work to date indicated that the niobium-carbon system is less complex than the niobium-oxygen system. A proposal has been presented for continuation of these investigations with the determination of the niboium-nitrogen and niobium-hydrogen systems in like detail.

Contractor: Armour Research Foundation, Chicago, Illinois

Contract: AT(11-1)-315

Brief Title: SYSTEM ZIRCONIUM-IRON-TIN

Investigators: L. E. Tanner and D. W. Levinson

Scope of Work

The purpose of this investigation is to establish equilibrium phase relations in the system zirconium-iron-tin over the composition range bounded by pure zirconium, Zr_4Sn and $ZrFe_2$ at temperatures between 1100°C and as low as possible. Of particular interest are the terminal solubility boundary and phases in equilibrium with the terminal solution.

Additional emphasis will be placed on the following features of the phase equilibria:

(1) Determination of the structure of Zr_4Sn and investigation of possible allotrophy

(2) Delineation of the compositional extent of the ternary phase based on Zr_4Sn

(3) Delineation of the compositional extent of $ZrFe_2$ phase

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: URANIUM PHASE EQUILIBRIA STUDIES

Investigators: A. A. Bauer

Scope of Work

Study of the constitution of uranium-zirconium-molybdenum alloys was completed. On the basis of these studies, a ternary cut between uranium-74 a/o zirconium and uranium-31.5 a/o molybdenum was determined and phase relationships in the ternary system were inferred. Alloys were studied to temperatures of 900 C by metallographic, thermal analysis, and x-ray diffraction techniques. The composition of a high-zirconium ternary eutectoid was approximated.

The solid-state portion of the uranium corner of the uranium-zirconium-silicon system has been completed. Isotherms to 950 C have been determined. Zirconium-silicides exist in equilibrium with uranium-zirconium alloy phases over most of the regions studied. Metallographic, thermal analysis, and x-ray diffraction techniques were employed in this study.

An investigation of the uranium-niobium alloy system is in progress to evaluate the effects of oxygen and nitrogen on the major features of the system.

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: BONDING FUNDAMENTALS

Investigators: F. Holden

Scope of Work

An investigation has been concluded to study on a microscopic scale the process of bonding of metals in which single asperity bonding is simulated. A two-stage mechanism is proposed for the massive solid-state bonding of metals. Initially, contact is made between asperities on the two mating surfaces. The application of pressure increases the number of asperities in contact, and the total area of the asperities is increased by deformation. Following initial contact, growth of the bond occurs. The total bonded areas for a given set of conditions may be expressed as the sum of these two areas: the initial bonded area and the area formed by growth.

A mathematical analysis of experimental results obtained using gold needles bonded to gold surfaces showed that the initial contact area was dependent on the size of the apserity, the temperature, and the pressure used. The growth function was found to be related to both volume and grain boundary diffusion. Initially, grain boundary diffusion was dominant. As the bond grew, the angle between the asperity and the flat surface became greater, grain boundary diffusion decreased, and volume diffusion dominated. An empirical expression relating bond growth to diffusion data was developed.

Contractor: Brookhaven National Laboratory, Associated Universities, Inc., Upton, Long Island, New York

Contract: AT(30-2)-Gen-16

Brief Title: BASIC STUDIES RELEVANT TO THE LIQUID METAL FUEL REACTOR

Investigators: D. H. Gurinsky, J. R. Weeks, J. S. Bryner, M. B. Brodsky, and R. A. Meyer

Scope of Work

Scope I - Solid-Liquid Reactions and Kinetics of Oxidation of U-Bi Alloys

This work has as its objective an explanation of the corrosion behavior of materials in liquid bismuth alloys and the reaction rates of U-Bi liquid alloys with air, oxygen and nitrogen.

Since it has been shown that corrosion inhibition by Zr and Mg additions to Bi is due to the formation of ZrC and ZrN films on steels, a study of the film formation as a function of composition and heat treatment of the steel, the liquid Bi alloy and the temperature is in progress. The above work is being expanded to include a more detailed study of the films formed. Films stripped from the steels by treatment with brome-methanol solution will be analyzed quantitatively for components and will be examined by x-ray and electron diffraction techniques and by electron microscopy. Zirconium tracer absorption experiments to determine continuity and coverage of the steels by the films is also continuing.

A study of the kinetics of oxidation of liquid Bi alloys has been started recently. In the initial work, air was bubbled through molten alloys (Bi containing varying amounts of U, Mg, Zr, Ca) and the rates of oxidation of individual components were determined by frequent analyses of the liquid. Interesting results have been obtained and should be useful in determining the activity coefficients of solutes in Bi.

Scope II - Solid-Liquid Equilibria

The purpose of this work is to determine the equilibria in systems pertinent to a bismuth liquid metal fuel reactor.

The solubilities of the components in steel, including Fe, Cr, Ni, Mn, and the solubilities of the structural materials, Mo and Nb, in liquid bismuth have been determined. Solubilities of the fission products Sr, Ba, Ce, Nd, La, Pd, Rh, Ru, Te and Y in liquid bismuth have been determined. Since Zr, Ti and Mg are useful additions to a U-Bi fuel alloy, the solubility of these elements as well as their effect on the solubility of Fe and Cr in Bi have also been determined.

The liquidus of the Bi corner of the U-Bi-Zr, and U-Bi-Mg-Zr systems is presently under investigation and phase diagrams of the Th-Bi and Th-Bi-Pb systems are nearing completion. The behavior of a dispersion of the intermetallic compound $ThBi_2$ in Bi with respect to crystal habit growth on thermal cycling is under investigation. The effect of third elements, such as Te, on crystal growth and the phase equilibria is being determined.

A study of the wetting of thorium compounds by Bi has been started. In this work it is planned to determine optimum conditions of surface preparation, solid composition, and liquid composition required to obtain good wetting of these compounds. Sessile drop experiments are planned to determine wetting angles as influenced by the above factors. An attempt will be made to explain the role of wetting on the surface chemistry of the dispersed thorium compounds.

Scope III - Effect of Radiation on Materials

This program is concerned with the effect of radiation on the physical properties of graphite, and structural materials, on the reactions of graphite with air and oxygen and on the behavior of structural materials in Bi fuel.

The effect of neutron and gamma-irradiation on the oxidation of graphite by oxygen and air has been studied over a range of temperatures and as a function of prior history of the graphite. Future oxidation experiments will be directed toward obtaining an understanding of the observed effects. Ignition temperatures for various grades of graphite as a function of their history and conditions of oxidation will also be continued.

The effect of radiation on the properties of AGOT and AGHT graphites has been determined in the BNL reactor as a function of reactor operating conditions and position in the reactor. These experiments will be continued in an effort to obtain a better understanding of these effects produced and with a view of reducing the radiation induced effects in the BNL reactor, i.e., such as frequent annealings, etc. This program has been expanded to include the low permeability graphites which are presently available. The effects of fission products bombardment on the properties of the more impervious grades of graphite are included. Stored energy, modulus of elasticity, compressive strength, dimensional changes, lattice parameter changes and thermal conductivity have been measured as a function of temperature of irradiation, environment, and exposure.

Another aspect of the radiation effects program is concerned with the effects produced in the properties of the low chrome steels as a result of neutron and fission product bombardment. Since the latter condtion may have some effect on the corrosion behavior in a U-Bi fuel, static as well as dynamic corrosion tests are under way. Methods are being developed for studying the effect of fission product bombardment on the ZrC-ZrN surface films formed on steels.

Contractor: Case Institute of Technology, Cleveland, Ohio

Contract: AT(11-1)-588

Brief Title: TRANSFORMATION CHARACTERISTICS OF ZIRCONIUM-NIOBIUM ALLOYS

Investigators: Robert F. Hehemann

Scope of Work

The mechanism, kinetics and interrelationships of phase transformations in zirconium-niobium alloys are being studied. The present investigation will study the transformations occuring both on isothermal transformation of beta and on aging of retained beta in alloys containing 5%, 12%, 17.5% and 25% niobium. Hardness, metallographic and dilatometric techniques will be employed to determine the kinetics and the interrelationship of the several reactions controlling the decomposition of beta in these alloys. These studies will be supplemented by x-ray diffraction work in order to clarify the mechanism of the reactions. The x-ray studies will concentrate on the tetragonal "Widmanstatten" phase observed in quenched alloys, on the omega phase and on the segregation phenomenon observed in the low temperature aging of retained beta.

Transformation in zirconium-niobium alloys are relatively complex as indicated in the following summary:

(1) Isothermal transformation of beta or aging of retained beta at high temperatures yields the equilibrium phases alpha and niobium rich beta ($beta_2$). Depending on composition or temperature, these phases may form individually or in combination via a degenerate pearlite mechanism.

(2) Two distinctly different reactions occur when retained beta is aged at lower temperatures; an omega phase analogous to that in titanium alloys and a segregation of beta into high and low niobium components. Undoubtedly, these same reactions will be encountered in the isothermal transformation of beta under appropriate conditions.

(3) Alloys containing less than about 7% niobium form alpha by a martensite reaction when quenched to room temperature from the beta field. Above about 7% niobium, M_s is displaced below room temperature; however, in this composition range, Widmanstatten platelets with a tetragonal structure are observed in quenched alloys.

Work to date on this program has been concentrated on the low temperature aging of retained beta in quenched alloys. These studies have revealed a complex reaction sequence involving several intermediate states preceeding the formation of an omega phase. The Debye-Scherrer powder method has been found incapable of answering many questions which arise concerning the mechanisms involved in this reaction sequence. Consequently, reaction kinetics will be studied by correlation of dilatometric, hardness and metallographic data with that obtained from a systematic x-ray study using single crystal methods. Conventional as well as stepped aging and retrogression treatments are planned in an effort to isolate the individual stages of decomposition for closer study.

Isothermal transformation of beta will be studied for the temperature range from the upper transus to about 200°C. It is anticipated that the isothermal formation of omega, particularly at the higher temperatures (below about 550°C) may differ significantly from that observed in aging treatments. Thus, isothermal studies may help considerably in understanding the beta to omega transformation and its influence on mechanical behavior of these alloys.

Contractor: Columbia University, New York, New York

Contract: AT(30-1)-1593

Brief Title: A STUDY OF INCLUSIONS IN URANIUM

Investigators: George L. Kehl

Scope of Work

The purpose of this research is to identify inclusions and intermediate phases in uranium metal by metallographic methods.

An experimental procedure was developed to remove selected constituents from the microstructure by means of the micromanipulation of an ultrasonic "jack hammer". Debris from this operation was subjected to x-ray diffraction analysis for identification. On the basis of existing differences in the cathodic behavior of the microconstituents in a given electrolyte and under prescribed cell conditions, metallographic identification at 200X magnification was established by noting the time required to electroplate the constituent with copper (and the character of the copper deposit in some cases).

Using these techniques the following microconstituents in uranium metal have been identified: UN, U(C,N) UC, Uo, Uo_2, UH_3, and U_6Fe.

Contractor: Illinois, University of, Urbana, Illinois

Contract: AT(11-1)-67, Proj. No. 20

Brief Title: FATIGUE BEHAVIOR OF DILUTE ALLOYS OF NIOBIUM

Investigators: G. M. Sinclair and C. A. Wert

Scope of Work

The purpose of this work is to explore the effects of interaction between dislocations and interstitial atoms on the elevated temperature fatigue behavior of dilute alloys of niobium. Initially the research undertaken is intended to provide base line information on the fatigue characteristics of unalloyed niobium sheet and rod as influenced by grain size and degree of cold deformation. Elevated temperature studies will be conducted on samples containing controlled amounts of nitrogen and oxygen as contaminants. Experimental determinations of fatigue strength at different temperatures and loading frequencies will be analyzed to investigate the accuracy with which modified strain aging concepts can be used to predict anomalous fatigue behavior.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-981

Brief Title: PHYSICAL METALLURGY OF UNCOMMON METALS

Investigators: John T. Norton and Robert B. Ogilvie

Scope of Work

The purpose of this program is to study fundamental physical phenomena of potential interest to the AEC. Topics for study are selected primarily on the basis of the curiosity of the investigators and on their suitability for use as doctoral theses problems. A summary of recent and current experimental investigations is given below.

The isothermal section of the Zr-Si-B system at 1400°C has been established. It has been confirmed that there are no compounds of Si-B. Silicides and germaides of yttrium have been prepared by arc melting. The structures of alpha YSi_2, beta YSi_2 and YSi have been determined.

The texture and mechanical properties of niobium sheet have been completed. The elastic constants have been evaluated from which the anisotropy factor was calculated. The interesting feature is that the anisotropy factor is less than unity.

The modes of deformation of 13 single crystal of alpha uranium were studied at low and elevated temperatures. The new (110) (001) slip system was found at liquid nitrogen temperature. Also, a micro-kinking process was found at 350°C.

The magnetic properties of the compound UFe_2 has been studied from room temperature down to liquid helium temperatures. Alloys of the type $U(Fe,Mn)_2$ will be studied over the same temperature range.

Diffusion studies and the determination of the miscibility gap by the diffusion couple techniques in the U-Nb system is well underway. Analysis of the diffusion couples is being done with the electron microbeam probe.

Contractor: National Bureau of Standards, Washington, D.C.

Contract: NBS Project No. 0802-11-4100

Brief Title: CONSTITUTION OF URANIUM ALLOYS

Investigators: L. L. Wyman and J. J. Park

Scope of Work

The primary objective of this program is the establishment of the constitutional diagrams of selected binary uranium alloys. The alloys of uranium with the six platinum metals are presently being studied by means of thermal, metallographic, and x-ray diffraction methods. These six separate systems have each shown a eutectic in the region of 90 - 85^{a}/o uranium, the existence of intermetallic compounds formed at relatively high temperatures, and strong tendencies for reactions with ceramic materials.

The phase diagram work on the U-Pt system has been completed and a publication version is being prepared. The U-Ru, U-Pd, and U-Rh systems are well advanced, with the investigations centering on the high temperature reactions in alloys of high-platinide-metal content. The U-Os and U-Ir alloys of high uranium content have thus far been studied. Because of certain similarities, the U-Re binary will be studied in conjunction with the platinide group. Upon completion of the studies on this group of metals, the alloys of uranium with the individual rare earth metals will be prepared for similar studies.

A potentiometric method of transformation kinetics has been developed and will be applied to alloys selected on the basis of results from the phase diagram studies.

A secondary objective of this program is the development of a method of predicting alloying characteristics of uranium, facilitated by these studies of the reactions of uranium with a series of metals of comparable properties and characteristics.

Contractor: Sylvania Electric Products, Inc., Bayside, New York

Contract: AT(30-1)-GEN-366

Brief Title: FUNDAMENTALS OF DIFFUSIONAL BONDING

Investigators: L. L. Seigle and L. S. Castleman

Scope of Work

The objective of this project is to clarify the process of formation and growth of intermetallic layers between two dissimilar metals bonded at an elevated temperature. The phenomena governing the formation and growth of intermediate phase layers occurring at the bonded interface are of importance to metallurgists engaged in the development of clad fuel elements. Although considerable experimental work has been done, it has been mostly of an empirical nature and the fundamentals of the processes occurring are not sufficiently well understood. As part of the current program, the kinetics of growth of intermetallic phase layers is being studied in several binary systems to determine if the results can be satisfactorily interpreted in the light of existing diffusional theory. Thus, an evaluation is being made of the effects of interface concentrations, interface displacements and diffusion coefficients on the course of interdiffusion. Also, applied pressure has been found to exert a profound influence on the kinetics of growth, the structure, and the mechanical properties of the zone of diffusion. Accordingly, an investigation is concurrently under way to determine whether the predominant effect of pressure is to alter the thermodynamic equilibria between phases in a system or to inhibit (unequally) the rates of atomic migration in the phases.

Contractor: Syracuse University, Syracuse, New York

Contract: AT(30-1)-1910

Brief Title: PHASE EQUILIBRIA STUDIES OF ALLOY SYSTEMS INVOLVING ONE OR MORE ALKALI AND ALKALINE EARTH METALS AS COMPONENTS

Investigators: A. J. King and F. A. Kanda

Scope of Work

The purpose of this study is to determine the phase equilibria of alloy systems involving as components one or more alkali and alkaline earth metals. An intensive study of the allotropy of calcium and strontium and their alloys is being made. It is hoped to obtain general trends and analogous behavior among the two families of metals toward solubilities in the liquid and solid states, intermediated phase formation, allotropy and the thermodynamics and mechanisms of phase formation, allotropy and the thermodynamics and mechanisms of phase transformations. Ultimately, it is anticipated that the study of these relatively uncomplicated or "ideal" metal systems may contribute to increased understanding of the metallic state. The phase systems Mg-Ba, Ca-Sr, Ca-Ba, Sr-Ba, Ba-N and Ba-Li have been completed and the systems Mg-Ca, Mg-Sr, Ca-Li and Ba-In are in various stages of completion. A sensitive method has been developed for the quantitative determination of liquidus temperatures and liquid phase immiscibility by automatic recording of the variation of liquid phase density with temperature. Selected results from these studies are summarized below:

A) The barium-lithium system has been studied over the entire range of compositions. One intermediate phase, $BaLi_4$, was observed, melting incongruently at 156°C and crystallizing on a hexagonal cell with $a = 10.92A^{\circ}$, $c = 8.94A^{\circ}$. A structure is proposed but further study is needed. No measurable solid solubilities were observed.

B) A study of the calcium-lithium system is in progress, one intermediate phase, $CaLi_2$, which melts incongruently at 233°C was observed. Solid state transitions and solubility limits are being investigated.

C) The phase equilibria in the strontium-calcium system has been determined. The liquidus-solidus curves show complete miscibility at all compositions in the liquid and solid state. There is a minimum in these curves at 78 ± 2 w/o Sr, M.P. 738°C. In the solid state, alloys of all compositions undergo the FCC $\rightleftarrows$ HCP $\rightleftarrows$ BCC transitions. The transition temperature-composition curves have been established by high temperature X-ray diffraction methods. An anomaly in the FCC $\rightleftarrows$ HCP transition is being examined further.

A phase equilibrium study of the Co-La system is in progress and the terminal solubility of Ni in Sr and Ba is being determined. It is proposed to continue the studies in progress and to initiate work in the systems Li-Sr, Na-Ba and Na-Sr.

Contractor: Tufts University, Medford, Massachusetts

Contract: AT(30-1)-1410

Brief Title: BASIC PROPERTIES OF LIGHT METAL HYDRIDES

Investigators: Charles E. Messer

Scope of Work

This research is concerned with a fundamental investigation of physical and thermodynamic properties of light metal hydrides, including phase relations, densities, heat capacities, hydrogen dissociation pressures and crystal structures.

Lithium hydride systems have been emphasized in this program and progress has been acceptable. A study of the solid-liquid equilibria in the system lithium-lithium hydride has been completed and a paper was published. Phase equilibria studies in the lithium hydride-lithium fluoride and lithium hydride-lithium oxide systems are in progress. It is proposed to extend this work to the systems lithium-hydride-calcium hydride, lithium hydride-titanium hydride and later to systems of lithium hydride with other alkaline earth and transition hydrides.

Section I-B

MATERIALS PREPARATION

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: PREPARATION, STRUCTURE AND PROPERTIES OF URANIUM COMPOUNDS

Investigators: A. Tripler and A. Austin

Scope of Work

Investigations are in progress to prepare and to observe physical and mechanical properties of refractory uranium compounds other than uranium dioxide. Properties of interest include melting point, chemical stability, irradiation stability, structure, thermal and electrical conductivity and mechanical behavior.

Recent work was concentrated on the preparation of high-purity, dense specimens of UBe_{13}, UC, UC_2, UN and UB_2 and measurements of their chemical and physical properties. A study of the effects of irradiation on the properties is in progress.

UBe_{13} specimens were made having densities approximating 100 per cent of theoretical and were essentially single phase. Reaction rates with oxygen, nitrogen, water vapor, and boiling liquid water were measured. Thermal expansion from room temperature to 1800 F was measured and thermal conductivity was measured to about 1500 F.

UC_2 specimens were made having a density of 89 per cent of theoretical and contaminated by only a small amount of UC. Reaction rates with nitrogen, oxygen, and water vapor were measured. Specimens of UC having densities of 95 to 98 per cent of theoretical were prepared. Thermal expansion was measured up to 1800 F.

The structures of uranium carbides, UC, U_2C_3, and UC_2 were studied by neutron diffraction. The positions of the carbon atoms, type of metal-carbon bonding, and of carbon-carbon bonding were determined. UC_2 has carbon-carbon double bond while U_2C_3 has more triple bond character.

The uranium-nitrogen-carbon system has been studied under vacuum and 1-atm nitrogen. There is complete solid solubility between UN and UC and essentially no solid solubility of nitrogen in UC_2 or U_2C_3, or of carbon in U_2N_3. At 1800 C, the two-phase fields, U(C,N) and UC_2, U(C,N) and C, and three-phase field U(C,N), UC_2, C exist in vacuum or argon. At 1-atm nitrogen the uranium nitrides are more stable than uranium carbides up to 1800 C, but at 2000 C, the uranium carbides, UC_2 and U(C,N) become more stable. The results are reported in BMI-1272 and are being prepared for publication.

Contractor: Flow Corporation, Arlington, Massachusetts

Contract: AT(30-1)-2049

Brief Title: METAL PURIFICATION AND CRYSTAL PREPARATION

Investigators: Richard S. Davis

Scope of Work

The purpose of this study is to establish a source of supply of metal single crystals with a purity greater than currently available for use throughout the AEC Research program. The effort is one activity in a "Pure Materials Program" sponsored by the AEC Division of Research. Initial objectives will be to produce high purity nickel, copper, gold, silver and single phase alloys in the form of single crystals.

Apparatus for growth from the melt of single crystals of nickel and nickel alloys has been constructed and is being used to establish suitable growth conditions. A device for locating and controlling the position of the solid-liquid interface is being developed. The distribution of selected impurities resulting from zone refining techniques is being studied.

Contractor: National Carbon Company, New York, New York

Contract: AT(30-1)-2051

Brief Title: PREPARATION OF SEMICONDUCTING MATERIALS

Investigators: R. D. Westbrook, R. L. Cummerow, E. Brazis, and R. M. Broudy

Scope of Work

The purpose of this study is to establish a source of supply of research quantities of semiconducting materials (elemental or compound) having a purity greater than available currently for use throughout the AEC research program. It is a further aim of this work to prepare various types of p-n junctions, such as pulled, alloyed, and diffused, as may be required by AEC contractors. The effort is one activity in a "Pure Materials Program" sponsored by the AEC Division of Research.

Techniques have been perfected to prepare single crystals of Ge, Si, and InSb by the usual pulling process. In addition, floating zone and horizontal zone refining equipment are available. A special zone leveler, essentially completed, will produce large single crystals of Ge containing low dislocation densities, and will be used to supplement the work on preparation of long lifetime crystals. Materials other than the three mentioned above will be prepared when needed.

Section I-C

REFRACTORY MATERIALS

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: BASIC RESEARCH IN CERAMICS

Investigators: M. Volpe and P. Stablein

Scope of Work

Present work is chiefly directed toward a study of the mechanism of sintering in Al_2O_3. This has been conducted by a technique of measuring of interface necks between sapphire spheres against a sapphire plate after firing to sintering temperature and cooling. Two deductions have been made: (1) the mechanism is always a volume diffusion process, and, (2) process rates depend on chemical interaction of the surfaces with the atomosphere. Interface growth is fast in dry H_2, but slows appreciably in moisture or in He. It nearly stops in a good vacuum.

The techniques developed for Al_2O_3 are presently being applied to studies on UO_2, with particular emphasis on effects of minor solution diluents, such as TiO_2 and ThO_2, on the self diffusion rates and on sintering.

Another program recently initiated deals with the kinetics of oxidation and reduction of UO_2 and U_3O_8 in CO and oxygen bearing atmospheres.

Contractor: National Bureau of Standards, Washington, D.C.

Contract: NBS Project No. 0901-11-4400

Brief Title: PROPERTIES OF CERAMICS AT ELEVATED TEMPERATURES

Investigators: F. P. Knudson, S. Spinner and L. E. Mong

Scope of Work

One phase of the study is a determination of the effect of porosity and grain size on the mechanical properties of polycrystalline ceramic bodies as a function of temperature. A general, emperical equation has been formulated to relate mechanical strength and microstructural conditions at room and elevated temperatures. The relation has been applied successfully to urania, thoria and chrominum-carbide bodies, and, in addition, has been useful in predicting the influence on mechanical strength of minor additions of other oxides to these materials.

A second objective of this investigation is to obtain an improved understanding of the role of microstructure and composition in the elastic and anelastic behavior of polycrystalline ceramic bodies as a function of temperature. The experimental methods being used are a sonic (resonance) method for determining elastic moduli and a "time of resonance decay" method for determinging internal friction.

Contractor: Pennsylvania State University, University Park, Pennsylvania

Contract: AT(30-1)-1710

Brief Title: RESEARCH ON GRAPHITE

Investigators: C. R. Kinney and P. L. Walker, Jr.

Scope of Work

The purpose of the work is to investigate the formation of different amorphous and graphitized carbons from pure organic compounds, to establish chemical and physical procedures to better define carbon, and to correlate the properties of carbon with its reactivity to gases.

Work on the high temperature pyrolysis of pure organic compounds is being continued. The mechanism of the decomposition of benzene at 1200°C, and the mechanism of the deposition of different types of carbon resulting from this decomposition are under study. Also refinements are being made in our method of determining the rate of decomposition of organic compounds by measuring the electrical conductivity of the carbon deposited under conditions which give a continuous film between two electrodes. When these refinements of method are completed, detailed kinetic studies of the high temperature decomposition of pure organic compounds will be made using this equipment.

Plans for continued study include (1) the effect of carbon structure and atmosphere on subsequent gas release upon heating graphite to elevated temperatures, (2) rate of diffusion of gases through irradiated and unirradiated porous carbon bodies, (3) the use of low angle x-ray scattering to obtain additional information on the closed pore volume in carbon and (4) the effect of impurities on the rate of carbon gasification.

Contractor: Stanford Research Institute, Menlo Park, California

Contract: AT(04-3)-115, Project No. 6

Brief Title: VOLUME CHANGE ON MELTING UO_2

Investigators: Frank H. Halden

Scope of Work

The major objective of this program is to determine the change in volume of sintered uranium dioxide as its temperature is increased from immediately below to immediately above the melting point. Also, this research will include attempts to determine the volume change that occurs when a specimen of known density and microstructure is heated to the melting temperature, the surface tension of molten uranium dioxide, the volume change upon solidification at the melting point, and the volume change that occurs when a fused specimen is cooled to room temperature.

The pendant drop and sessile drop techniques will be employed in this investigation, utilizing the solar furnace as a heat source. Photographs will be taken of the specimen during the complete heating and cooling cycle.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: CERAMICS RESEARCH

Investigators: C. E. Curtis, L. A. Harris, A. G. Tharp, and G. D. White

Scope of Work

Scope I - Effect of Atmosphere on Ceramic Reactions

It is known that the sintering behavior of some ceramic materials and the high temperature reactions between others are affected by the nature of their atmosphere. Studies are being conducted on the rare earth oxides (excluding promethium oxide) at temperatures to 1700°C in oxygen and hydrogen at atmospheric pressure. Future studies will include the effect of pressure. A second study is concerned with the ThO_2-SiO_2 phase equilibrium diagram. A preliminary diagram has been determined in air. Further investigations will determine the effects of reducing and oxidizing atmospheres.

Scope II - Structure of Some Rare Earth Compounds

A study is being made of the structures of the carbides, nitrides, lower oxides, and silicides of samarium, europium, and gadolinium. The structure and properties of these compounds will be compared with similar compounds of the group IV A and actinide elements. From such studies, one may obtain information regarding the atom size, bonding directional properties of silicon and carbon, and the degree of metallic bonding.

Scope III - Thermodynamic Properties of Some High Melting Compounds of Thorium and Uranium

The free energies and heats of formation of thorium and uranium silicides are being measured by the Knudsen effusion method. Later these properties will be determined for the nitrides and carbides. The data will be useful in predicting the possible course of high temperature reactions and in estimating the strength of bonds in these compounds.

Scope IV - Diffusion of Thorium in Thorium Oxide

The temperature dependence of the diffusion coefficient of Th^{+4} in ThO_2 is under study using Th^{230} as a tracer. These measurements will be extended to include the effects of small additions of Ca^{+2} and Nb^{+5} on the diffusion of Th^{+4}. Any change in diffusion rate may give data which can be used to determine the type of defect controlling material transport in such systems.

Section I-D

CORROSION AND OXIDATION STUDIES

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: BASIC CORROSION STUDIES

Investigators: J. E. Draley, W. E. Ruther, S. Mori, R. D. Misch, and F. E. DeBoer

Scope of Work

The purpose of these studies is to determine mechanisms and controlling factors in the aqueous corrosion of reactor materials. The primary materials being investigated are Al, Zr, U, and Mg.

1. Aluminum

Higher temperature tests (above 200°C) are being conducted as a function of flow rate in a dynamic loop. An apparatus has been constructed to measure the diffusion rate in a dynamic loop. The role of hydrogen is being investigated through measurements of the diffusion rate of corrosion-produced hydrogen through an iron-backed aluminum diaphragm in an attempt to relate penetration rate of the corrosion film to general corrosion behavior. Satisfactory techniques have been developed for measuring polarization potentials. The techniques are being applied to problems in the corrosion mechanisms of Al and Zr.

2. Zirconium

The electrical resistance of the oxide film on zirconium and its relation to corrosion behavior is being studied by the use of two different methods. In one method a specimen is coated with spots of evaporated aluminum or silver, and resistance is measured between the spots and the base metal. In the other method the emf across the film is measured during corrosion by using one electrode touching the oxide surface and another spot welded to the base metal. Both methods have shown that the zirconium oxide film on zirconium-titanium alloys becomes a better conductor with increasing titanium. It was also shown that this alloy system has a corrosion rate maximum at about 15 a/o titanium. The effects of various other alloying elements are under investigation.

Contractor: Case Institute of Technology, Cleveland, Ohio

Contract: AT(11-1)-258

Brief Title: SCALING OF ZIRCONIUM AT ELEVATED TEMPERATURES

Investigators: W. M. Baldwin, Jr.

Scope of Work

The purpose of this investigation is to obtain an improved understanding of the mechanism of oxidation of zirconium at elevated temperatures. It has been demonstrated that zirconium oxidized in air or in oxygen-nitrogen mixtures at temperatures between 600 and 1300°C exhibits (1) a black, adherent, protective film which changes progressively after a finite time to a less dense, non-protective, white oxide, (2) a large increase in the oxidation rate after a finite time, designated "breakaway", and (3) severe lateral surface growth of the zirconium after some critical time. All three phenomena are time and temperature dependent and also appear to be sensitive to the scaling atmosphere, specimen geometry and dimensions, and metal purity.

An experimental result, to which great significance was attached, indicated that when iodide zirconium was scaled in pure oxygen none of these changes was noted and the resultant surface film was dark and adherent. This observation led to a hypothesis that the mechanism for oxidation of zirconium required the presence of a zirconium nitride layer between the metal and the zirconium oxide, coherent with the metal lattice. During the past year the experimental work was centered in testing the validity of this mechanism and the effects of metal impurities on oxidation behavior.

Continued studies of the scaling mechanism include: a) the role played by impurity elements such as hafnium, and b) the distribution of the reacting gas (the scaling reaction leads to scale formation as well as to gas dissolution). Once the scaling mechanism is known, effective controls can be designed to minimize or eliminate growth and breakaway.

Contractor: Florida, University of, Gainesville, Florida

Contract: AT(40-1)-1826

Brief Title: STUDY OF THE FILMS FORMED ON ZIRCONIUM-NIOBIUM ALLOYS IN OXYGEN ATMOSPHERES

Investigators: Mary Louise Brey

Scope of Work

The purpose of this project is to study the reaction, at elevated temperatures, of alloys of zirconium and niobium with pure oxygen at reduced pressure. Data are obtained by two separate techniques:

(1) Volumetric measurement at definite time internals of the oxygen, at constant pressure consumed by the alloy

(2) X-ray diffraction pattern of the oxide layer when it is formed in sufficient quantity to obtain a diffraction pattern of the free oxide.

The kinetic data obtained by the former procedure are of fundamental importance to an understanding of the mechanism of the oxidation. From such data activation energies for the oxidation of the various alloys will be calculated.

From its X-ray diffraction spectrum the overall composition of each oxide is determined. The oxidation products of these alloys have been found in this study to consist of one or more of the following crystalline phases: monoclinic ZrO_2, orthorhombic $6ZrO_2 - Nb_2O_5$, orthorhombic Nb_2O_5. Because the various oxide phases require considerable time to reach equilibrium, even at 900°C., a study of their progress toward a condition of equilibrium is planned. Finally, the crystallinity of the oxides varies with temperature and may be determined from the X-ray diffraction line widths.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-2081

Brief Title: INVESTIGATION OF PHENOMENA RELATED TO LIQUID METAL CORROSION

Investigators: David A. Stevenson and John Wulff

Scope of Work

This is a fundamental investigation of liquid metal corrosion under the influence of thermal gradients, known as mass transfer.

The type of corrosion of particular interest is precisely described as temperature gradient mass transfer. This is best illustrated by considering a closed loop in which liquid circulates in a solid metal container from a hot zone to a cold zone. In such a loop, transfer of container material from the hot zone to the cold zone is found to occur. This transfer has been explained as solution of the container material in the liquid metal in the hot zone, bulk transfer of the liquid alloy to the cold zone, and consequent growth of container material in the cold zone due to supersaturation of the liquid alloy. Conditions exist whereby the container material is depleted in the hot zone and deposited in the cold zone.

In the search for a mechanism governing this process, the following factors are being studied: (1) equilibrium solubilities and their dependence on temperatures; (2) the kinetics and mechanism of solution; and, (3) factors influencing nucleation and growth in supersaturated solutions.

Contractor: Missouri, University of, Rolla, Missouri

Contract: AT(11-1)-73, Proj. No. 5

Brief Title: CORROSION OF NUCLEAR METALS

Investigators: M. E. Straumanis and W. J. James

Scope of Work

It is the purpose of this study to determine the basic reactions of dissolution, the rates of dissolution, the effect of added salts, and the extent of polarization, all as related to the behavior of metals (Hf, Zr, Ti, Be, U, Th), their oxides and nitrides in hydrofluoric and either inorganic acids.

The results of these studies are not necessarily translatable into any practical service, but they do give some information as to the fundamental behavior of the metals or solid solutions in corrosive environment. The nature of the measurements gives a quantitative evaluation of the effect of impurities either in the metal or in the corrosive medium.

Contractor: Oregon, University of, Eugene, Oregon

Contract: AT(45-1)-535

Brief Title: ELECTROCHEMICAL STUDIES ON THE CORROSION OF NUCLEAR REACTOR METALS

Investigators: Wendell M. Graven

Scope of Work

Interesting photogalvanic effects associated with anodic oxide films formed on zirconium and niobium have been observed using ultraviolet radiation. An attempt to gain a better understanding of this effect will be made by study of the shifts in potential of zirconium and niobium electrodes resulting from irradiation with ultraviolet light of known intensity and wavelength. Tests will be made with the electrode immersed in various electrolytes maintained at a variety of temperatures, and also in the absence of an electrolyte. Correlation of the oxide absorption spectra with the variations in photocurrents which occur when the electrodes are illuminated with radiation of various wave lengths will be attempted.

Section I-E

PHYSICAL CHEMISTRY OF REACTOR MATERIALS

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: REACTION KINETICS OF HYDROGEN AND NITROGEN WITH NIOBIUM

Investigators: W. M. Albrecht

Scope of Work

A study is in progress to determine the kinetics and mechanisms of the reactions of hydrogen and nitrogen with niobium. The investigation includes the determination of the following:

(a) Temperature-pressure-composition equilibria and phase diagrams of the Nb-H and Nb-N systems.

(b) Kinetics and mechanisms of the sorption of hydrogen and nitrogen in niobium.

(c) Diffusion rates of hydrogen and nitrogen in niobium.

Consistent, reproducible equilibrium data have been obtained for the Nb-H system for ratios of H/Nb between 0.01 to 0.85, at pressures between 0.03 to 1000 mm mercury and up to 900°C. In addition, diffusion coefficients and kinetic data are being obtained. Similar studies in the Nb-N are in progress.

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: MIGRATION OF HYDROGEN IN ZIRCONIUM HYDRIDE

Investigators: H. H. Krause, Jr.

Scope of Work

Under the influence of a thermal gradient, hydrogen in zirconium hydride migrates toward the cooler part of the material. In this investigation, the effect of this migration is to be studied principally in the delta phase of the hydride. Some effort will be directed toward the investigation of the diffusion rates, but the emphasis is on the steady state, which is attained when the influence of the thermal gradient has been balanced by the resulting concentration gradient. An attempt will be made to achieve a linear, one-dimensional thermal gradient in a sample which can be sectioned readily or otherwise prepared for hydrogen analysis. Distribution of hydrogen after the attainment of the steady state is to be determined.

The methods of the thermodynamics of irreversible processes, as developed by de Groot and others are to be applied to the steady state results in order to correlate the thermal diffusion results with other properties of the system, such as the phase relationships, the equilibrium pressures of hydrogen, ordinary diffusion constants, and thermal conductivity. The isothermal diffusion constants for the delta phase zirconium hydride have already been determined as part of the program. A permeability technique for measuring the diffusion constants in the solid solution beta phase is being investigated at present. These experimental data, with that obtained by thermal gradient measurements, will be combined with previously known data in an effort to specify equations that will predict hydrogen migration under a given set of conditions.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1024

Brief Title: ELECTROCHEMICAL STUDIES OF NON-AQUEOUS MELTS

Investigators: G. Derge

Scope of Work

The broad objectives of this contract are to expand knowledge of the chemical characteristics of high temperature non-aqueous melts as typified by fusions of sulfides, oxides, and salts. The study originated by direct measurements of such properties as electrical conductivity and transport phenomena. However, it has become advantageous to supplement these by determination of diffusion coefficients, density and surface tension which contribute to more detailed interpretation of the data. Appropriate experimental procedures have been developed for work in the range 1000° - 1600°C.

The electronic conductivity of several molten sulfides resembles that exhibited by solid semi-conductors. The compositions of these sulfides can be altered through control of the sulfur pressure over them and the resultant conductivity changes in both the Cu-S and Fe-S systems permit interpretation analogous to the energy level diagrams used for crystalline semi-conduction. For the low S range of the Cu-S system the gap between valance and conduction bands is about 0.7 electron volts. Such studies are being extended to other systems to provide a more complete and quantitative picture.

It has also been shown that molten FeS has a high thermoelectric power. Such measurements will be extended to other electronically conducting melts and in special cases it is hoped that the related Hall effect can be measured.

Metal solubility has been observed or postulated in several molten systems and the exact nature of this solubility has been interpreted in several ways. It is expected that the thermoelectric power and Hall potential studies will help resolve these questions.

Contractor: New York University, New York City, New York

Contract: AT(30-1)-1837

Brief Title: STUDIES ON SODIUM-POTASSIUM ALLOYS

Investigators: B. R. Sundheim

Scope of Work

The activity and associated thermodynamics properties of the components of a series of sodium-potassium alloys are being determined by absorption spectrophotometry of the vapor over the temperature range 150°-350°C.

Solutions of sodium and potassium metals in polyethers are being characterized by means of physical techniques such as electrical conductivity, potentiometry and electrical paramagnetic resonance spectrophotometry.

Contractor: New York University, New York, New York

Contract: AT(30-1)-1902

Brief Title: THERMODYNAMICS OF TITANIUM - OXYGEN AND NIOBIUM - OXYGEN ALLOYS

Investigators: Kurt L. Komarek

Scope of Work

Thermodynamic properties of oxygen in titanium-oxygen, zirconium-oxygen, hafnium-oxygen and vanadium-oxygen, niobium-oxygen, tantalum-oxygen alloys will be determined by equilibrating these alloys with alkaline earth metal vapors of known partial pressure and solid alkaline earth metal oxide. The work done so far concentrated on establishing a reliable experimental technique and proving the reproducibility of the method proposed.

An arc-welding unit has been constructed to weld and seal specimen tubes in inert atmosphere or in vacuum. Calcium oxide was prepared by techniques used in analytical chemistry. Several successful runs were completed and the thermodynamic properties of oxygen in the alloys calculated. A plot of partial molal free energies vs. weight percent oxygen in the metals titanium and zirconium show that partial molal free energies increase first slowly then exponentially with decreasing oxygen concentration. The curves show a distinct break at the a/a+b - boundary. The values for zirconium are higher than for titanium, indicating that the activity for oxygen of zirconium is higher than for titanium. It was found that specimens of different transition metals with similar partial molal free energies of oxygen (titanium, zirconium, hafnium and vanadium, niobium, tantalum) can be equilibrated in one run. By this modification the time for the investigation of these systems can be reduced quite considerably.

It is planned to investigate first the thermodynamic properties of oxygen in metals of group IV B of the periodic system (titanium, zirconium, hafnium) up to approximately 30 atomic % oxygen and then include metals of group V B (vanadium, niobium, tantalum). The latter will be investigated up to 10 atomic % oxygen.

Contractor: Sylvania Electric Products, Inc., Bayside, New York

Contract: AT(30-1)-GEN-366

Brief Title: SELF-DIFFUSION OF NIOBIUM

Investigators: L. L. Seigle, L. S. Castleman and R. Resnick

Scope of Work

The objective of this research project is to determine the self-diffusion coefficient of niobium as a function of temperature. A method has been developed whereby diffusion couples are prepared by the vapor decomposition of $Nb^{95}Cl_5$ at the surface of a niobium disc. The Nb^{95} forms a thin adherent layer on the surface and the specimen is subsequently diffused at high temperatures in vacuo. The rate of diffusion is determined by slicing and measuring the gamma ray activity of the slices. Values of the self-diffusion coefficient have already been obtained at 1800° and 2000°C, and it is planned to complete the program by obtaining additional data in the temperature range 1600° to 2200°C.

Section II

PHYSICAL RESEARCH IN THE NATURE OF MATTER

Section II-A

GENERAL SOLID STATE PHYSICS AND PHYSICAL METALLURGY

Contractor: Ames Laboratory, Iowa State College, Ames, Iowa

Contract: W-7405-eng-82

Brief Title: SOLID STATE STUDIES

Investigators: R. G. Barnes, G. C. Danielson, R. H. Good, D. E. Hudson, L. D. Jennings, J. M. Keller, S. Legvold, R. E. Rundle, F. H. Spedding, and C. A. Swenson

Scope of Work

Scope I - Thermal Properties of Metals and Alloys at High Temperatures

The general purpose of this investigation is to increase knowledge of the thermal properties of metals and alloys at high temperatures. The first specific objective has been to develop methods for the measurement of the thermal diffusivity (k), the specific heat (c), the density (d), and the thermal conductivity (K = kcd) to a precision better than two percent over the temperature range 20°C to 1000°C.

A modified Angstrom method has been developed by which thermal diffusivity may be measured to a precision of two percent over the temperature range 20°C to 1000°C, and the method has been applied to the following pure metals: uranium, thorium, iron, nickel, copper, silver, gold, and tantalum. Discontinuities associated with Curie temperatures and phase changes are clearly discernible, and can be examined quantitatively. The electrical resistivity and thermoelectric power can be measured at the same time as the thermal diffusivity, and these electrical measurements are often helpful in interpreting the thermal properties.

A pulse-heating method has been developed by which the specific heat of metals may be measured to a precision of two percent over the temperature range 20°C to 1000°C, and the method has been applied to pure platinum, iron, nickel, and thorium. Variations of the specific heat near phase transitions can be studied in some detail. The electrical resistivity is always measured at the same time as the specific heat. It may also be possible to obtain, with the same equipment, the thermal conductivity of the wire sample. The possibility of making thermal measurements on a sample inside a reactor is being examined.

Scope II - Properties of Semiconducting Materials

The general purpose of this research program is to increase the understanding of electronic conduction in all solids, and particularly in semiconducting materials. Our first specific objective has been to grow single crystals of various semiconducting solids, and to investigate the temperature dependence of the electron and hole mobilities.

Single crystals have been grown of Mg_2Si (both n and p type), Mg_2Ge (both n and p type), Mg_2Sn, Mg_2Pb, and Cu_2Se. Electrical resistivities and Hall co-

efficients of Mg_2Si and Mg_2Ge have been measured over the temperature range $77^{\circ}K$ to $1000^{\circ}K$. The energy gaps and mobilities have been determined, and the importance of optical-mode scattering and impurity scattering has been demonstrated quantitatively. The temperature coefficient of the energy gap has been determined from infrared absorption measurements; the ratio of electron mobility to hole mobility in the intrinsic region has been determined from thermoelectric power measurements.

Resistivities and Hall coefficients have been measured for microscopic single crystals of boron. The mobilities of the charge carriers were very low, and, in contrast to the situation in most semiconductors, the hole mobility was greater than the electron mobility.

Future work will include the following:

(a) the extension of the temperature range to one degree K; (b) the measurements of the cohesive energies, elastic constants, dielectric constants, and Debye temperatures, which enter into the theory of electron scattering; (c) the study of additional compounds.

Scope III - Properties of Rare Earth Metals and Alloys

The purpose of research on the rare earth metals is to increase the understanding of solid state phenomena by examination and correlation of properties in these closely related elements.

The resistivities of the rare earth metals have been measured as a function of temperature, and anomalies have been found which agree in temperature with previously known magnetic transitions. Clearly, the magnetic properties of the shielded 4f electrons have a strong effect on the electrical conductivity. Many of the rare earth metals have been found to exhibit paramagnetism, antiferromagnetism, and ferromagnetism as the temperature is lowered. Measurements on single crystals have revealed cases of very pronounced anisotropy in the magnetic properties.

The thermodynamic properties of the rare earth metals have been studied in some detail. Specific heats have been measured with high precision from 1 K to $300^{\circ}K$, and these results are being correlated with magnetic susceptibility, electrical resistivity, and thermal expansion. Dilatometric measurements have shown plastic flow just below the melting points.

The cohesive energies of all but one of the stable rare earth metals have been measured by the mass - spectrometric method developed in this Laboratory. The outstanding feature of the data is the unexpected wide variation in cohesive energies among the rare earth metals.

The effect of alloying a magnetic rare earth metal with a non-magnetic similar metal has been studied, particularly in Gd-La and Gd-Y alloys. For alloys of high Gd content, paramagnetic to ferromagnetic transitions were observed as the temperature was decreased. As the Gd content was decreased,

ferromagnetic ordering was changed to antiferromagnetic ordering. When lanthanum, which is non-magnetic and a superconductor, was diluted with gadolinium, which is magnetic and a non-superconductor, the superconducting transition temperature decreased linearly with Gd composition. A change of 1.5°K for 0.6 atomic percent Gd was observed. Recently, magnetic measurements have been made on Gd_3C and Tb_3C at temperatures from 4°K to 375°K and at magnetic fields from 2 kilogauss to 14 kilogauss.

Scope IV - High Pressure Effects at Low Temperatures

This work is directed toward an application of high pressure techniques to the study of solid state phenomena at temperatures close to absolute zero. In particular, we have studied the effect of pressures up to 10,000 atmos. on the superconducting transitions of tin, indium, thallium, tantalum, and mercury. The resulting data show that the transition temperatures for all of these metals except thallium (which shows a previously observed anomalous behavior) is more nearly a linear function of volume than of pressure. Solid hydrogen is used as a pressure transmitter, and since it is not a true fluid, some deformation of the samples was found. In order to evaluate this effect, we are currently deforming tin samples (both polycrystalline and single crystal) in liquid helium at low pressures, and determining the effects of this deformation on the critical field curve for tin. A study also is being made of the effects of annealing and alloying on the superconducting transition in tantalum, since anomalous results were obtained in the pressure effect for this metal.

The high pressure-superconducting experiments also showed that two separate modifications of solidified mercury (differing in density by 1.5 percent) are stable at zero pressure below 90°K. The P-V-T relationships for both of these have been studied, as well as the transition between the two phases. It is not definitely known whether the new phase is a superconductor or not, and experiments to determine this and the structure are now in programs.

Scope V - Magnetic Resonance in Solids

The purpose of the program in magnetic resonance is to investigate phenomena such as: self-diffusion in metals and alloys, magnetic ordering, interactions between nuclei and electrons, relaxation processes, and the dependence of these phenomena upon external parameters such as temperature and pressure.

The nuclear quadrupole resonance spectra of the group III B trihalides $AlBr_3$, AlI_3, $GaCl_3$, $GaBr_3$, GaI_3, $InBr_3$, and InI_3 have been shown to be essentially equivalent and indicate that these compounds are dimeric in the solid state. By contrast, the compounds $TlCl_3$ and $TlBr_3$ are monomeric in the solid state.

An experimental and theoretical investigation into the existence of electron-coupled interactions between nuclei in the halides of some heavy metals has shown such interactions to be very weak. This may be largely attributed to the strongly ionic nature of the bonding in such compounds.

The nuclear magnetic resonance of sodium in the sodium tungsten bronzes (Na_xWO_3) has been observed and the Knight shift has been measured.

Scope VI - Crystal Structure

Crystal structure research provides information which is basic to the understanding of most solid state phenomena.

The effect of crystalline field upon the structure of complex transition metal ions has been studied, and such fields shown to be influenced by the size of the accompanying cation. The magnetic electron distributions in these complex transition metal ions and their effect on magnetic ordering have also been investigated both experimentally and theoretically, and the observed and predicted structures have been compared.

Some compounds (such as Na_xWO_3, $Ca_{1-x}La_xC_2$, and $Ca_{1-x}La_xB_6$) behave as metals because they have closed-shell structures plus extra electrons. By means of neutron diffraction, a super-lattice and distortion of the oxygen positions from the ideal perovskite structure have been found in $Na_{0.75}WO_3$. Also, by neutron diffraction, the C-C distances in MC_2 compounds have been shown to increase as the electron density increases.

Delocalized bonds (which are intermediate between metallic bonds and ordinary electron pair bonds) have been found. The causes of bond delocalization have been discovered and extensions of the principles learned are being extended to organo metallic compounds.

Scope VII - Special Studies

The general purpose of this research is to increase knowledge of the solid state by the exploitation of unusual techniques and unusual properties.

The conduction counting properties of diamond have been used to explore directly the trapping levels responsible for charge capture. We have found that the cross-section for photoelectric emptying of the traps rises very abruptly for photons of energy near 3.0 ev.

The Hall coefficients of the sodium tungsten bronzes have shown that the number of free electrons is exactly equal to the number of sodium atoms over a wide concentration range. The anomalous minimum in the resistivity versus concentration has been explained in terms of demonstrated (a) inhomogeneity of the crystals, (b) long range order of the sodium atoms, and (c) abrupt changes in the density of electronic states.

The resistivity of thin potassium films as a function of thickness has followed theoretical predictions and has shown that the scattering of electrons from surfaces is completely diffuse. The unusual electrical properties of unannealed arc evaporated carbon films strongly suggests that this form of carbon has much greater atomic disorder than any other form of solid carbon.

A study of the ionization of rare-earth atoms at the surfaces of tungsten and iridium has revealed some effects which are contrary to present theories and which are closely related to the nature of the solid surfaces involved.

Contractor: Atomics International, A Division of North American Aviation, Inc., Canoga Park, California

Contract: AT(11-1)-GEN-3

Title: ELECTRONIC PROPERTIES OF REACTOR MATERIALS

Investigators: G. W. Lehman, T. G. Berlincourt, R. R. Hake and F. J. Hon

Scope of Work

The objective of this investigation is to establish a better understanding of electronic structure and behavior in reactor materials. In order to gain information on the magnitude of the Fermi energy and the shape of the Ferni surface in metals, a program of high field de Haas-Van Alphen studies has been initiated. The nature of the Fermi surface is also being explored by low temperature measurements of the electronic specific heats of various metals and alloys. These same measurements also determine the DeBye temperature as a function of alloying thus providing information on the influence of lattice parameter and electron concentration on the elastic constants.

The effects of alloying on electronic wave functions in metals is being studied by means of nuclear magnetic resonance techniques. These techniques are also being used to investigate the crystallography, defect structure, crystalline electric fields, and diffusion mechanisms in the hydrides and deuterides of zirconium and uranium. Theoretical effort is being devoted to correlation of the experimental information with electronic energy band calculations. A Hartree type approach coupled with the auguented plane wave method is being used to calculate the energy band structure in thorium and uranium.

Contractor: Atomics International, A Division of North American Aviation, Inc., Canoga Park, California

Contract: AT(11-1)-Gen-8

Brief Title: TRANSPORT PHENOMENA IN SOLIDS

Investigators: W. G. Brammer and C. J. Meechan

Scope of Work

The study of transport phenomena in solids is directed toward obtaining knowledge of the basic mechanisms of mass transport in such systems as pure metals, alloys, and gases in metals. It is the intent to determine: 1) the species which is mobile with respect to the lattice; (2) the nature of the diffusion step processes; and, (3) the properties which determine the redistribution of atoms when these systems are placed in large temperature gradients.

Extensive measurements on the effect of a large temperature gradient ($2500^{\circ}C/cm$) or mass transport in iron in the alpha-phase have shown that there is no change in the Kirkendall-type-marker spacings greater than the uncertainty of these spacings. A similar experiment under less ideal conditions has been performed in copper. No marker motion was observed here, also. Tentative conclusions can be formed on the basis of these data but further work is necessary to determine whether the results are fortuitous or indicate a need for revision in the theory.

The effect of a large temperature gradient on the large vacancy flow that appears to exist during the early stages of interdiffusion in sintering experiments is also being studied. Temperature gradients of around $15,000^{\circ}C/cm$ in the region of contact of the 1/8 inch metal cylinders have been achieved while the interface fillets are .010 inch wide and the interface temperature is $900^{\circ}C$.

Contractor: Brookhaven National Laboratory, Associated Universities, Inc., Upton, Long Island, New York

Contract: AT(30-2)-Gen-16

Brief Title: NEUTRON DIFFRACTION RESEARCH

Investigators: G. J. Dienes, B. C. Frazer, M. Kay, T. Mitsui, R. Nathans, G. H. Vineyard, W. Whittemore, R. Pepinsky

Scope of Work

The neutron diffraction program continues to be concerned mainly with structural studies on magnetic and ferroelectric crystals. Some studies are also in progress in which crystal structure is of secondary importance. These studies are directed at some fundamental questions in the theory of magnetism, the theory of the liquid state, and the character of crystal transitions.

Scope I - Magnetic Crystals

Magnetic form factors have been studied using the polarized beam spectrometer. Some interesting conclusions have been drawn from comparision of the neutron results on iron, nickel, and cobalt and the similar measurements with x-rays made by Weiss and DeMarco. For iron, the x-ray and neutron results are in agreement while those of cobalt and nickel show differences both in the absolute magnitude and in the shape of the form factors as a function of angle. Furthermore, since the x-ray results (after subtraction of the argon core) represent the contributions of all the 3d localized electrons while the neutrons scatter only from the unpaired electrons, the two sets of measurements allow one to calculate the radial distribution for the two spin states separately. This has been done for nickel and shows a separation of several hundredths of an angstrom between the peaks of the electron densities for the two spin states.

A further study has been made on the neutron scattering of Fe_3Al at low temperatures. This material was previously shown to possess iron atoms with two distinct magnetic moment values. The low temperature neutron measurements definitely rule out the possibility that this behavior could be explained by an antiferromagnetic coupling between next nearest neighbor atoms via an aluminum atom. Additional work on Fe_3Al single crystals with polarized beams also demonstrated that the form factors of the magnetic electrons surrounding the two different iron sites are essentially the same. All this evidence tends to support the view that there is a transfer of electrons between the iron atoms and the nearest neighbor aluminum atoms.

As a part of the continuing program on the spin structures of magnetic oxides the mixed systems $MnFe_2O_4$-$MnCr_2O_4$ and $FeTiO_3$-Fe_2O_3 have been investigated. The

former is of interest because of the wide differences between the observed magnetic moment values and those predicted by the Neel model. Measurement of the individual sublattice magnetizations showed that: (1) it is the octahedrally coordinated sites which always possess a magnetic moment less than expected; (2) the temperature dependence of the sublattice magnetizations are in agreement with those calculated by J. S. Smart from the Neel theory; (3) all the magnetic scattering data can be fitted to an antiparallel arrangement for the two sublattices thereby excluding ordered triangular spin configurations; (4) there is a definite connection between the difficulty in saturating the material and the anomalous behavior of the octahedral magnetization.

The $FeTiO_3$-Fe_2O_3 system is of interest because of the appearance of ferromagnetism in a system combining two antiferromagnetic substances. It was shown that $FeTiO_3$ possesses a magnetic unit cell twice as large as the chemical cell. Such a large cell calls for the existence of an antiferromagnetic coupling between the Fe^{2+} atoms over a considerably larger distance than had been expected. For the mixed structures containing more than 20% Fe_2O_3, the presence of ferrimagnetism has been shown to come from an ordered antiparallel arrangement of layers of Fe^{2+} atoms and mixed $Fe^{2+}Ti^{4+}$ layers.

Scope II - Ferroelectric Crystals

The final refinement of the ferroelectric Rochelle salt structure has been completed. Among the many atomic displacements found in the structure (relative to the paraelectric symmetry), the most significant one appears to be that of one of the hydroxyl groups on the tartrate molecule. The experimental work has been followed by a theoretical local field treatment based on the proper structure. In the theory it was also possible to generalixe Mueller's phenomenological theory of Rochelle salt, and to describe the dielectric properties of the clamped crystal.

A study is now in progress on the atomic Debye-Waller parameters for $BaTiO_3$, in the cubic paraelectric phase. It is hoped that this work will result in a determination of the shape of the potential well for Ti. The thermal vibrations of Ba and Ti must be isotropic, but anisotropy may be present in the case of oxygen. According to a theory of Devonshire, the complete ferroelectric behavior of $BaTiO_3$, can be worked out from a thorough knowledge of the nonferroelectric cubic phase. Two additional ferroelectric structure analyses were started in cooperation with x-ray diffraction studies at the Pennsylvania State University: $(CH_2NH_2COOH)_3H_2SO_4$ (tri-glycine sulfate), and $(ND_4)_2BeF$. The x-ray work on both of these crystals is well along, but the neutron analysis is still in the stage of data collection.

Scope III - Order-Disorder Transformations

The study of short-range order in brasses is of fundamental interest but is complicated by the similarity of the scattering powers of copper and zinc for both x-rays and neutrons. The neutron scattering lengths of Cu^{63} and Cu^{65} have been measured and are significantly different from natural copper. The Cu^{65} isotope has the larger scattering length, and the short range order scattering in

brass with Cu^{65} as the copper constituent is six and one-half times more intense than that with natural copper. With the above enhancement of short range order scattering the order-discorder transformation in beta-brass becomes experimentally accessible to neutron diffraction techniques. The diffuse scattering of monochromatic neutrons from a single crystal of beta-brass isotopically enriched in Cu^{65} has been measured at three temperatures above the critical temperature of 468°C. Analysis of these data is in progress. At each temperature Fourier inversion of the scattering gives the local correlation of atoms, i.e., the degree to which an atom influences the distribution of atoms on neighboring sites. These data will serve as criteria for establishing the validity of the various theories of such cooperative phenomena.

The effect of temperature on diffuse short range order scattering has been neglected in the past. In connection with the above experimental work, a concise expression for the intensity of the diffuse scattering from binary alloys has been developed in which the effects of temperature are explicity included. This temperature effect cannot be neglected in measurements of short range order at elevated temperature and at high scattering angles. For example, failure to include this effect places the published value of the first neighbor short-range order coefficient in Cu_3Au at 405°C in error by approximately 25%, and the error is larger for the more distant neighbors and the higher temperatures.

Scope IV - Liquids

A new approach to the scattering of slow neutrons by liquids has been found. By means of this approach the cross section for scattering with change of momentum and energy has been successfully factored into a part dependent on simultaneous correlation of atomic positions and a part dependent on the details of motion of a single atom in the liquid. Approximate theories of the latter motion give useful expressions for the cross section, and, conversely, measurements of neutron scattering by a liquid can now be used to determine the motion of an atom. The extension of these methods to molecular liquids is now being considered. The time of flight technique has been used to study the scattering of neutrons by lead just below and just above the melting point and at a temperature of 300°C above the melting point. This study was made to check the new theory of neutron scattering discussed above. One of the simple models, the diffusional one, suggested by Vineyard does not describe the temperature dependence of the neutron scattering for lead, whereas the gas model does come near to agreeing with the experiments.

Contractor: California Institute of Technology, Pasadena, California

Contract: AT(04-3)-221

Brief Title: FUNDAMENTAL STUDIES OF MATERIALS PERTAINING TO NUCLEAR ENGINEERING

Investigators: Pol Duwez

Scope of Work

A project of limited scope will be initiated in each of the following categories as soon as promising graduate students and research fellows become available: (1) Effect of Radiation on Crystalline Solids (2) Alloy Systems of Importance in Nuclear Engineering (3) Non-Metallic Materials for High Temperature Applications (4) Liquid Metals and their Interaction with Solids.

Work in the first category will be related to the problem of radiation damage in solids. The objective will be to use high energy particles as a tool for the study of the solid state.

The second category involves the determination of phase diagrams and a study of the kinetics of phase changes. This would include studies of diffusion of substitutional and interstitial atoms and of martensite-type reactions. Metals such as Zr, Nb, U, and Th May be considered.

The third category will include the study of refractory materials for fuel elements and structural materials. These materials include oxides, carbides, nitrides, borides, and silicides.

The fourth category is concerned with a study of the structure and properties of liquid metals and liquid alloys. Also the mechanism of corrosion by liquid metals and the effect of cavitation may be included.

Contractor: California, University of, Berkeley, California

Contract: AT(11-1)-34, Proj. #28

Brief Title: DISLOCATION ORIGIN AND PROPERTIES

Investigators: J. Washburn

Scope of Work

The purpose of this investigation is to study the origin of dislocation networks produced during crystal growth from the liquid and the relationship between dislocation distribution and crystal properties. The general plan of attack is a systematic study of the changes in dislocation distributions accompanying changes in crystal growth conditions (temperature gradients and rates of growth) and then a correlation of the dislocation populations with structure sensitive properties.

Lithium fluoride crystals have been used for the experiments because in these transparent crystals it has been possible to make individual dislocations visible by formation of a string of fine precipitated particles along the lines. Techniques have been developed for the growth of highly perfect lithium fluoride crystals from the melt; the detection of dislocations on crystal surfaces by etch pits; and the decoration of dislocations within crystals by precipitation along dislocations lines, making it possible to follow microscopically individual dislocations through great thicknesses of crystal. During the coming year it is planned to extend the experiments to metal crystals using high sensitivity X-ray diffraction techniques to follow dislocation networks.

The experiments so far have lead to the conclusion that most of the dislocations in crystals grown from the melt under typical conditions are formed by stress induced multiplication processes. The formation of prismatic edge dislocation loops by collapse of plate shaped clusters of vacant lattice sites during growth from the melt has not yet been definitely established.

Contractor: Case Institute of Technology, Cleveland, Ohio

Contract: AT(11-1)-623

Brief Title: SOLID STATE PHYSICS

Investigators: Charles S. Smith

Scope of Work

THIN FILM PHYSICS: The origin of uniaxial magnetic anisotropy and the resultant domain structure in evaporated Fe films is being investigated. The spontaneous magnetization is also being studied at low temperatures. Macrostress in Fe films resulting from imperfection decay is being measured. X-ray examination for preferred orientation is in progress. Large crystals of Cu, grown epitaxially on substrates at room temperature, will be examined for imperfection decay during various annealing cycles.

COHESIVE PROPERTIES: The restrictions imposed by the Third Law of Thermodynamics on the energy spectrum of macroscopic systems are being studied by the methods of statistical mechanics. Relationships to the theory of phase equilibrium will be explored.

A semi-quantitative theory of the electronic band structure of low-symmetry metals (Bi, Ga, In, Sn) will be developed, with emphasis on a perturbed simple structure which provides the essential electronic features.

Approximations made in calculations of the elastic constants of simple metals will be re-examined in an attempt to explain the experimental elastic constants and their pressure derivatives.

The single-crystal elastic constants of magnesium alloys in the composition range from 2.00 to 2.02 electrons per atom will be measured at $4^{\circ}K$ in an extension of the search for predicted anomalous behavior.

The measurement of the elastic constants of single crystals of K, Rb and Cs will be attempted.

LOW TEMPERATURE PHYSICS: The initial effort in the low temperature program will be de Haas-Van Alphen measurements in pure magnesium and indium single crystals. Such measurements are directed to shedding light on the band structure of these metals.

Contractor: Chicago, University of, The Institute for the Study of Metals, Chicago, Illinois

Contract: AT(11-1)-357

Brief Title: RESEARCH ON THE SCIENCE OF MATERIALS

Investigator: Earl A. Long

Scope of Work

The Institute is primarily concerned with basic understanding of the properties of metals and of the solid state in general. To this end it believes in the mutual interaction of its staff members (metallurgists, physical chemists, and physicists) so that the talents and facilities of widely-varying disciplines may if necessary be applied to attain knowledge of the properties of materials.

Present activities include work on the following topics:

Theoretical and experimental research on the Group V semi-metals and their alloys.

Plastic deformation of single crystal alloys at low temperatures.

X-ray determination of crystal structures of metals and alloys over a wide range of temperatures.

Electrical and thermal properties of semi-conductors at low temperatures, with particular reference to impurity band conduction.

Studies on surface diffusion using the field emission microscope.

Nuclear magnetic resonance used as a tool for studying the superconducting state; optical properties of metals and alloys.

The effect of high intensity shock waves on the structure of metals and alloys.

Development of zone-refining and zone-leveling techniques.

Studies on synthetic graphite.

Self-diffusion and impurity diffusion in metals, alloys, and in certain ionic crystals.

Thermodynamics of liquid metals and alloys.

Contractor: Colorado, University of, Boulder, Colorado

Contract: AT(11-1)-377

Brief Title: RESEARCH ON METALS AND ALLOYS AT LOW TEMPERATURES

Investigators: William F. Love

Scope of Work

The ultimate purpose of this program is to obtain information on the energy band structure of metals and semiconductors through measurement of galvanomagnetic properties, and to determine the influence of very strong magentic fields on these properties. This includes measurements made with apparatus using pulsed magentic fields and oscilloscope recording techniques in addition to the conventional electromagent and D. C. measuring equipment. The pulsed magnetic fields available range up to 320 kilogauss. Investigations of these properties in bismuth and indium antimonide are in progress and will be continued. An investigation of the Hall effect in tin bismuth alloys at low magnetic field strengths is presently being carried out. Also in progress is the development of apparatus for measuring magnetic susceptibilities by a pulsed field technique.

Contractor: Illinois, University of, Urbana, Illinois

Contract: AT(11-1)-67, Proj. No. 22

Brief Title: PROPERTIES OF MATERIALS AT LOW TEMPERATURES

Investigators: J. C. Wheatley and R. I. Hulsizer

Scope of Work

The properties of materials at temperatures below 1°K have been studied up to this time primarily by classical thermodynamic types of measurements. This study will determine the properties of magnetic crystals below 1°K using nuclear and electronic resonance techniques. Resonance techniques are useful since they give a microscopic picture of events occuring in the crystals. The nuclear resonance measurements also provide either an absolute thermometer or a thermometric parameter in the temperature region where other methods of thermometry fail.

It is proposed to study first the properties of cerium and dysprosium ethyl sulfate. As suitable thermal contact techniques are developed, the work will be extended to materials which do not cool themselves.

Contractor: Illinois, University of, Urbana, Illinois

Contract: AT(11-1)-67, Project No. 15

Brief Title: DIFFUSIONLESS PHASE CHANGES IN NON-FERROUS METALS AND ALLOYS

Investigators: T. A. Read and D. S. Lieberman

Scope of Work

Metallographic, x-ray, and resistivity studies will be continued on both isothermal and athermal transformations in alloys of the gold-cadmium system of several compositions. Emphasis will be placed on the reaction kinetics and thermodynamics of the transformations rather than on the crystallographic features since the later phase of the work has been studied in considerable detail.

The effects of lattice defects on the transformation will be studied using impurity atoms and point and line defects introduced by plastic deformation and quenching. Investigations of the several stabilization phenomena will also be made. Radio-active tracer diffusion experiments will be carried out on alloys of several compositions in order to ascertain the diffusion constants and the activation energies and to study the nature of the defects introduced by quenching. In connection with the thermodynamic studies, a calorimeter will be constructed to measure the latent heat of transformation, specific heats, and if possible, the twin boundary energy. The latent heat is of particular interest because it has already been determined from purely mechanical measurements by a rather novel experiment. Information concerning the twinning and transformation dislocations will be sought using high resolution electron microscopy in order to further understand the detailed mechanism of martensitic transformations.

Contractor: Illinois, University of, Urbana, Illinois

Contract: AT(11-1)-67, Proj. No. 9

Brief Title: ANNEALING OF COLD WORKED METALS

Investigators: Paul A. Beck

Scope of Work

This is an investigation of phenomena associated with the recrystallization of high purity metals. First attention is being given to aluminum and the following problems are being examined:

1) Study of the effect of solute atoms on polygonization.

2) Correlation of information on substructures obtained by dislocation etching methods and by x-ray diffraction methods.

3) Investigation of the recrystallization texture in a rolled artificially nucleated aluminum crystal, in relation to the rolling texture.

4) Study of grain boundary migration and strain relief in fine grained high purity aluminum by means of microscopic and x-ray diffraction techniques.

Contractor: Johns Hopkins University, Baltimore, Maryland

Contract: AT(30-1)-2185

Brief Title: ESTABLISHMENT OF THE ANGSTROM AS A STANDARD FOR X-RAY WAVE LENGTHS

Investigators: J. A. Bearden

Scope of Work

Relative X-ray wavelengths, crystal dimensions in the study of the physics of solids, and energy states of atoms can be measured easily to a few parts in 10^6. The absolute values, however, depend on the ruled grating measurements of x-ray wave lengths, which, because of basic experimental difficulties, may be in error by 50 to 100 parts per 10^6. The purpose of this investigation is to establish quantitative primary x-ray wave length standards.

In the new wave length measurement a high precision double crystal x-ray monochromator will be used to select a portion of an x-ray emission line which will then be measured by a ruled grating in the usual x-ray manner. This method will avoid the difficulties due to the asymmetry of the spectral lines and the shift in wavelength caused by impurities in the x-ray target which were present in all previous ruled grating measurements.

Contractor: Maryland, University of, College Park, Maryland

Contract: AT(40-1)-2068

Brief Title: PROCESSES OF DIFFUSION AND ELECTRICAL CONDUCTION IN SOLIDS

Investigators: Homer W. Schamp, Jr.

Scope of Work

The purpose of this research is to elucidate certain aspects of diffusion in the alkali halides and, further, to study the effect of some experimental variables on the process. The diffusion mechanism in the case of the alkali halides is known to be a vacancy mechanism and the effect of changing the variables can thus be more clearly followed than in the case of material for which the mechanism is not determined with certainty.

The research program will include the following parts: (1) the development of techniques for precise measurement of diffusion coefficients and electrical conductivities, (2) the application of these techniques to measurements over a wide temperature range in several alkali halides, and (3) measurement of the diffusion coefficients and electrical conductivity of alkali halides under varied conditions and investigation of other properties related to ionic motion. The measurements will include the diffusion of divalent metal ions in alkali halides such as Ca^{++}, Cd^{++}, and Sr^{++} in NaCl and KCl, self-diffusion and electrical conductivity under pressure, and investigation of the effect of a thermal gradient on the crystals.

Currently, the experimental effort is directed toward the measurement of diffusion coefficients of several different ions in the alkali halides and to the design and assembly of apparatus to measure electrical conductivity under pressure.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1002

Brief Title: FUNDAMENTALS OF SOLID SOLUTIONS AND RECRYSTALLIZATION

Investigators: M. B. Bever, B. L. Averbach, and M. Cohen

Scope of Work

Scope I - Metallurgical Thermodynamics (Bever)

The investigation of the energy effects and changes in physical properties of metals resulting from various physical and chemical processes will be continued. Past research has been concerned mainly with the measurement by tin solution calorimetry of the stored energy of cold work in gold-silver alloys, the energy of ordering of Cu_3Au, the energy of formation of various metallic compounds and solid solutions and the excess energy in electrodeposited silver. The effects of cold work at room temperature on ordered and disordered gold-copper alloys have also been investigated. Recently completed work includes a detailed investigation of the effects of variables, in particular strain, strain rate and temperature of cold working on the stored energy in gold-silver wires and an investigation of the changes during annealing of ordered and disordered Cu_3Au after cold work. An associated study of the vapor pressure in the systems Ag-Cd-Zn and Cu-Ni-Zn has recently been completed.

In the current period the investigation of the effects of cold work on the energy content of metals will be extended to working at $4^{o}K$. In particular, the effects of strain and strain rate in torsion at this temperature will be investigated. Measurements of the energy stored in specimens deformed by explosive loading have been begun. Changes in such other properties as electrical resistivity and hardness will be followed.

Further research on ordered alloys will include the measurement of the energy stored during deformation of ordered and disordered Cu_3Au at $-195^{o}C$. Other property changes resulting from this deformation will also be investigated. Measurements of the energies of formation and of ordering of $CuAu_3$, are under consideration.

Accumulated data on the heat of solution of various elements in tin and of heats of formation, specifically of copper-nickel alloys, will be evaluated. The principles of tin solution calorimetry will be considered from an analytical viewpoint.

Scope II - Thermodynamics and Structure of Solid Solutions (Averbach and Cohen)

This research program is concerned with x-ray and thermodynamic studies of the structure of solid solutions. The main focus of this program is the study of the details of the local structure by x-ray methods and the thermodynamic properties on the same alloy system in order to advance our understanding of solid solutions. The principal activity is on the Fe-Al system. New x-ray techniques for the determination of local atomic displacements and the Debye temperature have been developed and are being applied.

Uranium-niobium alloys are also being studied. This system forms a miscibility gap with a continuous series of solid solutions at temperatures above 900°C. Preliminary data indicate that interesting variations in chemical behavior, electrical resistance and mechanical properties occur in alloys quenched from 900°C. X-ray studies of diffuse scattering and local displacements are being undertaken in an effort to see if the mechanical and physical property changes are associated with the short range order or with the rearrangement of imperfections in the quenched alloys.

Scope III - Fundamentals of Cold Work and Recrystallization (Averbach)

X-ray techniques are being applied to the study of imperfections in metals and alloys introduced by plastic deformation, radiation damage, and martensitic transformations. One of the principal x-ray techniques involves the careful measurement of diffraction line shapes and the representation of these shapes as a Fourier series. The Fourier coefficients are then interpreted in terms of local strains, particle sizes, and stacking fault probabilities. Radiation damage is being studied by this technique in Li-Mg alloys. Lithium fissions under nuclear bombardment, and it is expected that metallurgical changes will be large and indicative of the processes occurring in a reactor fuel element. X-ray diffraction line broadening and short range order changes will be studied and observations on the local atomic displacements will be made by means of diffraction intensity measurements.

Deformation and annealing studies are also being made on alpha brass which has been deformed by cold rolling and subsequently annealed in the recovery and recrystallization ranges.

Contractor: Michigan State University, East Lansing, Michigan

Contract: AT(11-1)-400

Brief Title: THERMAL PROPERTIES OF SEPARATED METALLIC ISOTOPES

Investigators: D. J. Montgomery

Scope of Work

In the exploitation of isotopic mass as a probe for the investigation of matter in the aggregate, two fields of particular attractiveness are lattice-vibration spectra in solid metals and salts, and the rheological and electrical properties of liquid metals. As specific investigations in these fields, the following topics are to be studied:

a) the electrical resistivity of isotopic alloys of Li^6 and Li^7 from liquid-helium temperature (4^oK to the melting point (180^oC)

b) the electrical resistivity, specific volume, and viscosity of Li^6 and Li^7 from 180^oC to 300^oC

c) the feasibility of specific-heat measurements on Li^6, Li^7; Li^6F, Li^7F; Li^6H^1, Li^6H^2, Li^7H^1, Li^7H^2

d) the feasibility of nuclear magnetic resonance and x-ray measurements on Li^6 and Li^7 through the melting point.

Contractor: Minnesota, University of, Minneapolis, Minnesota

Contract: AT(11-1)-477

Brief Title: STUDY OF THE SOLID SOLUBILITY OF INTERSTITIAL ELEMENTS IN TRANSITION METALS

Investigators: Morris E. Nicholson

Scope of Work

During the period September 30, 1958, to September 30, 1959, the investigation of the solid solubility of interstitial elements will consist of four parts: determination of the solubility of carbon in silver-palladium alloys, determination of the solubility of carbon in certain ternary alloys near the silver paladium binary, determination of the solubility of nitrogen in cobalt-nickel alloys, study of the cobalt-nickel system for indication of ordering.

The study of the carbon solubility in the several alloy systems will be determined by saturating the alloys with carbon in a carbon monoxide atmosphere. The nitrogen solubility in the cobalt-nickel system will be determined if suitable analytical techniques can be developed. The possible ordering in cobalt-nickel alloys will be studied by dilotometry and electrical resistivity.

All of these investigations are directed toward a more fundamental understanding of the factors which control the solid solubility in transition metals.

Contractor: Ohio State University Research Foundation, Columbus, Ohio

Contract: AT(11-1)-191

Brief Title: SOFT X-RAY SPECTRA OF METALS AND ALLOYS

Investigators: C. H. Shaw and E. L. Jossem

Scope of Work

The purpose of the project is to make experimental and theoretical studies of the characteristic x-ray emission and absorption spectra of titanium, zirconium, hafnium, and thorium, and alloys of these metals. This work is related to that in the Metallurgy Department of ORNL, which is furnishing specimens of the pure metals and such alloys as are of significance.

The experimental part of the program involves measurements of the spectral energy distribution of the x-ray emission and absorption spectra of the above mentioned materials with instruments of high resolving power and with a linear intensity scale. Measurements are now made in the range of wavelengths between 1 and 15 Angstroms. Extension of such measurements to the long wavelength region - 50 to 1000 Angstroms - is proposed when a suitable instrument for exploring this region is constructed.

The theoretical part of the program is concerned with the interpretation of the experimental data in an effort to understand in greater detail the electronic structure of metals and also the effects of alloying on this structure. Such improved understanding is expected to lead to better understanding of physical and mechanical properties of metals and alloys.

Contractor: Pennsylvania, University of, Philadelphia, Pennsylvania

Contract: AT(30-1)-2151

Brief Title: ON THE ORIGIN OF DISLOCATIONS

Investigators: Doris Kuhlmann-Wilsdorf and Robert Maddin

Scope of Work

Although it is generally agreed that the slip in crystalline substances in general and metals in particular is due to the motion of dislocations, the origin of these is still in some doubt. A recent theory suggests that during plastic deformation, dislocations are released from prismatic dislocations acting as Frank-Read sources which have formed through the condensation of thermal vacancies.

In order to test this idea, and, to attempt the preparation of metal specimens with high mechanical strength, it is intended to prepare single crystals of f.c.c. metals in such a way that no condensation of thermal vacancies can take place. This is to be done using three different methods: (1) Growing thin single crystalline wires or foils and cooling them so slowly that any excess of thermal vacancies can diffuse out of the free surfaces, (2) Growing thin single crystalline wires or foils, taking care that the temperature in no part of them drops more than a few degrees below their melting point, then quenching them rapidly with the intention to inhibit the formation of large prismatic dislocations, (3) Producing monocrystalline metal foils through epitaxial electro-deposition at room temperature.

The mechanical strength of specimens obtained by these methods will be determined and their crystal perfection will be investigated with x-ray techniques, both in their initial state and after the completion of heat treatments designed to introduce prismatic dislocations.

Contractor: Pittsburgh, University of, Pittsburgh, Pennsylvania

Contract: AT(30-1)-2163

Brief Title: CALORIMETRIC, STRUCTURAL AND MAGNETIC STUDIES OF METALS AND INTERMETALLIC COMPOUNDS

Investigators: R. S. Craig

Scope of Work

The general purpose of this investigation is to study the nature of the metallic bond. Specific topics now under investigation or planned for future consideration are the following:

1. The determination of the temperature variation of the long-range order parameter in Mg_3Cd and $MgCd_3$ using x-ray diffraction techniques.

2. The determination of the heat capacities of Laves phases of $MgCu_2$ and $MgZn_2$ between 10 and 500°K and for zinc between 200 and 300°K to establish the entropy of these materials. Certain needed improvements in the existing calorimetric facilities are to be made before these studies are undertaken.

3. The preparation of solid solutions of several rare earth metals in gadolinium as a solvent. A study will then be made of the effect of solute concentration on the lattice parameter, saturation moment and Curie point.

4. A study of the Ti-H system, using x-ray diffraction techniques, to establish the primary solid solubility of hydrogen in titanium at room temperature and to locate the phase boundary separating the primary solid solution from the intermediate (cubic) phase formed with increasing hydrogen content.

5. The determination of the electronic heat capacity of certain "electron compounds" such as NiAl, Cu_3Ge, etc., to ascertain the density of states in these materials.

Contractor: Rensselaer Polytechnic Institute, Troy, New York

Contract: AT(30-1)-2159

Brief Title: MECHANISM OF THE EFFECT OF RARE EARTH SOLUTES ON THE ALLOTROPIC TEMPERATURE OF ZIRCONIUM

Investigators: A. A. Burr, D. J. Lam and L. Ianniello

Scope of Work

The allotropic transformations which occur in metals such as iron, titanium, zirconium, and uranium enable the properties of these metals to be greatly modified through alloying. The effect of alloying elements on the transformation is naturally of importance in understanding and predicting the properties of these metals. It is the purpose of this investigation to study the effect of solute elements on the transformation temperature of zirconium.

A theory, first developed from a study of iron-base phase diagrams and later extended to titanium, zirconium and uranium, characterizes solute elements wih a differential heat of mixing (Q_B). This Q_B value is specific for each element and establishes the manner in which the transformation temperature of the solvent metal changes with alloying. The proposed research will attempt to extend this theory more fully to zirconium and fill in the missing data showing the periodic nature of the Q_B value versus atomic number. Of special interest are certain rare earth binary combinations with zirconium because of their potential use.

During the first year, the zirconium rich regions of the following systems will be investigated; Zr-Sc, Zr-Yt, Zr-La (or possibly Zr-Pr or Zr-Nd if La does not prove to be appreciaLy soluble). The phase boundaries will be determined by both high temperature x-ray and metallographic techniques A modern Heraeous vacuum - arc furnace has been installed and will be used to produce the alloys. The heat treatments of the alloys will be carried out under argon atmosphere.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: MICRO-STRESSES IN CRYSTALS

Investigators: B. S. Borie, Jr., and C. J. Sparks, Jr.

Scope of Work

Crystal imperfections influence the x-ray diffration pattern of a material by causing some of the intensity normally concentrated at Bragg maxima to be distributed throughout reciprocal space. Measurements of the intensity and distribution of such diffuse scattering may often be interpreted to identify the nature and concentration of such defects. An x-ray diffractometer employing strictly monochromatic radiation has been constructed to carry out such studies, and measurements are in progress. Current primary interest includes short range order in metallic solid solutions, diffraction effects in disordered alloys associated with atomic size differences of its constituents, and radiation damage in metals.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: FUNDAMENTAL PHYSICO-METALLURGICAL RESEARCH

Investigators: L. K. Jetter, J. O. Betterton, Jr., G. D. Kneip, Jr., C. J. McHargue, and H. L. Yakel, Jr.

Scope of Work

Scope I - Alloying Behavior of Group IVA Elements

The object of this study is to determine the factors affecting the alloying behavior of the Group IVA elements - titanium, zirconium, hafnium. The alloy systems investigated to date are those of zirconium with gallium from Period 4 and with silver, cadmium, indium, tin, and antimony from Period 5, and with lead from Period 6. Experimental studies are being made of the phase boundaries, the axial ratio of the close-packed hexagonal phase, and the electronic specific heats at helium temperatures for these alloy systems. The interrelationship of these properties will provide a test of a current theory of alloying based on electron concentration.

Scope II - Preferred Orientation in Metals

The investigation of the type and degree of preferred orientation offers an opportunity to study the nature of the atom rearrangement in metals brought about by mechanical deformation and by annealing. New techniques for the quantitative determination of texture have been developed and are being employed to follow the development of textures as a funtion of amount of deformation, to determine the paths of movement of the reference axes toward stable and orientations, to study differences in texture of metals of the same crystal structure, and to investigate the changes in preferred orientation brought about by annealing. The data obtained from the experimental program will be employed to evaluate critically the theories of texture formation. Associated studies are concerned with the difference in deformation behavior of single crystals and polycrystalline aggregates.

Scope III - Low and High Temperature Crystal Structures

X-ray diffraction facilities for studies of structures in the range $4^{\circ}K$ to $1800^{\circ}K$ are available. Of interest in the low temperature program are the body-centered cubic metals, the lanthanide and actinide elements, and hydrides of titanium and zirconium. The nature and the kinetics of the transformations in cerium and titanium hydride have been extensively studied. A high-pressure x-ray camera will be used to study the effect of pressure on the low temperature transformations.

Contractor: Western Reserve University, Cleveland, Ohio

Contract: AT(11-1)-493

Brief Title: POINT IMPERFECTIONS IN METALS

Investigators: Stefan Machlup

Scope of Work

Point imperfections (lattice vacancies and/or interstititals) have been shown to be important in the process of interdiffusion of metals, as evidenced by the Kirkendall effect; they are thus very likely to be responsible also for self-diffusion. The influence of such defects on the properties of metals, such as resistance, thermo-electric power, specific heat, elastic constants, internal friction, yield stress, will continue to be studied in this research. A calculation for alkali metals is in progress.

The following approaches have been planned: (1) using analytical expressions for the potential in the vicinity of an imperfection, study the influence of changing the lattice constant upon the formation energy of the imperfection, and, (2) study the contribution to thermal resistivity of scattering of phonons by point imperfections and impurities.

Section II-B

PHYSICAL CHEMISTRY OF STATES

Contractor: California, University of, Berkeley, California

Contract: AT(11-1)-34, Proj. #33

Brief Title: THERMODYNAMIC FUNCTIONS FOR THE METALLIC STATE

Investigators: R. R. Hultgren

Scope of Work

The purpose of this program is to: (1) examine critically the published thermodynamic data for metallic systems (2) select the most reliable data on the basis of calculations and correlations and (3) compile and publish the selected data as it becomes available in the form of mimeographed sheets for retention in a loose-leaf binder. The data considered includes vapor pressures, electromotive force measurements, calorimetry, phase equilibria, and standard thermodynamic functions.

Progress has continued to be very satisfactory and a total of 47 systems have been examined since the project began in June 1955. When the survey is completed it is planned to publish the results in the form of a book for general distribution.

Contractor: California, University of, Berkeley, California

Contract: AT(11-1)-34, Proj. #27

Brief Title: CARBIDE AND OXIDE SPECIES

Investigators: Alan W. Searcy

Scope of Work

An investigation of the nature and stabilities of the molecules produced by heating carbides and by heating metals in the presence of carbon is in progress. The studies completed for the aluminum-carbon system and for the silicon-carbon system make possible the predicition and understanding of the behavior of combinations of these materials when used in high temperature environments such as found in jet and rocket engines. The study of the molecules formed by thermal decomposition of silicon carbide is additionally helpful in elucidating the conditions most fruitful in preparing silicon carbide single crystals for production of high temperature transistors. An experimental survey to determine the importance of carbide molecules of other metals is revealing which other metal-carbon systems require intensive study.

A concurrent theoretical analysis has led to a new model for prediction of the shapes of molecules. This model appears significantly more successful than previous ones and should prove of great assistance in the problems of interpretation of spectroscopic data and of estimation of approximate thermodynamic data for molecules that have not been studied experimentally. The model suggests new interpretations of the kinetics of certain reactions, on the nature of dipole interactions and on the nature of hydrogen bonds.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1825

Brief Title: STANDARD FREE ENERGY OF FORMATION OF CERTAIN RARE EARTH CARBIDES

Investigators: C. L. McCabe

Scope of Work

This work is concerned with the determination of the Free Energy of Formation of rare earth carbides. Two attacks are being made on the problem. One is by the measurement of the pressure of the Rare Earth over the carbide in equilibrium with graphite. The other is the measurement of the CH_4/H_2 in equilibrium with the hydride of the Rare Earth and the carbide. The work also consists of determing the composition of the solid phases by x-ray and analytical techniques.

Contractor: (The) Franklin Institute Laboratories for Research and Development, Philadelphia, Pennsylvania

Contract: AT(30-1)-1484

Brief Title: STUDIES ON DIFFUSION IN METALS AND ALLOYS

Investigators: R. L. Smith

Scope of Work

This work is concerned with various problems of diffusion in solids. Currently, attention is directed into two categories: (a) Surface diffusion; and (b) Diffusion of components in defect alloys and compounds.

Surface diffusion studies are being conducted on metal (zinc) single crystals. Because of the importance of obtaining accurate concentration-distance determinations, a study of the limits of resolution and detectability of the interaction of Zn^{65} radiation with the photographic emulsion is being studied. This is done by observing apparent changes in the concentration contours as a function of the photographic variables, exposure and development time, temperature and film type. The effects of volume diffusion and surface preparation on surface diffusion are also being investigated. Finally, the effects of some physical and chemical conditions of the surface are being studied.

Diffusion studies of indium and tin into indium-tin alloys from zero to four percent indium are being carried out. This phase of the work includes a study of the self diffusion of tin in single crystals.

Contractor: Illinois, University of, Urbana, Illinois

Contract: AT(11-1)-67, Proj. No. 3

Brief Title: MECHANISM FOR SUBSTITUTIONAL DIFFUSION IN METALS

Investigators: Frederick Seitz and David Lazarus

Scope of Work

The program is concerned with experimental and theoretical studies of the fundamental mechanism for diffusion in metal systems. The experimental techniques involve precision sectioning methods using radioactive tracers, anelastic relaxation measurements, measurements of electrical resistivity, and methods for generating high hydrostatic pressures. To date, particular attention has been devoted to tracer diffusion measurements in the noble metals, copper, silver and gold, and their dilute binary solid solutions. Measurements have been extended recently to diffusion in concentrated solid-solutions, both with and without the presence of chemical concentration gradients.

Much of the data have been successfully interpreted in terms of a model which considers the electrostatic interactions between screened impurity atoms and solvent atoms. However, various singular exceptions have been found, particularly for the copper system. Measurements of the effect of isotopic mass on diffusion, still in progress, indicate strong correlations between motion of solvent and impurity atoms for faster-diffusing impurities. Work has begun on a study of the effects of high hydrostatic pressure on anelastic relaxation in alpha silver-zinc. Pressures of 10,000 atmospheres cause a four-fold increase in the relaxation time.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1975

Brief Title: EFFECTS OF DEFORMATION ON THE DIFFUSION PROCESS

Investigators: B. L. Averbach and M. Cohen

Scope of Work

This research is concerned with the measurement of self- and interdiffusion rates in metals during the application of deformation. Preliminary data have indicated that there is a considerable enhancement in the self-diffusion of iron during compressive creep. The diffusion under stress may be as much as one hundred times larger than the static diffusion coefficient. Quantitative relationships are being obtained in iron as a function of strain rate and temperature and it is expected that these data will help in a better understanding of the mechanisms of diffusion and the relationship of diffusion to creep. Similar studies on high-purity nickel and gold are also being undertaken.

Early results indicate that the activation energy for diffusion decreases with increasing strain rate and appears to reach a minimum value. If it is assumed that this minimum activation energy corresponds to the activation energy for migration, it is then possible to calculate the activation energy for vacancy formation and the equilibrium concentration of vacancies. Considerable experimental verification is required and this is now in process.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1888

Brief Title: ACTIVITIES IN LIQUID AND SOLID BINARY METAL SYSTEMS

Investigators: John F. Elliott

Scope of Work

The purpose of this research is to evaluate the thermodynamic activities, and related thermodynamic properties, of liquid and solid binary metal systems in the temperature range of 600 to 1100°C. The systems studied will usually contain lead, silver, or bismuth as one of the componenets. The experimental techniques to be used in the main are the electrode potential method and the method of distribution of a component between two phases.

In the last year the solubility of iron-nickel alloys in liquid lead between 700 and 1100°C has been measured. From this work the activities of iron and nickel in the iron-nickel binary were computed. A surprisingly large negative deviation from Raoulation behavior for nickel is indicated. A technical paper reporting this work is now being completed. Some of the phase relationships in the iron-nickel-lead ternary system and the iron-nickel-lead-carbon quaternary system between 1350 and 1550°C have also been measured. These results are being prepared for publication. The activity of manganese in the liquid region of the manganese-lead-bismuth ternary system has been determined with the cell:

$$Mn \;\big|\; Mn^{++}\ (KCl - MnCl_2) \;\big|\; Mn \text{ (in liquid Mn-Pb-Bi)}$$

Some rechecking of the data has been found to be necessary.

Considerable experimental difficulty has been encountered in measuring the activity of aluminum in luquid aluminum-silver alloys with the cell:

$$Al \;\big|\; Al^{+++}\ (KCl + NaCl) \;\big|\; Al \text{ (in liquid Al-Mg)}$$

The cell is now functioning properly and the system is being completed. The next step is to determine with this cell the activity of aluminum and the phase relationships in the ternary aluminum-lead-silver system between 700 to 900°C.

Exploratory work is being started for the development of a satisfactory electrode potential cell for the study of activities in liquid lithium alloys. An electrolyte of solid LiCl is being developed. An apparently anomalous behavior of manganese when precipitated from manganese-bismuth solutions is being studied with electrode potential measurements.

Work also is being initiated on the influence of added elements on the solubility of nickel from iron-nickel alloys in liquid lead. If progress in the work is satisfactory other solvents will also be studied.

Contractor: Michigan, University of, Ann Arbor, Michigan

Contract: AT(11-1)-543

Brief Title: INVESTIGATION OF THERMODYNAMIC ACTIVITIES IN BISMUTH SYSTEMS

Investigators: David V. Ragone and Edward E. Hucke

Scope of Work

The object of this investigation is the determination of thermodynamic properties of bismuth alloys (in particular U-Bi alloys) through the determination of bismuth vapor pressure. The pressure is being determined by a method involving absorption of characteristic radiation by the bismuth vapor. This method was chosen because of the presence of both Bi and Bi_2 species in the vapor phase.

The equipment currently being used includes an absorption cell, furnace and vacuum equipment, and a spectrograph. Measurements are to be made in the U-Bi system in the range 700-900°C. Thermodynamic activities, and partial molal enthalpies and entropies will be calculated for both components. Work will be extended into ternary systems involving U-Bi and one other constituent.

Contractor: North Carolina, University of, Chapel Hill, North Carolina

Contract: AT(40-1)-2036

Brief Title: RESEARCH IN INTERMETALLIC DIFFUSION

Investigators: Lawrence M. Slifkin

Scope of Work

Radioactive tracers will be utilized in a study of the fundamental interactions involved in the mobility of metal atoms in metal crystals. One problem being investigated is the effects of large chemical concentration gradients on the diffusion process. Studies of the diffusion of both silver and gold tracers in solid solutions of silver and gold are under investigation. The silver-gold system was chosen because it is as close to an ideal solution (thermodynamically) as may be obtained.

Another problem being studied is the anomalous diffusion of rare earth tracers in such metals as silver and lead. It appears that the insoluble rare earths are very strongly segregated at boundaries and dislocations, even when present at concentrations below 10^{-6}. The possible use of such behavior in studying dislocations will be considered.

Thirdly, it is hoped to initiate diffusion measurements in several alpha solutions of silver, in order to provide tracer activation energy data to compare with anelastic relaxation activation energies being determined by Dr. Thomas Turner at Wake Forest College.

Contractor: Pennsylvania, University of, Philadelphia, Pennsylvania

Contract: AT(30-1)-1879

Brief Title: EFFECT OF TORSIONAL STRAINS ON SELF-DIFFUSION IN SILVER

Investigators: R. Maddin

Scope of Work

This research is concerned with the influence of plastic stress and strain on the self-diffusion coefficient in single crystals of silver. Currently, the effort is devoted to a study of the effect of torsional strain and strain rate on diffusion.

Cylindrical single crystals of Ag(99.99), 1/2 inch in diameter, grown by the Bridgman method, were plated with Ag^{110}. The crystals were twisted to a constant total surface strain during the idffusion-anneal at temperatures of 600, 700, 750 and 800°C as a function of the rate of straining. The diffusivities were measured by sectioning and counting.

The ratio of the self-diffusion while straining occurred (D_s) to the self-diffusion at no strain (D_u) varied linearly with rate of twisting and attained values of greater than 100 for the rates used. The enhancement of diffusion by straining was inversely proportional to the temperature at which diffusion occurred. The activation energy for diffusion (Q_D), determined from the data plotted as a function of the rate of twisting decreased from 44,000 cal/mol for zero strain, to an asymptetic value of 17,000 cal/mol for strain rates of $20 \times 10^{-5} sec^{-1}$. The activation energy for motion of vacancies Q_M was considered to be 16,000 cal/mol and that for formation Q_F was 28,000 cal/mol. The experimental data permitted the calculation of N_V, the concentration of defects in equilibrium at steady-state (equal to the thermal equilibrium number of vacancies plus those produced by deformation less the number lost due to annihilation). Analysis, as first suggested by Cohen[1] based upon the generation of vacancies produced by the deformation will be discussed in detail in a publication.

Based upon these results the research being continued is aimed at observing the effect at very low rates of strain, i.e., $1 \times 10^{-7} sec^{-1}$. In view of recent results by Balluffi[2], an additional variable to be considered is the total strain. Our previous results were obtained with a constant surface strain of 0.91, and, consequently, an attempt to determine the effect of strains for surface strains less than 0.91 will be made.

(1) M. Cohen - private communication.
(2) R. Balluffi - private communication.

Contractor: Pennsylvania, University of, Philadelphia, Pennsylvania

Contract: AT(30-1)-1976

Brief Title: VAPOR PRESSURES OF THE CARBIDES OF REACTIVE METALS

Investigators: Nev A. Gokcen and Shiro Fujishiro

Scope of Work

Vapor pressure of the carbide of titanium is being investigated at present by means of a graphite effusion cell in the range of 1800°C to 2200°C. A satisfactory cell made of very high density graphite, has been developed for this purpose. The experimental procedure has been perfected to minimize errors resulting from the weight loss of graphite cell and the effusion through the joints and the pores.

The molecular species in the gaseous phase is monatomic titanium, as determined by the complete vaporization of known amounts of TiC.

Pure titanium, melted in graphite crucibles in the range of 1900°C to 2400°C indicates that the stoichiometric composition of the carbide corresponds is TiC.

The data will be used in determining the thermodynamic properties such as the free energy, enthalpy and entropy of formation of titanium carbide.

Similar investigations are planned for Zr, Hf, V, Nb and Ta, and possibly for Cr, Mo and W.

Contractor: Pittsburgh, University of, Pittsburgh, Pennsylvania

Contract: AT(30-1)-647

Brief Title: THERMODYNAMICS OF ALLOY FORMATION

Investigators: **W. E. Wallace**

Scope of Work

This research has as an ultimate goal the understanding of the nature of bonding forces acting between atoms in solids and the interpretation of structure, energetics of processes and properties in terms of these forces. The investigation, conceived originally as a study of the thermodynamics of alloy systems, has been broadened in scope to include structural studies by x-ray diffraction and neutron diffraction, kinetics of some of the reactions studied and magnetic susceptibility determinations.

The current program is divided into two general areas - (1) determination of the residual entropies of selected binary systems, and, (2) studies of metal-hydrogen systems. The systems involved in the first category are (a) Ta_2H, (b) V_2H, (c) Zr_2H_3 and (d) KCl-KBr solid solutions. Room temperature entropies of the KCl-KBr system have been obtained by a cycle involving the ternary aqueous system and these are being reduced to $0^{\circ}K$ by appropriate heat capacity determinations. A similar procedure is used with the metal-hydrogen system except that the starting point is the entropy determined by vapor pressure measurements at temperatures in the range 200 to $400^{\circ}C$.

Other studies of metal-hydrogen systems include (a) determinations of the activation energy for interstitial diffusion of hydrogen in Ta, Nb and V by internal friction methods, (b) attempts to locate the hydrogen atoms in Ta_2H by neutron diffractions techniques, (c) crystallographic alterations associated with the $55^{\circ}K$ thermal anomaly in Pd_2H, (d) the effect of interstitial oxygen and nitrogen on the vapor pressure of hydrogen over the Ta-H system and (e) determination of the limits of primary solubility in the V-H system.

In addition to the above studies the kinetics of disordering of MgCd are being studied calorimetrically at temperatures between 210 and $300^{\circ}K$.

Contractor: Rensselaer Polytechnic Institute, Troy, New York

Contract: AT(30-1)-1044

Brief Title: ANISOTROPIC DIFFUSION

Investigators: H. B. Huntington

Scope of Work

This research is concerned with the general problem of studying diffusion in anisotropic lattices as a contribution to the continued development of the theory of diffusion in materials. Information on the crystallographic dependence of the rate of self-diffusion in single crystals of metals and semi-metals is obtained. In addition, experiments to determine the influence of high electrical currents on atom mobility are pursued to investigate the interaction of electrons with defect atoms in the process of moving from one lattice position to another.

During the past year, substantial progress was made in these studies. Self-diffusion studies in indium were presented as a doctoral thesis and were submitted for publication. An investigation of the diffusion of radioactive cadmium in a gold-cadmium alloy is nearing completion. Observations are being interpreted in terms of mechanisms postulated to explain the results; additional work is planned to explore the influence of gold-cadmium alloy composition and also to study the diffusion of a gold isotope. An investigation of the self-diffusion of antimony continues to be plagued with experimental difficulties, due principally to: (a) experimental brittleness of the single crystals, making specimen preparation difficult and (b) inability to grow crystals of proper orientation to determine diffusion parallel to the trigonal axis. It has been possible, however, to prepare specimens to study diffusion in the trigonal plane and these investigations are now in progress.

Probably the most interesting development in the past year was the establishment of definite marker motion under the influence of high current density in pure gold wires. An interpretation of these results is now being made and it is planned to present the work as a paper in the near future. The study will be extended to include work on copper.

It is planned to initiate new experimental work to study impurity diffusion in anisotropic lattices. First experiments will study the directional dependence of diffusion of radioactive silver in zinc.

Contractor: Sylvania Electric Products, Inc., Bayside, New York

Contract: AT(30-1)-GEN-366

Brief Title: FUNDAMENTALS OF SINTERING OF METALS AND OXIDES

Investigators: L. L. Seigle, L. S. Castleman, and J. Brett

Scope of Work

The objective of this research project is to clarify the details of mass transport occurring during the sintering of metals, oxides, and metal-oxide mixtures. Attention has been focused initially on a study of the rate of growth of interwire welds, and rate of void elimination in fine wire compacts of Cu, Ni, Fe and other metals at elevated temperatures, in order to confirm the Herring-Nabarro mechanism. Although self-diffsuion appears to be the predominant mass transport process occurring during the sintering of these non-volatile metals, a variety of diffusion paths are of importance under various circumstances. Direct evidence of the importance of grain boundaries for void elimination in Cu and Ni has been obtained, but the data suggest that voids may shrink even in the absence of grain boundaries. An experimental effort is under way to study the sintering of porous metal compacts in which grain boundaries are absent, to determine if shrinkage occurs under these conditions.

In the future, it is intended to investigate the sintering of oxides and metal-oxide mixtures to ascertain the rate-controlling processes and effect of microstructure.

Contractor: Tennessee, University of, Knoxville, Tennessee

Contract: AT(40-1)-1068

Brief Title: APPLICATION OF HIGH TEMPERATURE ADIABETIC CALORIMETRY TO METAL SYSTEMS

Investigators: E. E. Stansbury

Scope of Work

This research project is concerned with the application of abiabatic calorimetry to metal systems over the temperature range 50-1000°C. The method permits determination of specific heats, heats of transformation, heats of solution in the solid state, and heat effects associated with non-equilbrium states. Measurements are made on bulk samples and an accuracy better than one per cent is generally realized.

The objectives of the research are to provide thermo-dynamic data of general applicability and specific data for theroretical studies of kinetics of phase transformations and of alloy formation.

Current investigations have been directed toward zirconium and zirconium base alloys. Preliminary data have been obtained on Zr, Zr-Ti, Zr-Ag, Zr-In, Zr-Nb and Zr-H alloys. Measurements in these systems are continuing with particular emphasis on Zr-Nb and Zr-Ti alloys. A calorimetric investigation of the formation of Ni_4Mo from the alpha solid solution is being initiated.

Contractor: Wake Forest College, Winston-Salem, North Carolina

Contract: AT(40-1)-2413

Brief Title: STUDY OF ATOMIC MOVEMENTS IN SOLIDS EMPLOYING ANELASTIC MEASUREMENTS

Investigator: Thomas J. Turner

Scope of Work

It is anticipated that a significant contribution will be made to an understanding of stress induced ordering and stress relaxation across grain boundaries by a careful correlation of anelastic and diffusion measurements on a number of systems. This correlation will be made using a series of silver substitutional systems, including Ag-Cd, Ag-In, Ag-Sn and Ag-Sb.

Relaxation times of the order of 1000 seconds will be determined from elastic after-effect measurements while those of the order of one second will be determined from torsion pendulum measurements. High frequency measurements, yielding relaxation times of the order of 10^{-5} seconds will be made by employing a resonant bar with an eddy current drive and an FM detection system. Thus, relaxation times measured over eight cycles of 10 will be obtained.

Because of the limited solubility of tin and antimony in silver, it may not be possible to detect an ordering peak in these systems; however, one should be able to measure grain boundary relaxation times.

These data will be compared with tracer diffusion studies which will be made on the same systems by Dr. Lawrence Slifkin and his group at the University of North Carolina.

Section II-C

LIQUID STATE PHENOMENA

Contractor: Arkansas, University of, Fayetteville, Arkansas

Contract: AT(40-1)-2096

Brief Title: STUDY OF THE STRUCTURE OF LIQUIDS BY X-RAY DIFFRACTION

Investigators: P. C. Sharrah and R. F. Kruh

Scope of Work

This investigation is concerned with structures in the liquid state (metals, glasses, and aqueous solutions of complex metal ions). The objective is to provide a basis for the correlation of properties of liquids with their structures. The structure of liquids will be determined by X-ray diffraction from a free liquid surface, using an experimental arrangement to permit intensity measurements of high precision.

An X-ray spectrometer has been constructed, aligned and put into operation. Measurement of the diffraction from low-melting metals, mercury, gallium, and indium, will be made as the high temperature specimen holder is being completed. When it is available, attention will be given to other metallic systems, including binary melts. Eventually the use of the instrument for study of complex ions in solutions, molten salt systems, and glasses will be considered.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1985

Brief Title: KINETICS OF REACTIONS BETWEEN LIQUID ALLOYS AND MOLTEN SALTS

Investigators: Thomas B. King

Scope of Work

The work under this contract is intended to elucidate the mechanism of reactions between a metallic alloy and an ionic liquid, such as a fused salt, which are in contact along a phase boundary. In such systems displacement reactions, for example, consist of transport steps, by diffusion and convection within the two liquids, and reaction at the phase boundary.

It can be shown that if overall transport control prevails and the transport of only one species is rate-controlling, the rate constant if proportional to the square root of the diffusion coefficient of that species. Rate studies and measurements of diffusion coefficients to establish this are being made on the general type of system,

M_1 (in alloy) + M_2^+(in halide) = M_1^+ (in halide) + M_2(in alloy)

In reactions involving gas evolution or reduction of a complex ion, overall rate control by the phase boundary reaction is possible. Rate measurements are being made on systems such as,

SiO_2(in silicate) + $\frac{4}{3}$ Al (in copper) = Si(in copper) + $\frac{2}{3}$ Al_2O_3 (in silicate)

under varying convection conditions. Electrolytic polarization techniques are also being instituted for study of such reactions.

Examples of mixed activation and transport control will also be examined under varying convection conditions. Tracer measurements will be necessary for some systems to establish with certainty that activation control is operative.

Contractor: Oklahoma, University of, Norman, Oklahoma

Contract: AT(40-1)-2409

Brief Title: DIFFUSION PHENOMENA IN LIQUID METALS

Investigators: W. R. Upthegrove

Scope of Work

The objective of this research is the study of diffusion phenomena in liquid metal systems to obtain an improved understanding of the effects of the system variables upon these phenomena and of the diffusion mechanisms involved. The initial phase of this study will be to evaluate the diffusion behavior of solute atoms in very dilute solutions.

The first system to be investigated will be the iron-mercury system. The solubility of iron does not exceed 1 ppm at temperatures below 700°C. This will necessitate the development of suitable experimental procedures for measuring the diffusion of solute atoms at these low concentrations. The analytical problems involved will be difficult, but should be amenable to sensitive radioisotope techniques. The diffusion measurements will be made by the capillary-reservoir technique.

The data of the iron-mercury system will be utilized to evaluate the present concepts of diffusion-controlled mass transfer in this system.

Application of the procedures developed during this study to other systems exhibiting very low solubilities are planned.

Contractor: Purdue University, Lafayette, Indiana

Contract: AT(11-1)-359

Brief Title: DIFFUSION IN LIQUID ALLOYS

Investigators: Richard E. Grace

Scope of Work

The long range purpose of this investigation is to determine rates and mechanisms of diffusion in liquid alloys. A variety of experiments are being carried out as follows:

(a) Determination of chemical and self-diffusion coefficients and their temperature coefficients in zinc amalgams and other low melting alloys by the capillary reservoir technique.

(b) Analysis of solution rates, diffusion processes and interfacial reactions at solid/liquid interfaces in low melting alloy systems. An evaluation of thin films operative at solid/liquid interfaces is planned.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: REACTIONS IN FUSED ELECTROLYTES

Investigators: G. P. Smith, C. R. Boston, J. J. McBride, and G. F. Petersen

Scope of Work

Scope I - Spectrophotometry of Fused Salts

Measurements are being made of the optical absorption spectra of fused salts. The purpose of these measurements is to determine the presence of complex ions and other light absorbing species in fused salt melts, and to determine the effect which ion neighbors have on the energy states of known light absorbing species.

Recently a study was made of chloronickel complexes dissolved in LiCl-KCl. Currently studies are being made of the effect of ion neighbors on the transition in the nitrate ion from the normal to the excited state.

Scope II - Nuclear Magnetic Resonances in Fused Salts

This study consists of a determination of the feasibility of measuring nuclear magnetic resonances in fused salts as a means of investigating chemical bonding and ion association. Measurements have been made of the proton and sodium resonances in fused sodium hydroxide and of the fluorine resonance in various fused fluroides.

Contractor: Yale University, New Haven, Connecticut

Contract: AT(30-1)-2029

Brief Title: STRUCTURE OF METALLIC LIQUIDS

Investigators: Robert B. Gordon

Scope of Work

Scope I - Ultrasonic Measurements

This research is concerned with the application of the methods of physical acoustics to the investigation of the structure of liquid metals and alloys. The experimentation involves the measurement of the velocity of sound as a function of temperature and composition in a series of alloy systems. The additional data required to calculate compressibilities is also being obtained.

Scope II - Galvanomagnetic Measurements

A study of the dependence of the Hall effect on the degree of order in solutions is being made. Solid solutions of gold in copper are now being investigated. Apparatus is being designed to extend the Hall constant measurements to liquid metals and alloys.

Contractor: Yale University, New Haven, Connecticut

Contract: AT(30-1)-1857

Brief Title: ELECTRICAL RESISTIVITY OF LIQUID SODIUM AND SODIUM ALLOYS

Investigators: William D. Robertson

Scope of Work

The present work concerning the electrical resistivity of liquid alkali metal solutions was undertaken to investigate size effects of solute atoms in the liquid state. When combined with previous work on the effect of solute charge it is felt the data obtained will provide a more comprehensive basis for a detailed understanding of the structure of liquid metals.

Since it is highly unlikely that the number of free electrons in monovalent metals changes on melting, the investigation is restricted to them. Sodium, in particular, was chosen as the solvent metal because of its simple structure, low melting point, and availability of data. Because of its high reactivity, however, a closed melting system has been perfected to make the alloys and protect them from oxidation. Since a glass conductivity cell is also prohibited by the reactivity of sodium solutions, a technique employing a stainless steel cell is employed.

Resistivity measurements, as a function of temperature and composition, have been completed on the following alloy systems: Na-Li, Na-K, Na-Cs, and Na-Rb. Preliminary analysis of the data indicates a direct dependence on the temperature and concentration of the solute and, also, on the size difference between the solute and solvent. A more complete analysis of the results is dependent on a chemical analysis currently being undertaken by the Knolls Atomic Laboratory.

Further work is now being done to investigate the effects of the so called "hard atoms", copper, silver and gold, on the resistance of Na and, in particular, to compare their behavior in changing the resistivity with the observed effects of the alkali solutes.

Section II-D

SURFACE PHENOMENA

Contractor: Cornell University, Ithaca, New York

Contract: AT(30-1)-1994

Brief Title: LIQUID-SOLID INTERFACIAL TENSIONS IN METAL ALLOY SYSTEMS

Investigators: C. W. Spencer

Scope of Work

This investigation is concerned with the penetration of liquid metals into the grain boundaries of solid metals. The effects of temperature, liquid composition, plastic deformation, and relative crystallographic orientation of neighboring crystals at the grain boundary are being explored.

It has been determined that the grain boundaries of a nickel base alloy, containing 0.22% Mn and 0.25% Si, are not completely penetrated by sulphur-nickel liquids but are completely penetrated by bismuth-nickel liquids. Measurement of the rate of bismuth-nickel liquid penetration as a function of temperature shows the activation energy for the process to be about 22,000 calories.

In the one-year period beginning June 1, 1958, the penetration of bismuth-copper liquids into copper grain boundaries is to be explored. Also the equipment and techniques utilized in the production of bicrystals possessing a particular orientation are to be perfected. A preliminary investigation of the effect of a small amount of plastic strain on the process is also planned.

Contractor: Harvard University, Cambridge, Massachusetts

Contract: AT(30-1)-1956

Brief Title: REACTIONS BETWEEN SOLIDS AND LIQUID METALS AND ALLOYS

Investigators: B. Chalmers

Scope of Work

There are three specific problems to which attention is being directed:

1. Theory

The theory of solid-liquid reactions is being extended to a further study of the detailed structure of the solid-liquid interface.

2. Crystal Growth

Dendritic growth in alloys is to be studied further; the effects of temperature and composition on the rate of growth and on the morphology will be studied, both experimentally and theoretically.

The imperfection content of crystals grown from dilute alloys will be studied in relation to the rate of growth and other parameters.

3. Effect of Liquid Alloys on Solid Metals

The attack of liquid gallium on grain boundaries of aluminum will be studied quantitatively; it is also intended to continue the study of the solution of metallic crystals in liquid alloys.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: REACTIONS AT METAL SURFACES

Investigators: J. V. Cathcart

Scope of Work

Scope I - Criteria for Protective Oxide Formation

Current theory suggests that the ratio of the volumes of equivalent amounts of a metal and its oxide determines the degree of protectiveness of the oxide. The inadequacy of this theory has been demonstrated by studies of the oxidation behavior of the alkali metals and certain of the refractory metals such as niobium and tantalum. It has been shown that the mode of diffusion during the oxidation process is probably at least as important as the volume ratio in determining the protectiveness of oxide films. These investigations are being continued with emphasis on electron optical studies of oxidation processes in an effort to understand the general conditions necessary for protective oxide formation.

Scope II - Radiation Effects on Oxidation

An investigation is in progress to study the changes induced in oxidation of niobium by radiation fields. Electron optical methods are being used to determine changes in the micro-topography of the oxide films. An effort will also be made to determine the rate of oxidation in the presence of a radiation field.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: CHEMICAL PROPERTIES OF METAL SURFACES

Investigators: L. H. Jenkins, F. W. Young, Jr.

Scope of Work

The purpose of this program is to understand the mechanism of chemical reactions at metal surfaces and to determine the effect of irradiation on these reactions. The rate of reaction on a metal surface is dependent on the crystal face which is reacting, and the further observation that the rate is not uniform over any one face has led to the investigation of the effect of imperfections in the crystal on surface reactions. This program includes investigations of gaseous oxidation, thermal etching in high vacuum, dissolution of metals in aqueous media, and observation of dislocations as etch pits. Particular emphasis is placed on determining the role of dislocations in the metal on these reactions.

Since dislocations can be observed as etch pits, the motion of dislocations in the crystal is studied using this technique. Also, the behavior of dislocations in copper crystals which have been subjected to irradiation is being investigated.

The effect of irradiation on the gaseous oxidation of metals is being studied.

Contractor: Virginia, University of, Charlottesville, Virginia

Contract: AT(40-1)-1768

Brief Title: GROWTH AND CHEMICAL PROPERTIES OF NEARLY PERFECT SINGLE CRYSTALS

Investigators: Allan T. Gwathmey

Scope of Work

Many crystals are known to grow under certain conditions by means of dislocations. It is therefore desirable to develop methods by which the number of dislocations in metals during growth can be controlled and reduced to a minimum. Such studies would not only give important information on the mechanism of growth, but they would make it possible to determine the influence of dislocations on the chemical and physical properties of crystals.

During the present period it will be directed primarily toward the further purification of copper nominally of 99.999% purity (and possibly of other metals) by means of zone refining techniques. This will comprise, at first, the development of procedures adapted to the use of induction heating equipment presently available in this laboratory. Single crystals will be grown from the processed metal. In these studies of growth the purpose is not only to prepare more nearly perfect crystals but also to understand better the growth characteristics under these conditions.

The crystals so prepared are to be examined by physical and chemical procedures now in use in this laboratory for study of chemical properties of metal crystals. In these studies the object will be to determine what relationships may exist between imperfections in the crystals, their chemical properties, the purity of the metal, and possibly the conditions of crystal growth. Such correlations would have considerable theoretical interest, and would hold possibilities of practical importance in many fields.

Section II-E

DEFINITION AND CONTROL OF STRUCTURE

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: ALLOY STRUCTURES AND PROPERTIES

Investigators: S. S. Sidhu, M. B. Nevitt, M. Mueller, A. Dwight and L. Heaton

Scope of Work

Scope I - X-ray and Neutron Diffraction Studies

A number of basic alloy systems are under study by use of diffraction techniques, from which determinations of crystal structure, lattice constants and neutron coherent scattering amplitudes may be made. These studies at present include the following topics:

(1) Structures of single crystals of Au_3Be and Au_2Be.

(2) Sigma Phases.

(3) Magnetic susceptibility and neutron diffraction of Fe-Os at low temperatures.

(4) Deuterides and hydrides of Li.

(5) Antiferromagnetic structures of synthetic $Fe_{1-x}S$, CrS and NiS.

(6) Neutron diffraction of Cs and Rb liquids.

(7) Antiferromagnetic structure of spinal type structures Fe_2O_3-ZrO-MnO_2.

Scope II - Occurrence of CsCl, Cr_3O, and Laves Type Phases

A survey of the occurrence of sigma, Cr_3O and CsCl type phases as a function of atomic size has been completed. A cryogenic magnetic apparatus has been completed and calibrated for the purpose of measuring the magnetic susceptibilities of some of these phases. The general objectives of this program are to elucidate the factors that dictate the occurrence and stabilities of these phases in transition metal systems. As part of this program, six Laves phases, three $TiNi_3$-type, and seven Ti_2Ni-type phases were discovered, lattice parameters were measured and selection rules in terms of atomic size and valence were sought.

Contractor: California, University of, Berkeley, California

Contract: AT(11-1)-34, Proj. 29

Brief Title: METAL DEFORMATION

Investigators: Earl R. Parker

Scope of Work

During the past year work has continued on fundamental studies related to:

1. Nature of High Temperature Creep.
2. The Principles and Processes of Solution Hardening.

New results have been obtained which show that the recognized effect of grain size on creep rate is not caused by the change of grain size per se, but due to the fact that the nature of grain boundaries changes when fine grain material is heated to a high temperature to increase the grain size. This may have important implications and so research along this line will be continued throughout the coming year. In regard to solution hardening, considerable progress has been made. A number of theories have been proposed to account for solution hardening, but the validity of these theories has never been checked by critical experiments. Experiments performed on this project have revealed for the first time the cause of solution hardening in single crystals. The next year's program will be concerned with extending this work to studies of the mechanism of solution hardening of polycrystalline alloys.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1826

Brief Title: FUNDAMENTAL STUDY OF THE EARLY STAGES OF SINTERING

Investigators: F. N. Rhines

Scope of Work

A topological model developed during the past year, describes the geometric changes that constitute sintering, without simplification with respect to the size or shape of the powder particles, or of their mode of packing. Based upon this model the first stage of sintering is identified as that during whcih the genus of the confining surface remains constant and equal to one more than the sum of all interparticle contacts minus the number of particles, in other words, the period prior to the beginning of closure of interparticle channels. Preliminary studies indicate that the density increases and the surface area decreases at a diminishing rate, but without interruption at the outset of sintering. The initial rate of densification appears to be propertional to the number of interparticle contacts. A creep-like process is thought responsible for the material transport involved in densification, because diffusion processes alone cannot provide the kind of transport required. It is planned to further test the predictions of the topological model, in order to establish more firmly the findings of the preliminary studies. It is hoped also to extend the methods of quantitative metallography to the measurement of more of the topological parameters of the sintering body.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1310

Brief Title: MECHANICAL PROPERTIES OF METALS AT LOW TEMPERATURES

Investigators: W. A. Backofen and R. L. Fleischer

Scope of Work

The purpose of this study is to characterize the processes operative during the low temperature plastic deformation of face centered cubic metal crystals and to establish mechanisms for the processes consistent with present understanding of the solid state. Particular attention is devoted to the tension stressing of single crystal, bi-crystal and polycrystalline specimens to examine the behavior of grain boundaries at 300°, 78° and 4.2°K in three types of observations:

(1) Low temperature deformation mechanisms as revealed by deformation markings and correlated with stress-strain relations.

(2) Grain boundary effects as manifested by crystal interactions at interfacial misfits.

(3) Crystal orientation dependence of strain hardening.

Polycrystalline specimens of aluminum, copper and lead and single and bi-crystal specimens of aluminum have been studied.

Low temperature tensile deformation of aluminum is being continued with the intent of further examining the nature of work hardening: (1) Relations between single crystal and polycrystal deformation are being approached by comparison of polycrystal behavior with that of single crystals oriented for single as well as multiple slip. Tensile tests will provide data to strains at which necking occurs and to temperatures as low as 4.2°K, (2) Creep properties at 4.2° are also of interest. Preliminary results will be explored more extensively, and, (3) Finally, the two values obtained of the stage II work-hardening slope at 4.2° will be studied further with single and bi-crystal stress-strain determinations and more sensitive x-ray examination.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1973

Brief Title: DEFORMATION AND FRACTURE OF MATERIALS AT ELEVATED TEMPERATURES

Investigators: Nicholas J. Grant

Scope of Work

This project concerns a study of deformation and fracture of materials at elevated temperatures embracing the following aspects: (1) a quantitative study of the contribution of grain boundary sliding to total elongation in hot creep; (2) the mechanism of intercrystalline cracking during creep; and, (3) the relationship of recrystallization temperature to high temperature strength.

It has been shown that prior deformation, up to about eight percent, has an exceedingly small effect on the creep rupture behavior of aluminum and solid solution aluminum alloys when tested at 500° F, with the prior deformation taking place at temperatures slightly above or slightly below this test temperature. These results appear to be contrary to those reported for nickel. Using a scribed grid pattern, with a spacing of 0.2 mm, measurements have been made on aluminum and solid solution Al-Mg alloys, in the temperature range 410 to 940°F, of the contribution of grain boundary sliding to the total elongation. Since the results are significantly different than those reported in the literature, they are of particular interest. A careful mathematical treatment and precise measurements would appear to indicate that the current results are more nearly correct. The grain boundary contribution to total deformation has been measured as a function of, composition, strain, and temperature.

In the extension of the work, the effects of strain rate, grain size, and still wider variations in composition will be evaluated. In addition, an extremely close examination is being made of the changes taking place in the grain boundary proper. Progressive serration of the grain boundaries has been observed, and these structural changes are being followed carefully in relationship to the creep curve.

Contractor: Nuclear Metals, Inc., Cambridge, Massacusetts

Contract: AT(30-1)-1565

Brief Title: DEFORMATION AND FRACTURE PROCESSES IN BERYLLIUM

Investigators: S. H. Gelles

Scope of Work

This program is aimed at determining the factors which are important in producing brittle fracture in beryllium. In particular, the effects of impurities and of substructure (dislocation networks, mosaic structure, twins and deformation markings) will be studied. The techniques to be used for carrying out this work will be microradiography, whereby mosaics and segregation of impurities on the scale of 1 micron and above will be studied; automicroradiography, in which possible impurities will be activated and would reveal their positions on a fine resolution photographic plate; fractography studies, in which the beryllium fracture surfaces will be studied metallographically and with the aid of the electron-microscope and etch-pit studies to determine the distribtuion of dislocations and the dilocation-impurity interactions.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

Contract: AT(30-1)-1565

Brief Title: ATOM MOVEMENTS IN TWINNING

Investigators: E. J. Rapperport

Scope of Work

An attempt will be made to infer the paths taken by various atoms during mechanical twinning of a hexagonal close packed alloy. The particular experimental information sought is the change in positions of the two species of atoms in ordered Mg_3Cd crystals upon twinning. Ordered samples of Mg_3Cd, with known positions of the magnesium and cadmium atoms, will be subjected to various stresses in attempts to induce mechanical twins. Twins, so produced, will be examined with x-rays to determine the state of order and the relative positions of the magnesium and cadmium atoms within the twin. It is expected that a knowledge of the positions of the magnesium and cadmium atoms before and after twinning will help in inferring the atom motions during twinning. The alloy Mg_3Cd was selected as a first choice because, in addition to being an ordering alloy, it is hexagonal close packed and occurs in an alloy system of complete solid solubility. There are, however, other ordering alloys which may also be considered in the study outlined above.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

Contract: AT(30-1)-1565

Brief Title: DEFORMATION PROCESSES IN ZIRCONIUM

Investigators: E. J. Rapperport

Scope of Work

This investigation seeks to determine the deformation modes operative in zirconium single crystals as a function of temperature and of oxygen content. The active modes for pure zirconium have been determined at 78°K, at 300°K, and at 575°K; the work is now being extended to 1075°K and to zirconium-oxygen alloys.

Such knowledge helps the understanding of deformation textures of zirconium and their variation with temperature and oxygen content. This metal affords an opportunity to observe a large number of twin systems in a single lattice, and so may also yield information on the process of twinning. (At room temperature there is one family of slip planes, and four families of twin planes.)

Plans for continued study include a determination of crystallographic directions in zirconium crystals, and to find the twin elements of $11\bar{2}1$ twins by means of electron microscopy.

Contractor: Pennsylvania State University, University Park, Pennsylvania

Contract: AT(30-1)-1516

Brief Title: SINGLE CRYSTAL NEUTRON DIFFRACTION ANALYSES

Investigators: Ray Pepinsky

Scope of Work

This is a program of single crystal neutron diffraction analyses, with neutron diffraction observations accomplished at the Brookhaven National Laboratory, but with chemical and crystal preparations, and preliminary x-ray studies carried out at Penn State, and computations on X-RAC and S-FAC and by IBM computing machines available for use by the Penn State Laboratory.

The research involved in this activity is in part related to solid state research conducted in the Penn State Laboratory, and in part is concerned with problems of interest to the Radiation Damage group at Brookhaven. In the latter category is the study of the lead azide structure; and in the former class are neutron-diffraction studies of crystal transition mechanisms (which cannot be solved unequivocally by x-ray or other methods), hydrogen-bonding studies, and, other problems involving the location of light atoms in the presence of higher-atomic-number elements. The program also includes instrumental developments associated with these studies.

Contractor: Pennsylvania, University of, Philadelphia, Pennsylvania

Contract: AT(30-1)-1893

Brief Title: THE EFFECT OF STRESS ON RECOVERY

Investigators: Norman Brown

Scope of Work

Recovery is defined as the decay of the effects of plastic deformation prior to recrystallization. The changes in structure associated with recovery are generally sub-microscopic in nature. The purpose of this work is to determine the effects of an applied stress during recovery and also, the effect of various types of plastic deformation on recovery.

Currently, the investigation is concerned with single crystals deformed in simple shear so that one slip system is activated. Strains are measured with a sensitivity of 10^{-6} by means of a capacitance-type gage. Since recovery is associated with a strain recovery of about 10^{-3} to 10^{-4}, which may be associated with an unpiling of dislocations from a barrier, stress will be applied during recovery in order to balance the unpiling. Previous work indicated that a negative strain both unpiles and annihilates dislocations. The annihilation process will be studied by the micro-strain technique. Mechanical hysteresis is associated with the piling and unpiling of dislocations. The amount of hystersis as a function of stress amplitude will also be investigated. Polycrystalline metals will be used to supplement the single crystal results.

Section II-F

PHYSICAL CERAMICS

Contractor: California, University of, Berkeley, California

Contract: AT(11-1)-34, Proj. #7

Brief Title: METALLIC CERAMIC BOND

Investigators: J. A. Pask

Scope of Work

The purpose of this research is to obtain information on the interfacial forces between molten liquid glass and metal at elevated temperatures as an aid in better understanding the bonding of metal to glass or a ceramic. The interfacial forces are being interpreted on the basis of measurements of contact angles between molten glass and metal under various conditions (wetting characteristics).

Combinations of a simple glass composition $Na_2O - 2SiO_2$, and gold, platinum, and iron were used. Contact angles were measured in vacuum, air, oxygen, nitrogen, hydrogen, argon, water vapor, carbon monoxide, and carbon dioxide. The nature of the atmosphere had no effect on the contact angle for gold. The angle on platinum, however, was definitely influenced: water vapor, carbon monoxide, and hydrogen absorb on the surface of the metal increasing the angle from 22° (vacuum) to 60°; oxygen reaches the interface between the metal and glass by diffusion through the metal as well as absorbing on the surface, decreasing the angle to 15°; and the inert gases have no effect on the contact angle. The iron-glass system is currently being investigated.

Contractor: Georgia Institute of Technology, Atlanta, Georgia

Contract: AT(40-1)-2420

Brief Title: MECHANISM AND ACTIVATION ENERGY STUDIES FOR DIFFUSION THROUGH SINGLE CRYSTAL AND POLYCRYSTALLINE HIGH TEMPERATURE MATERIALS

Investigators: Willis E. Moody, Jr.

Scope of Work

The principal objective of the program is to advance, in a systematic manner, the basic scientific knowledge of imperfections and mass transport phenomena in high-temperature ceramic, metallic oxide, materials by means of diffusion studies. A subsidiary objective is the collection of diffusion data.

The materials selected for the diffusion studies are polycrystalline and single crystals of UO_2 and Al_2O_3 which have cubic and hexagonal structures respectively. The inert gases, He, Ne, A, Kr, and Xe, were selected primarily to obtain information concerning diffusion as a function of atom size without the complications of valence. It is planned to determine and to relate the activation energy for diffusion to the mechanism of diffusion and associated imperfections. The accomplishments during the first year will be largely contingent upon establishment of successful analytical techniques for small quantities of inert gases. The studies for A and Kr should be completed, in addition to working out analytical techniques.

Future work would be the completion of the systematic study by inclusion of single valent, divalent, and higher series of ions in addition to the inert gases. The results obtained with the different series and crystal types should lead to generalized theory of mass transport and associated phenomena at elevated temperatures.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1852

Brief Title: BASIC RESEARCH IN CERAMICS

Investigators: W. D. Kingery and F. H. Norton

Scope of Work

This research is designed to investigate fundamental factors affecting the formation, properties, and uses of ceramics. That effort is concentrated particularly on studies relating to: (1) atom movements in ceramics and kinetics of ceramic processes, (2) surface and wetting behavior in ceramics as related to fabrication and use, (3) development of microstructure in ceramics, (4) effects of structure on electrical and mechanical properties in ceramics, and (5) thermal properties, particularly heat conduction, in ceramics. Major effort is devoted to items (1), (3), and (4).

Contractor: Materials Research Corporation, Yonkers, New York

Contract: AT(30-1)-2178

Brief Title: STUDY OF GRAIN BOUNDARY PHENOMENA IN CERAMIC MATERIALS

Investigators: Josef Intrater and Sheldon Weinig

Scope of Work

The objective of this investigation is to determine the magnitude of the grain boundary effects in the plastic behavior of ceramic materials. An understanding of the atomistic mechanism of the boundary influence on the flow properties of ceramic materials and their control is sought.

The initial phase in this investigation will consist of studies of both single and bi-crystal specimens of the identical high purity MgO and Al_2O_3. The magnitude of applied shear stress required to produce a specific amount of shear strain will be measured as a function of test temperature. Bi-crystals of MgO have been prepared successfully and experimental study of this material is progressing.

Contractor: Utah, University of, Salt Lake City, Utah

Contract: AT(11-1)-82, Project No. 9

Brief Title: RECRYSTALLIZATION AND SINTERING OF OXIDES

Investigators: Ivan B. Cutler

Scope of Work

The objective of this research is a greater understanding of the mechanism of vacancy migration in oxides. Aluminum oxide will receive the major emphasis. Vacancy forming additives will be utilized to assist the various investigations. Electrical conductivity will be pursued to differentiate between the ionic and electronic regions of conductivity. Direct current as well as alternating current measurements will be made.

Diffusion constants measured from shrinkage of powdered compacts will be utilized along with conductivity determinations to differentiate between cation vacancy, anion vacancy, and vacancy pair migration. Previous work has shown the effect of nucleation on recrystallization. It is planned to measure the rate of nuclei growth in the various crystallographic directions. This will be done with oriented nuclei in powder compacts.

Section III

INTERACTION OF RADIATION WITH MATTER

III-A

CRYSTAL DEFECTS AND MECHANISMS OF INTERACTION

Contractor: Atomics International, A Division of North American Aviation, Inc., Canoga Park, California

Contract: AT(11-1)-GEN-8

Brief Title: CRYSTAL DEFECTS AND MECHANISMS OF DAMAGE

Investigators: A. Sosin and J. A. Brinkman

Scope of Work

The purpose of this study is to identify the crystalline defects produced by irradiation, mechanical working, and thermal treatment, and to describe their annealing kinetics and the interactions which occur between various defects.

Recent work on this project typical of the proposed future efforts includes the following:

A. A study of the recovery of electrical resistivity in cold-worked gold, copper, and nickel between $4^{\circ}K$ and $100^{\circ}K$. The absence of sharp recovery near $30^{\circ}K$ (Stage I) indicates a different distribution of point defects from that observed after irradiation.

B. A detailed study of the recovery of electrical resistivity in nickel above room temperature. A recovery stage near $100^{\circ}C$ (Stage III) was found after cold work and electron irradiation. The kinetics (second order for irradiation, diffusion to infinite-capacity sinks, such as dislocations, for cold work) and activation energy (1.05 ± 0.05 ev) were determined. A recovery stage was also found near $260^{\circ}C$ (Stage IV) following cold work but not irradiation. The kinetics were similar to Stage III. The recovery behavior in nickel closely parallels previously observed behavior in copper, allowing a migrating defect assignment to be made.

C. The release in the range from $20^{\circ}X$ to $60^{\circ}X$ of energy stored in copper by irradiation below $20^{\circ}X$ was measured. The energy release observed (about 5 ev per interstitial-vacancy pair) compares favorably with theoretical estimates. The spectrum of energy release as a function of temperature is similar to the results reported for electrical resistivity recovery.

Future effort will be concentrated on low temperature irradiations correlated with investigation of defect migration over the entire temperature range in which recovery is observed.

Contractor: Brookhaven National Laboratory, Associated Universities, Inc., Upton, Long Island, New York

Contract: AT(30-2)-Gen-16

Brief Title: RADIATION EFFECTS

Investigators: G. J. Dienes, G. H. Vineyard, A. Damask, L. Porter, D. Keating, P. W. Levy, A. Goland, D. Rosenblatt, R. Hatcher, and J. Gibson

Scope of Work

One of the major research activities in solid state physics is the study of defects in crystals. Radiation effects and other departures from perfect periodicity are under investigation with many diverse techniques.

Scope I - Neutron Transmission Experiments

An investigation has been carried out of the effect of reactor irradiation on single crystals of alpha-Al_2O_3, primarily a continuation of the use of long-wavelength neutron transmission for determining the concentration and types of defects produced in solids by high energy particles. The aluminum oxide exhibited crystallographic stability to fast neutron irradiation at temperatures less than 40°C, and the results indicate a total number of defects approximately 40 times less than that predicted by current theories. Correlation of the transmission with the macroscopic density changes was good. The fact that the fractional change in density was always greater than the fraction of defects present suggests that the lattice relaxation is outward around a vacancy as well as around an interstitial. Annealing of the material produced no decrease in the concentration of defects from room temperature to 400°C, a steady decrease from 400°C to 1250°C and irregular changes beyond 1250°C. Annealing at 1800°C did not remove the coloring, although the density returned to its preirradiation value.

With the completion of the study of alpha-aluminum oxide, attention has been turned toward a similar investigation of beryllium. Plans have been made to correlate changes in the resistivity with changes in the neutron transmission of the identical beryllium samples as a function of irradiation dose. This experiment, as well as many others, requires low temperature irradiation. The new low-temperature irradiation facility is nearing completion.

Scope II - Optical Properties of Insulators

The neutron transmission experiments on Al_2O_3 are closely related to the optical studies on this material. One of the important color centers in alpha-Al_2O_3 absorbs at 2040 A. It has been shown previously that this band is formed by reactor irradiation but not by purely ionizing radiation. By careful annealing studies it has now been shown that upon heating the crystal the 2040 A band is removed in two stages. The first, or low temperature stage, appears to be finished when the crystal is heated to approximately 300°C. The second or high temperature component is not completely removed until temperatures in excess of 1000°C are reached. It is interesting to correlate this result with the measure-

ments of radiation damage in Al_2O_3 made by the long wavelength neutron transmission technique.

These neutron transmission measurements indicate that radiation damage does not begin to anneal out until the sample is heated to 340°C which is close to the temperature at which the second annealing stage begins. The tentative conclusion is that the high temperature component represents removal of defects while the low temperature component is related to an undetermined electronic process (rearrangement of electrons). The activation energies associated with the first component are about 1 ev while 1.5 to 2.5 are obtained for the second group.

In another series of experiments on alpha-Al_2O_3 samples were heated near their melting point and cooled in a controlled manner. Simply heating alpha-Al_2O_3 for several hours at 1800°C removes all of the absorption present in the samples when received from the manufacturer, in the range 2000 to 10,000 A. Since this treatment can increase the transmission at 2000 A from approximately 50% to that given by the Fresnel reflection loss it is a useful and practical way of improving the UV transmission of Al_2O_3 windows. Subsequent exposure of the heat treated samples to ionizing radiation restores the coloring to the original level. Another result of these high temperature experiments is that the defect responsible for the 2040 A band may be present, to a small extent, in unirradiated samples.

These high temperature experiments will continue with particular emphasis on the possibility of "quenching in" defects. Annealing measurements on the radiation induced color centers in very pure fused silica gave similar results. However, the fused silica data is extremely laborious to analyze, since the bands overlap considerably and it may be some time before a reasonable understanding is obtained.

Scope III - X-ray Studies

Heavily irradiated diamond has been investigated further both by x-ray techniques and mechanical measurements. The heavily irradiated structure is highly strained, causing the interference effects to be weakened and producing an x-ray pattern of an amorphous-like structure. Diffraction data are complete and the integral Fourier inversion for the radial distribution of the number of atoms between (r) and (r + dr) is being computed for three diamond samples of lesser damage. No sudden onset of the amorphous like structure is evident, but a gradual degradation of the interference effects with increasing irradiation is observed. For small irradiations the x-ray reflections are shifted indicating a lattice expansion in agreement with density measurements, and the intensities are attenuated with increasing scattering angle. For moderate irradiations the peak shifts are more pronounced, a diffuse intensity peaking near the undamaged line position builds up and the reflections at large scattering anles are replaced by broad diffuse peaks. The hydrostatic density is characteristically less than that determined from the lattice expansions. For heavy irradiation the entire pattern is diffuse.

The mechanical properties of irradiated diamond have been investigated by measuring the amount of wear of neutron irradiated diamond against silicon carbide abrasive and comparing it with that for unirradiated diamonds. The volume removed from diamond phonograph needles was calculated geometrically from photomicrographs of four groups of six diamonds irradiated with different total fluxes. For an irradiation of 2.6×10^{18} NVT (fast) the hardness was 50% of the unirradiated hardness, and for 7.8×10^{20} NVT (fast) the hardness was 20%. The densities were measured by flotation in warm thallium malonate solution to an accuracy of about $\pm$ 0.7%. For the 2.6×10^{18} NVT irradiation the density has decreased by 1% and by $\pm$ 4% for the 7.8×10^{20} NVT irradiation. The decrease of coherent energy of an irradiated diamond was estimated crudely from the number of broken bonds and the strain energy of the density change. The calculated fractional decrease in cohesive energy is about one-half of the fractional decrease in hardness. Thus, diamond becomes softer on irradiation contrary to the response of most metals. In the case of metals dislocation motion is held up by the radiation induced defects. Thus, the hardness of daimond is apparently controlled by the cohesive energy, which decreases upon irradiation as indicated above rather than by dislocation motion.

Scope IV - Effect of Irradiation on Solid State Reactions

Very little work has been done on the dynamics of the production of crystalline defects by radiation at temperatures where the defects tend to disappear by various annealing mechanisms, such as direct recombination of vacancies and interstitials and migration to dislocations. Simultaneous production and annealing processes can now be studied by the technique of radiation enhanced diffusion. The phenomenon of enhanced diffusion in the alpha-brass system has been studied in a quantitative way. A simple theory of radiation enhanced diffusion has been worked out which describes the dependence of this enhancement on flux and temperature under steady state conditions. The theoretical study also shows that the measurement of enhanced diffusion as a function of temperature can indicate the mechanism by which defects are removed from the lattice. The enhanced diffusion rate during irradiation in the Brookhaven reactor has been measured in alpha brass at several temperatures in the range from 0°C to 190°C. This enhancement is independent of temperature in the range from 0°C to 150°C, in excellent agreement with the theoretical predictions for the case when the radiation induced defects finally disappear at internal surfaces. The data from 150°C to 190°C also follow very accurately the predictions of this simple theory. This new technique will be applied to a number of problems.

A related study is concerned with the effect of radiation on the martensite transformation in an iron-nickel (25%Ni) alloy. A high temperature irradiation produces resistivity changes which are indicative of phase separation, probably by means of enhanced diffusion. Irradiation at lower temperatures inhibits the subsequent martensite transformation as well as produces some transformation during irradiation. The data show that a neutron exposure of 4×10^{17} NVT

epicadmium lowers the M_s temperature (martensite start temperature) in this alloy by approximately 6°C. The decrease in M_s is believed to be due to the same type of damage which raises the critical shear stress, since it is known that the M_s temperature is structure sensitive. A low exposure experiment indicated that the effect is not due to a change in elastic modulus (via dislocation pinning) since such changes should saturate below 10^{13} NVT and since there was no change in M_s for such an exposure.

Scope V - Chemical Effects of Irradiation

The cooperative program with Picatinny Arsenal continues. This effort is primarily concerned with obtaining a fundamental understanding of the effects of radiation on explosives and related materials. One aspect of this program, the study of the thermal decomposition kinetics of lead styphnate monohydrate (an explosive used in detonators) which has been subjected to gamma-ray and reactor irradiations has been concluded. Cobalt^{60} gamma-ray irradiations up to 1.8×10^8 r did not significantly alter the thermal decomposition curves. However, material subjected to reactor irradiations decomposed as much as 3 times as fast as unirradiated material and the increase in rate was roughly proportional to the irradiation. The activation energy for the decomposition reaction was not changed by the irradiations. One concludes that the irradiation has increased the number of decomposition nuclei but did not alter the decomposition mechanism.

Studies of the color centers formed in the azides by radiation continue although this work is seriously hampered by our inability to grow really good crystals routinely. Several new crystal growing techniques show considerable promise but need to be developed further. A large amount of data on the coloring of potassium azide by gamma-rays at room temperature has been analyzed. Most of the observed absorption bands are probably color centers although one broad band might be caused by colloidal potassium which aggregates in the crystal during irradiation.

The x-ray study of radiation effects in sodium azide has been completed. Stacking faults are produced by mechanical deformation, gamma-irradiations, and reactor irradiations. The azide molecule is decomposed (three molecular decompositions per 100 ev or energy absorbed) by ionizing radiation. The local stresses produced by decomposition produce dislocations and associated stacking faluts similar to those produced by mechanical deformation. Annealing studies show recovery of the damaged state with return to stoichiometry of the irradiated crystal. Calculations of the energy absorbed in this material through ionization from x-rays, gamma-rays of the Co^{60} source, and from the various ionizing reactions during reactor irradiation are in substantial agreement with the observed effectiveness of these radiative sources in producing damage in sodium azide.

Some further experiments have been done on the increase in the oxidation rate of graphite by nuclear radiation. Two variables, impurity content and heat treatment of the graphite, had not been studied in detail in previous experiments. Experiments have now been completed on unirradiated and irradiated graphite annealed at 600°C and 2800°C and subsequently oxidized at 300°C. Annealing did not affect

the oxidation rate of unirradiated graphite indicating that the impurities in unirradiated graphite do not cause catalytic burning: the impurity content was found to decrease during annealing at 2800°C from about 0.1% to about .001% without altering the combustion rate. Irradiated samples annealed at 600°C burned at about the same rate as unannealed irradiated graphite, but after annealing at 2800°C they burned at the preirradiation rate. Thus, the radiation effect was removed by annealing between 600°C and 2800°C.

Scope VI - Theory

The effect of the regularity of atomic arrangement on the production of displaced atoms by irradiation of crystals has been considered. Because of a tendency for energy to be "focussed" down closely packed lines of atoms it is predicted that displacements may be somewhat harder to make than had been contemplated in conventional theories, and the consequences of this have been worked out semiquantitatively. A calculation of the details of the displacement process with the aid of a large electronic computer is under way.

It has been suggested some time ago by Varley at Harwell that neutral interstitial halogen atoms may be produced by ionizing radiation in the alkali halides. There is some support for this idea based on optical and magnetic resonance experiments. A theoretical investigation of the properties of interstitial halogen has been started based on the classical treatment of an ionic crystal. In particular, the most stable configuration of an interstitial and the energy barrier for its diffusion are being determined. Preliminary results indicate that the chlorine interstitial in KCl resides in one of the cube faces and that the neighboring chlorine ions have suffered a large displacement. This configuration, obtained by minimizing the energy of the system (Coulombic, polarization and repulsive terms), is in reasonable agreement with the available experiments.

Contractor: Brown University, Providence, Rhode Island

Contract: AT(30-1)-1772

Brief Title: STUDY OF RADIATION EFFECTS IN SOLIDS

Investigators: Rohn Truell

Scope of Work

Changes in the physical characteristics of solids can be observed in a very sensitive way by high frequency ultrasonic methods where irradiation induced defects are used to produce these changes. The changes in question may be of several types, such as those arising when cobalt gamma irradiation of alkali halide materials produces ionization which in turn produces point defects capable of pinning dislocations. The examination and study of these effects as a function of deformation of the sample, temperature, frequency, and irradiation rate has shown that the high frequency dislocation damping theory appears to account for the observed behavior. In addition the velocity dispersion behavior together with the irradiation experiments shows that the L^2 - L^4 (dislocation loop length behavior of velocity and attenuation measurements is confirmed in the low frequency region (5-15 mc/sec) as it should be.

In contrast with the dislocation damping effects and the associated changes as the result of irradiation, there are fast neutron damage effects which produce ultrasonic velocity and attenuation changes of a different type with the consequence that dislocation damping effects can be distinguished from scattering effects in radiation experiments on solids. Details of theory and experiment are being investigated for both scattering and dislocation damping effects.

Highly sensitive measurements of fractional velocity changes with temperature (made by an ultrasonic interference method) are being used to determine among other things how small an irradiation flux can be determined under various conditions. In addition data for $\frac{1}{v}\left(\frac{dv}{dT}\right)$(velocity v, temperature T) as a function of frequency from ten to two hundred megacycles gives new information about elastic constants. Such measurements made with compressional and transverse waves before and after irradiation yield additional information about irradiation defects.

Sizes of damaged regions (regions of strain) have been obtained from a combination of velocity and attenuation measurements. Directional bombardment effects will be continued again when the facilities are rebuilt.

Materials are being studied

Contractor: Brown University, Providence, Rhode Island

Contract: AT(30-1)-1880

Brief Title: RADIATION DAMAGE STUDIES IN SOLIDS; NUCLEAR RESONANCE ABSORPTION TECHNIQUE

Investigators: P. J. Bray

Scope of Work

Nuclear magnetic resonance techniques have now been employed successfully to study radiation damage in single crystals of NaI, KI, LiI and KBr. These investigations at room temperature of neutron-irradiated crystals will be extended to cesium and rubidium halides. Investigations at liquid nitrogen temperature will be carried out on metal powders and alkali halide crystals which have received neutron or high energy electron radiation. The latter irradiation is particularly sought in order that damage known to be simple Frenkel defects can be studied. Annealing behavior of the damage in these materials will be studied as a function of temperature.

Investigations of the structure of boron carbide and glasses containing boron, lithium or sodium will be continued and studies will be made of neutron-irradiated specimens. The structure of boron and its behavior under neutron irradiation will be investigated.

Effects of neutron and gamma irradiation on molecular solids containing hydrogen, nitrogen, aluminum or chlorine will be studied with nuclear magnetic resonance and pure quadrupole resonance techniques. It is anticipated that the latter method will be employed for an investigation of neutron radiation damage in some selected metals.

Contractor: Brown University, Providence, Rhode Island

Contract: AT(30-1)-2024

Brief Title: RADIATION DAMAGE STUDIES USING THE TECHNIQUES OF ELECTRON-SPIN PARAMAGNETIC RESONANCE

Investigators: P. J. Bray and A. O. Williams, Jr.

Scope of Work

Electron-spin resonance techniques will be used to study radiation damage in alkali halides, glasses, sodium azide, and metals.

The studies of alkali halides will be concentrated on specimens irradiated at low temperatures with subsequent selective bleaching by F-band or other light. R_1, M and various V centers will be studied and attention will be given to the formation of colloidal metal by large radiation doses.

Boron-containing and alkali silicate glasses will be studied to determine the amount and location of impurities and to determine the environment of radiation-induced color centers. Silica systems subjected to neutron irradiation will receive attention.

The investigation of sodium azide will atempt to identify the origin of the color bands found in irradiated specimens of the material.

An attempt will be made, in very pure specimens at very low temperatures, to locate the conduction electron-spin resonance in metals of such high atomic number that very broad lines are to be expected. Studies of paramagnetic solute ions in metals are anticipated.

Much of the electron-spin resonance work mentioned here will be supplemented by nuclear magnetic resonance investigations and optical studies.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1828

Brief Title: RESEARCH IN THE DEFECT NATURE OF SOLIDS

Investigators: R. Smoluchowski

Scope of Work

This project aims at the development of sensitive methods of examining the imperfections that can be introduced into crystal lattices by, for example, deformation, irradiation, and thermal treatments, and at the application of such methods to the quantitative study of imperfections in particular materials. The principal techniques to use and under development are those of small angle x-ray scattering studies and fine-focus studies of individual Bragg reflections.

Scope I - Introduction and Effects of Lattice Imperfections

Single crystals of germanium have been plastically bent and analyzed with the convergent beam technique. The platelets are bent about an axis parallel to the long direction at a temperature of 700°C. For the case of bending that produces pure edge type dislocations an etch pit count can be made so as to verify the dislocation density. Thus far good correlation has not been obtained. Density of dislocations has been determined by studying the structure of monochromatic diffraction spots of deformed single crystals of super pure aluminum (99.9999+%). The density of dislocations as function of distance from the surface and its change during annealing has been observed. Speculation has been made as to the mechanism of deformation and the relative amounts of screw and edge components have been estimated.

The electronic stabilization and filtering of the high voltage power supply for the low angle x-ray scattering apparatus has been essentially completed. Tests have shown that there is a 120 cycle ripple of only 25 volts on 40 kv d.c., and the d.c. voltage level is constant to about 1 volt in 2000. The new system should produce nearly ten time the x-ray intensity when operating at its full potential of 50 kv. This will be used to study small angle scattering as function of temperature and deformation.

Isothermal annealing studies of ionic conductivity of gamma and proton irradiated alkali and silver halides will be continued in order to obtain activation energies for the various stages of annealing. A comparison of the influence of corpuscular and ionizing radiation will be made. Parallel measurements of density changes will be made including the influence of optical bleaching. Growth curves for V and F centers will be measured on KCl in order to identify the mechanism of defect formation. Correlation of growth curves in LiF with dislocation count will be made. Photoelastic measurements at low temperatures will be made with the hope of finding evidence of interstitials. Nu-

clear resonance studies of irradiated alkali halides will be made: It is hoped to obtain information about distance between defects and size of clusters. Work is to be continued in search for an evidence of thermal spikes and displacement spikes in irradiated Ni-Cu alloys by means of magnetic susceptibility measurements. It is planned to adapt the internal damping apparatus to a study of irradiation effects.

Scope II - Irradiation Effects on Surface Reactions of Metals

We plan to make isothermal conductivity measurements on irradiated NaCl in the temperature range 100-200°C as an extension of the work done thus far. The purpose of this would be to clarify the mechanism responsible for the drop of resistivity in this temperature range. Isothermal annealing studies of density of irradiated NaCl will be undertaken with a view to clarify the annealing mechanism in the range of 180-400°C. A study will be made of the growth of color centers in KCl crystals in order to determine the oscillator strength of positive ion vacancies. Studies will be continued on the apparent diffusion of defects out of the range of irradiation by x-rays. In an effort to further clarify the mechanism of annealing of volume expansion in LiF at low temperatures, this annealing will be compared with color center annealing in other alkali halides than LiF. This program may require an extension of the temperature range to liquid helium. Behavior of free carriers in alkali halides will be made by means of photoconductive studies. A study of the influence of irradiation on magnetic properties of copper-nickel will be continued. Small angle x-ray scattering of dislocation configurations in metal singel crystals will be made. Study of the influence of irradiation on decomposition of formic acid on irradiated copper-nickel polycrystalline and single crystal sheets will be continued. A field omission microscope study of irradiation effects will be continued.

Contractor: Cornell University, Ithaca, New York

Contract: AT(30-1)-2150

Brief Title: THE STUDY OF SOLIDS BY ELECTRON RESONANCE TECHNIQUES

Investigators: Robert H. Silsbee

Scope of Work

The purpose of this research program is to apply electron spin resonance techniques to the study of solids. The emphasis in the initial phase of the program will be on the study of relaxation times of paramagnetic imperfections in solids. The principal aims of the first year of the program are the construction of a versatile spin resonance spectrometer, using balanced bolometer detection and a cryostat to provide temperature between liquid helium temperature and room temperature, and, the conduction of exploratory experiments on two problems. One involves the study of the dipolar interaction between defects in neutron irradiated quarts which should give information about the uniformity of the defect density in the material. The other problem involves the study of the relaxation times of impurities and F-centers in M_gO, with particular emphasis on the effects on the relaxation behavior of the interactions between centers.

Contractor: (The) Franklin Institute Laboratories for Research and Development, Philadelphia 3, Pennsylvania

Contract: AT(30-1)-2002

Brief Title: A STUDY OF RADIATION EFFECTS IN SOLIDS BY ELECTRON MICROSCOPY

Investigators: H. G. F. Wilsdorf and I. G. Greenfield

Scope of Work

Lattice distortions which have been produced by neutrons and alpha-particles are being studied in two ways:

Scope I: Radiation Damage In Thin Metal Foils

An attempt is being made to observe "spikes" in thin metal foils directly with the electron microscope. The spikes, which according to theory are between 30Å and 100Å in diameter, should be visible due to diffraction effects that produce a distinct image contrast between the disturbed and undistrubed lattice. The optimum conditions for the production of suitable copper and nickel foils as well as for the obersvation of lattice defects in them have been established. The irradiation of foils with pile neutrons and alpha-particles is now in progress. Also, the behavior of dislocations in thin metal foils during elongation has been observed in the electron microscope and will be investigated in both irradiated and unirradiated foils.

Scope II: Investigation Of Slip Lines On Deformed Irradiated Crystals

Slip lines on deformed irradiated copper crystals, as observed by Blewitt and co-workers (Phys. Rev. 86, 641 (1952) with the light microscope, have shown a similarity with those on alpha-brass. Studies with the electron microscope reveal that the slip steps on irradiated copper are indeed deeper and that more cross-slip is observable than noted in untreated copper crystals of the same orientation and at the same elongation. However, a marked difference exists between slip lines on alpha-brass and on irradiated copper. While the lines on the former appear in pronounced clusters, they are more uniformly distributed on irradiated copper crystals. Stress strain data will be correlated with different slip line structures.

Contractor: Illinois, University of, Urbana, Illinois

Contract: AT(11-1)-182

Brief Title: RESEARCH ON RADIATION DAMAGE

Investigators: F. Seitz and J. S. Koehler

Scope of Work

The research aims to determine the nature and number of lattice imperfections introduced into crystalline solids by nuclear irradiation at liquid helium temperature. In addition, it would like to discover the nature of the various annealing processes which occur as the temperature of the specimen is increased. The activation energy or spectrum of activation energies associated with the motion of each particular lattice defect also constitutes a portion of the desired information. The project also aims to determine the nature and magnitude of the changes in physical properties which result from a given concentration of a particular kind of lattice defect.

The defects which are believed to be of importance are interstitial atoms, lattice vacancies, crowdions, small inclusions of another solid phase, and possibly precipitates of interstitial atoms. The properties investigated are resistivity, volume, lattice parameter, and stored energy. The crystals examined are copper, silver, gold, iron, nickel, cobalt, Ga Sb, In Sb, germanium, and various alloys.

The research at the University of Illinois on the nature of the defects introduced into copper, silver and gold at 10°K by cyclotron irradiation has used measurements of resistivity, length change and lattice parameter. This work, together with theoretical calculations, tends to support the following conclusions.

(a) The damage consists of clusters of lattice vacancies and interstitial atoms plus close vacancy interstitial pairs.

(b) The interstitial atoms are able to move about at temperatures of 36°K and above in copper. The vacancies can only move at much higher temperatures (i.e., about 300°K).

(c) During annealing interstitials can recombine with lattice vacancies.

(d) They can also form clusters of interstitial atoms.

Experimentally about 60% of the damage produced by 10 Mev. deuterons anneals out on warming to 60°K.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-858

Brief Title: X-RAY STUDY OF RADIATION DAMAGE

Investigators: B. E. Warren

Scope of Work

The goal of this project is the development and utilization of x-ray diffraction methods for the study of randomness, imperfections and damage in materials. Cold work distortion in metals is a very important type of imperfection. In the last two or three years, it has become evident that faulting plays a far more important role in cold work than had previously been suspected. Measurements of deformation faulting and twin faulting for a few FCC and BCC metals have already been made. Current effort involves the process of rounding out the theoretical development of the effect of different kinds of faulting on the x-ray diffraction pattern, and of finding the simplest and most reliable methods for obtaining fault probabilities from the x-ray patterns. Additional measurements on a number of metals cold worked in various ways at various temperatures will be made. Coupled with the measurements already available, it is expected to have sufficiently extensive data to correlate the importance and kind of faulting in the different metals.

Contractor: Northwestern University, Evanston, Illinois

Contract: AT(11-1)-89, Proj. No. 11

Brief Title: A STUDY OF RADIATION DAMAGE RESULTING FROM ELECTRON BOMBARDMENT

Investigators: John W. Kauffman

Scope of Work

The purpose of this work is to study the effect of radiation on the physical properties of metals. Through the proper choice of experimental procedures it is hoped to separate effects and determine concentrations of the various types of lattice imperfections resulting from the radiation as well to study interactions between them and to account for the kinetics of the recovery process of the irradiation induced damage.

Modification of a Van de Graaff generator has been completed and either electrons as protons are now available up to approximately 5 Mev. The main irradiations will be carried out with the specimens at the temperature of liquid helium. Using electrical methods, threshold energies and the subsequent annealing at higher temperatures are to be determined. X-ray diffraction and elastic constant measurements will also be used to separate effects of imperfections such as vacancies and interstitials and to determine concentrations. These studies can be grouped into low energy electron bombardment, near the threshold energy, where the damage is of least complexity and higher energy irradiation where the effects should begin to become more complex.

The metals used initially will be (1) the noble metals because of their simplicity and the fact that they allow, and have been subjected to, theoretical calculations and (2) the transition metals iron, cobalt, and nickel which contain the three typical crystal structures (i.e., b.c.c., hex.c.p., and f.c.c.) in order to study the effects of crystal structure.

Contractor: Purdue University, Lafayette, Indiana

Contract: AT(11-1)-125

Brief Title: BASIC RADIATION DAMAGE STUDIES

Investigators: H. M. James

Scope of Work

This research is concerned with a fundamental study of the theory of solids with emphasis being placed on the electrical, magnetic, and thermal properties of elemental and compound semiconductors. Studies are conducted on the character and concentration of defects introduced by electron bombardment in the energy range 150 Kev to 6 Mev; the range 150 Kev to 1.5 Mev now being available as the result of the completion during this past year of a low energy linear accelerator. Plans for continued study are summarized in the discussion below.

Radiation damage studies on semiconductors of the diamond-type structure will be continued with electrons of various energies with the goal to establish (a) a minimum threshold necessary for displacement in the lattice, (b) whether this threshold depends on direction and the current used, and, (c) whether various thresholds may be observed in selected polyatomic materials.

Through the use of a newly constructed cryostat, allowing bombardment at liquid helium temperatures, maximum removal rate in degenerate samples will be determined. It will also be determined whether the annealing procedure which has been established by bombardment with alpha particles at liquid helium temperatures can be duplicated with electron bombardment.

A particular attempt will be made to establish a definite energy threshold at these very low temperatures where annealing does not take place, as indicated in our experiments which have been carried out in this temperature range with alpha particles.

Contractor: Rensselaer Polytechnic Institute, Troy, New York

Contract: AT(30-1)-1995

Brief Title: RADIATION DAMAGE IN SOLIDS

Investigators: E. Brown

Scope of Work

Theoretical research is being carried out to ascertain the nature of defect production and thermal anneal in metals. The major effort is to determine the sensitivity of the predicted results on the parameters which are used in the various models.

Numerical calculations have been made for defect production using a screened coulomb interaction between atoms instead of the usual hard sphere approximation. With this model the production of defect pairs is very nearly linear with the energy of the primary particle. This holds true for a variety of models for the energy dependence of the escape probability of an atom from its lattice site. The agreement with the hard sphere approximation is good for the usual threshold models but deviates if one assumes there are only a few directions in which an atom can leave its normal site.

Future plans include a calculation of the activation energy for interstitial diffusion using variable parameters for the range and strength of atomic interactions. This may help clarify some questions on the problem of thermal anneal of damage. In line with this, an attempt will be made to make a detailed calculation on the diffusion recombination problem.

Contractor: Rutgers University, New Brunswick, New Jersey

Contract: AT(30-1)-1730

Brief Title: FUNDAMENTAL STUDY OF RADIATION DAMAGE BY MEANS OF SPECIAL X-RAY DIFFRACTION METHODS

Investigators: J. J. Slade, Jr. and S. Weissmann

Scope of Work

The experimental x-ray studies on radiation damage will cope with essentially two major problems. The first problem is the elucidation of the type of defects introduced in single crystals by irradiation with fast particles. The second problem will center around the study of precision lattice parameter measurements and density determination of irradiated covalent, ionic and insulator crystals; simultaneously, anisotropic lattice strains and lattice distortions will be investigated by special x-ray diffraction methods.

A. Nature of Lattice Defects after Irradiation of Single Crystals by Fast Particles

Systematic line profile studies of various (hkl) reflections will be undertaken by means of the x-ray double-crystal diffractometer. It is hoped that from the analysis of the line profiles it will be possible to decide whether the radiation damage introduced is caused by Frenkel defects, clusters of vacanceis or interstitials, or by generation of dislocations. In case of superposition of all these defects it may be possible to give the percentage of these defects present.

B. Precision Lattice Parameter Measurements, Density Determination and Anisotropic Strain Studies

It is proposed to carry out precision lattice parameter measurements, x-ray density determinations and anisotropic strain studies by means of the "divergent beam method." A special x-ray unit utilizing a capillary x-ray tube with electromagnetic focussing will be used. This tube was developed by Dr. T. Imura, who has recently joined the staff of this laboratory. This special tube permits irradiation of the single-crystal specimen with a strong x-ray beam possessing a divergence of nearly 180°. Consequently, various reflections can be simultaneously studied. From the geometry of the Kossel-line patterns, the lattice parameters can be determined with a precision of nearly 1:100,000 and anisotropic structural defects can be conveniently investigated. From the observed changes of the lattice parameters, the density change is easily computed.

Contractor: Sylvania Electric Products, Inc., Bayside, New York

Contract: AT(30-1)-GEN-366

Brief Title: MECHANISM OF DIMENSIONAL INSTABILITY OF URANIUM

Investigators: L. L. Seigle, L. S. Castleman, R. Resnick

Scope of Work

The objective of this project is to clarify the mechanism responsible for the dimensional instability of uranium under neutron bombardment. It is generally agreed that the growth of alpha-uranium under irradiation is somehow related to the anisotropy of its crystal structure, and two hypotheses have been proposed: (1) that anisotropic deformation under the action of thermally generated internal stresses is the governing factor; and, (2) that the anisotropic diffusion of vacancies and interstitials to grain boundaries and other sinks results in the observed growth. It is the purpose of the present program to check the validity of the diffusional theory by experimental verification of certain of its predictions. Accordingly, work is under way to determine the extent to which the principal diffusion coefficients in alpha-uranium are anisotropic. The diffusion couples are being prepared by evaporating U^{233} isotope and depositing it as a thin layer on properly oriented surfaces of alpha-uranium single crystals. In addition, "perfect" and imperfect single crystals and polycrystalline samples of alpha-uranium are being prepared for irradiation tests to determine if controlled variations in the number, kind and distribution of vacancy and interstitial sinks affect irradiation-induced growth in a measurable way.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: RADIATION EFFECTS IN SEMICONDUCTORS

Investigators: J. H. Crawford, Jr., J. W. Cleland, D. K. Stevens, H. C. Schweinler, and E. Sonder

Scope of Work

There are two intimately related phases of this program. The first concerns a detailed study of the nature of radiation-induced lattice disorder and specific differences that result from different types of radiation, i.e., fast neutrons vs gamma-rays or fast electrons. The second phase relates to the defect-energy level structure of the individual defects. The latter objective is not only essential from the standpoint of relating electrical property changes to the nature and distribution of radiation-induced defects in the lattice but, equally important, it holds intrinsic interest for those concerned with the electronic structure of semiconductors and insulators containing lattice defects.

Attention has been confined primarily to those semiconductors whose electrical behavior and band structure have been extensively investigated, including primarily the diamond type elements and compounds Ge, Si, InSb, InAs and GaSb. Studies of the rate of defect introduction and the subsequent annealing behavior are being studied after exposure to both fast neutrons and Co^{60} gamma-ray at both room temperature and low temperature ($78^{\circ}K$ and $20^{\circ}K$). Property measurements which serve as an index of the extent and type of lattice disorder include conductivity, Hall coefficient, optical absorption, magnetic susceptibility, minority carrier lifetime, and photoconductivity. A program devoted to effects of reactor irradiation on thermal conductivity and thermoelectric power of selected semiconductors is also being initiated.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: RADIATION EFFECTS IN METALS

Investigators: D. S. Billington, T. H. Blewitt, R. R. Coltman, D. K. Holmes, R. H. Kernohan, C. E. Klabunde, T. S. Noggle, V. K. Pare, J. K. Redman, D. O. Thompson, and M. S. Wechsler

Scope of Work

In fundamental studies of the nature of lattice disorder introduced by fast particle bombardment, it is necessary that all of the lattice damage be examined. Since it is known that a considerable portion of the damage in metals anneals well below room temperature, a comprehensive study requires irradiation at as low a temperature as possible to preserve for observation the thermally unstable lattice defects. Therefore, studies of the electrical and mechanical properties of pure metals and certain alloys are being performed in a gaseous-helium-cooled reactor-cryostat at temperatures near 20°K. This procedure not only permits an examination of the rate of defect introduction at a temperature where essentially all of the defects are preserved, but relaxation processes during warming according to a desired schedule are conveniently studied.

The rate of resistivity increase and the low temperature annealing spectrum of the enhanced resistivity in a number of metals have been studied in detail. Stored energy release measurements have also been made for copper single crystals. Mechanical property studies include measurements of internal friction and Young's Modulus in copper single crystals during irradiation at 20°K and subsequent warming. These studies have been extended to include tensile strength in copper.

Recently irradiation at 4°K has been achieved by means of an in-pile helium liquidifier. Development of an internal friction spectrometer has led to detailed examination of low temperature internal friction peaks arising from irradiation and cold working.

The effect of neutron irradiation-produced defects on the course of solid state reactions in alloys is being studied. The reactions studied include diffusion, precipitation, short and long range order. The interaction of irradiation-produced defects with other defects in metals, such as dislocations, impurities and quenched-in vacancies, is another area of importance in the program.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: RADIATION EFFECTS IN NON-CONDUCTORS

Investigators: J. H. Crawford, Jr., R. A. Weeks, C. M. Nelson, D. K. Stevens, and R. H. Silsbee

Scope of Work

It is well known that the crystal structure of certain insulating crystals is destroyed while that of others is little affected. The purpose of this research is to investigate the effect of radiation on the electronic structure and interatomic binding in non-conductors in order to better understand the influence of the binding type and radiochemical behavior on the radiation sensitivity of these materials. The optical and magnetic behavior of typical insulating crystals of both covalent and ionic type are being investigated after exposure to a variety of energetic radiation (X-rays, Co^{60} gamma-rays, Van de Graaff electrons and reactor radiation). Measurements of optical absorption, magnetic susceptibility, and paramagnetic resonance are being performed, and attempts are being made to correlate changes between these properties after exposure and various annealing treatments. The materials being investigated include the alkali halides, the various forms of silica (both crystalline and glass), magnesium oxide, corundum, rutile, and diamond. In the case of ionic crystals the ionic conductivity is being studied. Particular attention is being paid differences between the effect of the different types of radiation. Where possible, electronic changes are correlated with existing information on structural changes.

SECTION III-B

EFFECTS ON PHYSICAL AND MECHANICAL PROPERTIES

Contractor: Bausch & Lomb Optical Co., Rochester, New York

Contract: AT(30-1)-1312

Brief Title: IRRADIATION DAMAGE TO GLASS

Investigators: N. J. Kreidl

Scope of Work

The purpose of the present project is to investigate irradiation effects in glass especially to develop a theoretical background from which practical problems arising from exposure of glass to radiation can be considered. Theories have been derived for irradiation effects in glass based largely on analogies to the well-documented and simple alkali halide systems. Recently, work by British groups resulted in an analysis of some defects found in the silica system which form color centers under radiation. The techniques developed in the present program for determining the influence of trace impurities in silica by color change and thermoluminescence seems capable of extending this work and yielding valuable data on this fundamental glass forming system.

To bridge the gap between silica with trace amounts of impurities and complex silicate glasses, techniques will be investigated for the preparation of pure silica glass with controlled purity, silica glass with small additions of other glass constituents (alkali, alkaline earths, as well as transition elements), up to the normal glass compositions.

The quantitative action of cerium as a function of dose and concentration in preventing the normal coloration of glass will be investigated in an attempt to determine whether the action of cerium is a preventative one which saturates depending on concentration or a competitive one which merely reduces the probability of normal color center formation.

In the coming year it is expected to have high pressure (60,000 psi) high temperature (1000°C) available for investigation of the relation between the similar effects of pressure and neutron bombardment on the silica system. The change in irradiation properties with pressure treatment should elucidate the effect of structure on these phenomena.

Contractor: Canisius College, Buffalo, New York

Contract: AT(30-1)-1810

Brief Title: INVESTIGATIONS IN IRRADIATED VITREOUS SILICA

Investigators: Herman A. Szymanski

Scope of Work

Recently, completed experimental work includes the following:

(1) A step-anneal study of the neutron damage in vitreous silica. It has been possible to obtain the initial activation energy spectrum for this damage, to assign a reaction order of one and a frequency factor of $_{10}$-10 seconds

(2) Annealing of color centers in vitreous silica and aluminum oxide has been done and an analysis of the results has begun.

(3) Reflection infrared spectra has been developed as another tool to supplement these investigations.

A major objective of the following year's work is to extend the mathematical treatment developed for annealing vitreous silica to other physical properties of the silica as well as to other materials, such as corundum. The new methods of attack will include transient high pressures to produce density changes in vitreous silica, reflection infrared spectra as well as visible and ultraviolet spectra and the refinement of the annealing apparatus.

New work is also planned on surface studies which will include high temperature effects. Apparatus is being developed to begin this work. This work will be a combined effort of the physics and chemistry departments.

Contractor: Georgia, University of, Athens, Georgia

Contract: AT(40-1)-2418

Brief Title: INFRARED SPECTRA OF PLASTICS AND ELASTOMERS AFTER NUCLEAR IRRADIATION

Investigators: William C. Sears

Scope of Work

Purpose

The purpose of this research is to investigate the changes in molecular structure of polymers produced by nuclear irradiation. Infrared spectra of the polymers will be measured and interpreted before and after irradiation. A Beckman IR-4 spectrophotometer will be installed in the new physics building for this purpose about Janualy 1, 1959. Polymer films will be sealed in vacuum in quartz tubes for irradiation at the Oak Ridge National Laboratory.

Proposed Investigations

1. Dr. W. W. Parkinson, Jr. at ORNL will continue the analysis for double bonds in polybutadiene, GR-S, hevea and polystyrene which we have irradiated previously. Analysis of existing spectra of irradiated polyvinyl chloride, polyethylene terephthalate, Teflon and Nylon will require special techniques.

2. New polymers to be studied by the senior investigator include the following:

   (a) polyvinyl alcohol, polyacrylonitrile and copolymers, polyvinyl carbazole, polyvinyl pyridine, polyvinylidene chloride
   (b) polyvinyl formate, polyvinyl acetate, polyvinyl propionate, polyvinyl butyrate
   (c) polymethyl acrylate, polyethly acrylate, polypropyl acrylate, polybutyl acrylate, polymethyl methacrylate
   (d) polyvinyl formal, polyvinyl butyral
   (e) polyurethanes
   (f) neoprene, polyisobutylene, polybutene—1, poly -1,1' dihydrofluorobutyl acrylate
   (g) polychlorotrifluoroethylene

3. A study will be undertaken to determine the role of irradiation produced free radicals and unsaturated compounds in post-irradiation oxidation of polymer films.

Contractor: Hanford Atomic Products Operation, General Electric Company, Richland, Washington

Contract: W-31-109-Eng-52

Brief Title: INFLUENCE OF NEUTRON IRRADIATION ON STRUCTURAL METALS

Investigators: J. J. Cadwell and J. M. Tobin

Scope of Work

The purpose of this program is to study the influence of neutron irradiation on the physical and mechanical properties of structural metals in order to provide a basic correlation between fast flux and radiation effects.

Specimens of polycrystalline metals, representing the three major crystallographic systems, are being studied. These include copper, nickel, titanium, zirconium, iron, molybdenum, and Type 347 stainless steel. The extensive information available on flux conditions during specimen irradiation is unique to this work. Evaluation procedures will include the determination of mechanical properties, electrical resistivity, density, lattice parameters, and metallographic observations. The work will also include a study of the annealing kinetics of radiation damage.

Contractor: Illinois Institute of Technology, Chicago, Illinois

Contract: AT(11-1)-90, Proj. No. 9

Brief Title: INVESTIGATION OF ENERGY TRANSFER PROCESSES BY FLASH PHOTOLYSIS

Investigators: Leonard L. Grossweiner

Scope of Work

The purpose of this program is the application of flash spectroscopy to an investigation of fundamental mechanisms of photochemical energy transfer in certain condensed systems. The procedure consists of subjecting a substance to an intense light flash, and then unstable intermediates are identified from their optical absorption spectrum taken microseconds after irradiation. In the group of liquid systems to be studied, energy transfer is displayed in the quenching of their fluorescence by added halide ions or phenolic compounds. Flash irradiation will be absorbed by the fluorescent materials, after which the spectra will be examined for transient products of the added quencher. It is hoped that the path of excitation energy from chromophore to fluorogen to quencher can be traced.

The optical bleaching of coloration in x-ray irradiated alkali halide single crystals will be studied in a separate investigation, which exemplifies an important case of energy transfer in solids. Transient electrical and optical effects in systems of high bleaching efficiency, such as silver-doped potassium chloride, will be examined for evidence of intermediates between unbleached and fully bleached color centers. It is anticipated that the initial year of this program will be required for construction of the flash photolysis apparatus and scouting experiments for specific systems suitable for further study.

Contractor: Minnesota, University of, Minneapolis, Minnesota

Contract: AT(11-1)-532

Brief Title: EFFECTS OF ATOMIC RADIATION AND OF MOLECULAR ORIENTATION ON MECHANICAL BEHAVIOR OF LINEAR SOLID HIGH POLYMERS

Investigators: C. C. Hsiao

Scope of Work

Molecular orientation and fracture strength of linear polymers have been studied by obtaining stress-strain properties at room and low temperatures. Samples of amorphous and oriented polymers have been investigated. Both the amount of molecular orientation and sizes of the specimens have been shown to play a significant role in the observed variation in the fracture strength. Tentative mathematical analysis on the basis of a network of molecular domains have been made for polystyrene and polymethyl methacrylate.

During the coming year, it is planned to explore the effects of thermal neutron and gamma irradiation on certain uniaxially and biaxially oriented nylon and polyethylene terephthalate specimens. In addition to studying the changes in static mechanical properties, it is hoped also to obtain some structural information of polymer samples in both the irradiated and non-irradiated conditions. Apparatus for making mechanical measurements over a broad low temperature range will be procured. Some mathematical investigation of wave propagation in visco-elastic medium will be attempted so as to allow more correlation between certain dynamic quantities and structural information. Efforts will also be devoted to developing a theoretical analysis of the effects of atomic irradiation and of molecular orientation on the mechanical behavior of linear high polymers.

Contractor: Nebraska, University of, Lincoln, Nebraska

Contract: AT(11-1)-525

Brief Title: STUDIES OF IMPERFECTIONS IN SOLIDS

Investigators: Edgar A. Pearlstein

Scope of Work

Experimental work is proceeding on: (1) Radiation effects on the electrical resistivity of metals; and, (2) Studies of stored energy in colored alkali halide crystals.

Apparatus has been constructed and tested for the irradiation of thin metal foils in the University of Nebraska Cockcroft-Walton accelerator, at room or liquid air temperature. Early experiments on copper at room temperature show an easily measurable change in resistance. Work will continue on the dependence of the radiation effect upon the energy and mass of the bombarding particles.

It is expected that the method used will also enable measurement of the ranges of low energy particles.

A double calorimeter is being constructed for measuring the heat of solution of alkali halide crystals which have been x-rayed, bleached, heat-treated, etc. Overall sensitivity will be such as to detect a concentration of less than 10^{16} F centers/cm^3.

Contractor: Pennsylvania State University, University Park, Pennyslvania

Contract: AT(30-1)-1858

Brief Title: EFFECT OF RADIATION ON DYNAMIC PROPERTIES OF HIGH POLYMERS

Investigators: John A. Sauer

Scope of Work

Transitions in polymers have been studied by observation of dynamic mechanical properties and nuclear magnetic resonance line shapes over a broad temperature range beginning at $77^{\circ}K$. Samples of amorphous and partly crystalline polymers have been investigated. Both the monomer composition and the configurational structure of the polymer chains have been shown to play a significant part in the observed relaxation processes. Tentative identifications in terms of molecular movements have been made for all of the observed transitions in both polyethylene and nylon. Tests made on irradiated samples indicate that irradiation is a useful tool for altering the crystalline-amorphous ratio and for studying the effects produced by primary crosslinks.

During the last year, the influence of pure gamma radiation on the mechanical relaxation spectra of polyethlene specimens was examined over the temperature range from $80^{\circ}K$ to $400^{\circ}K$ and the results compared with those on similar specimens subject to pile irradiation. On the basis of comparable alteration of the mechanical relaxation spectrum, the pure gamma dose of 10^{9} r/hr corresponded approximately to a reactor does of about 5×10^{17} nvt. However, the gamma radiation had surprisingly little effect on the crystallinity content. To explore the question further of the relative effects of gamma rays and pile radiation on crystallinity content and on crosslinking, it is proposed to study the properties of gamma irradiated atactic and isotactic polypropylene and of high-density polyethylene.

The influence of side branches on main chain mobility will also receive further study. Deuterated samples of polypropylene and polymethyl methacrylate will be examined by NMR techniques and various poly-a-olefins will be studied by both NMR and mechanical measurements. It is also expected to extend our temperature range for studying molecular and chain mobility to liquid helium temperatures and to extend our frequence range from 5 kcs to 20 MCS using both resonance techniques and pulse techniques.

Contractor: Union Carbide Nuclear Company, Oak Ridge National Laboratory, Oak Ridge, Tennessee

Contract: W-7405-eng-26

Brief Title: DEFECT STRUCTURES IN ALLOY SYSTEMS

Investigators: D. S. Billington, R. H. Kernohan, M. S. Wechsler, and R. E. Jamison

Scope of Work

The primary aim of this project is to investigate the influence of lattice defects, particularly those produced by fast particle bombardment on metallurgical reactions in solids. Considerable attention is being given age- or precipitation-hardening alloys, such as copper-beryllium, nickel-beryllium, and silver-aluminum, and copper-base solid solution alloys of aluminum. It has been found in the first case that fast neutron bombardment has enchanced the rate of precipitation of beryllium from solution-quenched specimens. The details of this process are being studied in the case of Ni-Be in which the amount of Be in solid solution can be readily determined by measurements of the ferromagnetic Curie point. Studies of aging at various temperatures are being made both during and subsequent to reactor exposure. In copper base solid-solution alloys, which exhibit an unexpected resistivity decrease during room temperature exposure, it appears that the enhanced defect density promotes a solid-state reaction which does not occur in the unirradiated alloy. This behavior is also being investigated by low temperature exposure and subsequent annealing experiments.

Another problem in this area is concerned with the thermal relaxation behavior of a certain type of defects, presumably vacancies, which can be quenched into cadmium-gold (50%) alloys in large concentration. The recovery of electrical resistivity and density changes and X-ray diffraction techniques are being used to study quenched Cd-Au specimens after various annealing cycles.

Section III-C

EFFECTS ON PHYSICAL PROCESSES

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: EFFECT OF IRRADIATION ON MATERIALS

Investigators: S. H. Paine, F. Taraba, A. D. Fischer, B. A. Loomis and K. Myles

Scope of Work

An understanding of damage mechanisms by neutrons in metals has been sought by programs on several fronts, including the self-screening effect of uranium, the effect of neutrons on single crystals, on phase changes, and on thermoelectric emf. The separate programs are described below.

Scope I - Distribution of Neutrons in Uranium Specimens

The surface of a specimen in irradiation serves as a screen to some extent for the interior of the specimen. An empriical mathematical expression for the variation of intensity of neutrons as a function of distance from center in a cylindrical specimen has been developed by employing stacked thin disks which were punched out at certain radial distances after irradiation and then measured for specific activity level. Longitudinal distributions measured in the specific activity level. Longitudinal distributions measured in the same manner were used to provide experimental check of the Wilkins type perturbation at specimen ends. An empirical modification of the Wilkins Effect was developed from this work.

Scope II - Effect on Phase Transformations

Apparatus is under construction to measure the electrical conductivity of in-pile specimens. This will be used to study the effect of neutrons on kinetics of phase changes. Among the systems being studied are U-Mo and U-Nb.

Scope III - Effect on Thermocouples

This program has been initiated to discover irradiation-sensitive metals for use as tools to examine damage mechanisms. Ultrasensitive potentiometric apparatus has been assembled and tested and out-of-pile pulse-annealing tests are in progress on chromel and alumel wires. Tests will be repeated after irradiation of the wires in CP-5 reactor.

Scope IV - Fission-Gas

The kinetics of fission gas accumulation in uranium alloys are being studied by following property changes during in-pile irradiation under controlled ambient temperature conditions.

Contractor: Connecticut, University of, Storrs, Connecticut

Contract: AT(30-1)-2047

Brief Title: RADIATION DAMAGE INVESTIGATIONS IN ALUMINUM OXIDE BY PARAMAGNETIC RESONANCE

Investigators: Otis R. Gilliam

Scope of Work

Paramagnetic resonance investigations will be made on single crystals of aluminum oxide to determine the nature of the damage resulting from exposure to fast neutrons and to gamma-rays. Measurements prior to irradiation will indicate the paramagnetic impurities in the Al_2O_3 crystals. After irradiation, the character of the resonance absorptions may permit an accurate identification of radiation-induced magentic defects. Samples subjected to different radiation dosages will be examined for possible hyperfine structure. Absorption patterns with anisotropic g-factors may assist in the determination of the environment of a particular defect. The g-factor and the line widths of the resonances will be measured and related to theory.

The growth behavior and the annealing behavior of the induced paramagnetic centers will be studied. Correlations between these experiments and similar optical studies reported by P. W. Levy and G. J. Dienes* will be attempted.

A comparison of the integrated intensities with the intensities of calibrated paramagnetic samples should be helpful in obtaining an estimate of the total number of defects created for a given dosage of radiation.

The project will be conducted in cooperation with the Solid State Physics Group of Brookhaven National Laboratory.

*Report of 1954 Bristol Conference, p. 256, Physical Society, London (1955).

Contractor: General Dynamics Corporation, San Diego, California

Contract: AT(04-3)-179

Brief Title: RESEARCH RELATING TO IRRADIATION EFFECTS ON SURFACE REACTIONS OF METALS

Investigators: Massoud T. Simnad

Scope of Work

This research effort is a continuation of the studies carried out during the past year under AEC sponsorship at Carnegie Institute of Technology, with which the present principal investigator was associated. The objective here will be to complement the research in this field which is being done elsewhere, and to widen its scope. This will include investigations on many different metals, metal oxides and hydrides to compare the relative effects of various types of irradiations, namely, the influence of neutrons, gamma rays, protons, and electrons. The experiments which are of special interest include the study of the effects of irradiation upon the following:

(1) Gas-metal and gas-metal-oxide reactions - The rates of these reactions may well be strongly influenced by the presence of ionizing radiations, since they are governed by the migration of ions across the solid films of reaction products.

(2) Corrosion rates of metals and solid oxides in various solutions - The corrosion rates could be measured in conjunction with electrode potential measurements. Changes in polarization of local anodes and cathodes may be related to changes in anode and cathode corrosion currents. A study of the corrosion of metals in irradiated aqueous solutions would be of special interest.

(3) The electrode potentials of metals in aqueous solutions - In particular, this should include metals whose normal electrode potential can be measured against a standard cell. Electrode potentials of metals are known to be sensitive to changes in the metal structure, such as those produced by plastic deformation, and they may be used as a measure of the changes in free energy and entropy produced by irradiation.

Contractor: National Bureau of Standards, Washington, D.C.

Contract: NBS Project No. 0509-11-3271

Brief Title: THERMOCHEMICAL STUDY OF IRRADIATION EFFECTS

Investigators: E. J. Prosen

Scope of Work

The objectives of this project are: (1) to develop methods and apparatus and make calorimetric measurements of heats of formation and of the increase in energy content of quartz, vitreous silica, silicon carbide, and other insulators or refractories resulting from exposure to radiation in nuclear reactors; and, (2) to determine the effects of annealing at different temperatures.

The heat-of-reaction data will yield heats of formation which cannot be obtained by ordinary room-temperature calorimetry and the stored energy measurements will aid in understanding of the changes which occur in exposed substances.

Work has been completed on the heat of combustion of irradiated and unirradiated silicon carbide. Work is proceeding on the development of a calorimeter for determination of heats of reaction and solution at temperatures up to 100°C and for use with aqueous hydrofluoric acid solutions.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

Contract: AT(30-1)-1565

Brief Title: RADIATION DAMAGE IN METALS - SURFACE EFFECTS

Investigators: W. L. Lees

Scope of Work

The successive-collision process, conceived for the dissipation of energy imparted by a fast neutron to a primary knock-on atom in a metal, can be tested by the rate at which the metal evaporates during irradiation. This neutron-induced analog of sputtering is observed by enclosing a source-surface of the test metal in an evacuated receiver of different material, irradiating the assembly, and then measuring the amount of source material on the surface of the separated receiver. The use of an iridium source permits high sensitivity in the detection of collected material after reactor irradiations, because of the high specific activity produced by thermalneutron capture. From the known mass of the source, its specific activity is determined, and thus the mass of evaporated source material is found. The fast-neutron flux is known roughly from comparison of certain radiation-damage effects with those produced by unmoderated fission neutrons.

Capsules, each comprising two coaxial cylinders as receiver and iridium source, were irradiated on the axis of heavy-water-filled tubular fuel assemblies in the CP-5 reactor. With 4.0×10^{11} joules developed in the 66-cm-long fuel tube of 6.cm diameter, the iridium sputtered per cm^2 of source surface was 3×10^{-8} gm, or rougly 1.6×10^{-10} gm atom. Data from W. Primak indicate that, in terms of damage to vitrous silica, the exposure of these capsules was roughly equivalent to 10^{20} fission-spectrum neutrons per cm^2.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

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Brief Title: RADIATION EFFECTS ON PRECIPITATION-HARDENING ALLOYS

Investigators: A. Boltax

Scope of Work

Studies of the effects of fast neutron irradiation on Cu-Fe precipitation hardening alloys were extended by a recent irradiation at the CP-5 reactor. Irradiation effects were studied on solution-treated, aged, and cold-worked samples by electrical and magnetic measurements as a function of integrated neutron flux. The present data are in agreement with the results obtained previously which indicated that the behavior of aged samples under irradiation could be described by the competitive processes of irradiation-induced precipitation and re-solution as a function of precipitate particle size.

The solution-treated samples showed definite indications of irradiation-induced aging similar to that reported by Murray and Taylor for Cu-Be alloys. The behavior of the aged samples under irradiation was a function of the aging temperature before irradiation which in turn determines the precipitate particle size. Samples aged below 500°C showed an increase in electrical resistivity consistant with irradiation-induced aging. The irradiation processes of re-solution and precipitation can be separated and analyzed as a function of neutron flux and heat treatment. Re-solution is considered to take place by localized homogenization by displacement spikes which affect 4×10^3 to 2×10^4 atoms per spike. On the other hand, precipitation, by means of the enhancement of diffusion by the presence of excess vacancies and interstitials, can be as large as 50 to 100 atoms per incident neutron in a 1.7 w/o Fe sample aged for short times at 250°C.

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