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METALLURGY AND CERAMICS

UNITED STATES ATOMIC ENERGY COMMISSION

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

Edited by
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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 studies are involved.

Under these contractual arrangements, physical research is being performed in metallurgy, chemistry, and physics. For each of these 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 summariezes the programs supported by the Division of Research in the fields of metallurgy, solid state physics, and ceramics. It includes the research conducted at the Commision's laboratories as well as that performed at the universities and other laboratories. The summaries have been 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. Alloy Theory and the Nature of Solids

III. Radiation Effects on Materials

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

Reports resulting from these contracts which are supplied to TISE may be abstracted in the journal of *Nuclear Science Abstracts*. The journal is published bi-monthly by the Technical Information Service Extension and is available on subscription at six dollars a year from the Superintendent of Documents, Government Printing Office, Washington 25, D. C.

Section I

PRODUCTION, TREATMENT, AND PROPERTIES OF MATERIALS

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, and W. R. Yario

Scope of Work

The physical metallurgical studies of uranium are designed 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 self-diffusion coefficients of gamma (body-centered cubic) and alpha uranium are being studied by tracer techniques. Polycrystalline samples of both modifications are being investigated. Self diffusion in single crystals of alpha uranium is being studied as a function of crystallographic direction.

Scope II - Elastic Constants of Alpha Uranium

Single crystal specimens of alpha uranium are being prepared for measurements of elastic constants as a function of crystallographic direction by use of ultrasonic techniques.

Scope III - Thermal Expansion Coefficients of Alpha Uranium Single Crystals

Dilatometric tests of alpha uranium single crystals are being made to measure expansion coefficients as a function of crystallographic direction.

Scope IV - Volumetric Behavior of Uranium

Physical measurements of the volumetric changes of uranium as a function of temperature are being made. The volume changes upon phase transformation (from alpha to beta, and from beta to gamma) are of particular interest. These data will be compared with values calculated from lattice parameter measurements of the three allotropic modifications and with alpha uranium thermal expansion coefficient data.

Scope V - Deformation Mechanisms 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. The program includes identification of operative deformation mechanisms as a function of temperature and crystallographic direction, determinations of the relative operability of slip and twinning deformations as a function of temperature, and studies of substructures formed during deformation.

Scope VI - Recrystallization and Grain Growth of High Purity Uranium

A systematic study of recrystallization and grain growth in alpha uranium is in progress. The work includes investigation of the effect of such factors as chemical compositions, amount and type of deformation, and time and temperature of annealing.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: URANIUM ALLOYS

Investigators: A. E. Dwight, S. T. Zegler, 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.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: X-RAY DIFFRACTION STUDIES

Investigator: M. H. Mueller

Scope of Work

Scope I - Lattice Parameters of Uranium as a Function of Temperature

In the study it is intended to obtain accurate lattice constants at various temperatures for the alpha, beta, and gamma forms of uranium. If these experimental results are obtained at a temperature just above and just below the two transformation temperatures, it should be possible to determine the change in volume associated with these transformations. A careful check will also be made of the diffraction patterns obtained within the beta range to determine if there is any evidence for a structural change which might confirm other reported evidence of a possible change within this phase.

Scope II - Structure of the U–Zr and U–Nb Intermetallics

The purpose of this study is to determine the nature and crystal structures of the delta phase found in the U–Zr binary system and the phase which is found in the high uranium portion of the U–Nb system. Although both phases are formed from a high temperature body-centered cubic solid solution phase they are not considered to have a similar structure.

Initial single crystal work on the delta phase has indicated that the unit cell may be considered as a large cubic cell with 54 atoms/unit cell. However, it has been observed that there is a very regular displacement of certain of the powder diffraction lines from cubic symmetry. Present efforts are directed towards a determination (1) of the nature of this displacement (2) the specific location of the U and Zr atoms within the unit cell and (3) possible other intermetallics with a similar structure.

In the U-6 to 9 w/o Nb alloys, a phase has been found whose X-ray pattern may seemingly be indexed on the basis of a tetragonal cell; however, there are a number of extra lines which cannot be accounted for by such a cell. A further study is being carried out to determine (1) if this tetragonal cell is the true unit cell (2) the arrangement of the U and Nb atoms within this phase and (3) the correlation of physical property changes with the development of this phase from the body-centered cubic phase.

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

Contract: NBS Project No. 0802-11-4100

Brief Title: BINARY URANIUM ALLOY SYSTEMS

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

Scope of Work

This program has as its primary objective the establishment of the constitutional diagrams of binary uranium alloys. The alloys of uranium with the six platinum metals are presently being studied by means of thermal, metallographic, and X-ray methods. The phase diagram work on the U–Pt system is practically completed; investigations of the U–Pd and U–Ru systems have covered almost the whole range of alloys from 100 to 0% uranium; and the studies of the U–Ir, U–Os, and U–Rh systems have but recently been initiated. 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.

The completion of the phase diagrams of uranium with the platinum metals will provide a basis for studies of the alloying characteristics of uranium as well as the similarities and differences within such a group of diagrams.

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: URANIUM CONSTITUTION DIAGRAMS

Investigators: R. F. Dickerson and C. M. Schwartz

Scope of Work

In order to increase knowledge in the field of uranium-alloy constitution, studies of the uranium–zirconium–molybdenum and uranium–carbon systems are being made. Additional future studies of the uranium–zirconium–silicon system are planned.

Alloys ranging in composition between the intermediate delta phases of the uranium–zirconium and uranium–molybdenum systems are being studied as a means of determining the phase relationships which occur between these phases. Thermal, metallographic, and X-ray diffraction techniques are being employed.

High-temperature X-ray techniques are being employed in a study of the phase region between the UC and UC_2 compounds of the uranium–carbon system. The objective of this investigation is to define accurately the high-temperature portion of this region.

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: PREPARATION AND PROPERTIES OF URANIUM COMPOUNDS

Investigator: W. H. Duckworth

Scope of Work

The objective of this project is to supply data on the properties of refractory uranium compounds, other than UO_2, to enable evaluation of their potentialities as high-temperature fuel compounds. Properties of interest include melting point, uranium content, chemical reactivity and stability, thermal conductivity, thermal expansion coefficient, electrical resistivity, and strength. The information available in the literature has been collected, and experimental work is under way to supplement that information.

Contractor: Knolls Atomic Power Laboratory, Schenectady, New York

Contract: W-31-109-eng-52

Brief Title: URANIUM – ZIRCONIUM EQUILIBRIUM

Investigators: C. A. Bruch, J. F. Duffey, and E. R. Boyko

Scope of Work

Uncertainties existed in the equilibrium phase diagram of the uranium – zirconium alloy system in the 50-50 composition range at temperatures below 700°C. This program was established to determine the equilibrium phase boundaries in the intermediate composition ranges and to determine the crystallographic structures of any new phases which may be present.

It has been established that, in approximately 50-50 weight per cent alloys, when the high-temperature gamma phase (body centered cubic) is cooled, it transforms to an ordered structure called either delta or epsilon. This phase can be indexed in a hexagonal space group, C3, which accounts for all observed intensities.

On the basis of cooling curves and metallographic studies of a series of alloys containing from 34 to 68 weight per cent zirconium, the boundaries of the delta or epsilon phase field have been established. The results are consistent with results reported for alloys in composition ranges bordering the range studied. The resulting diagram does not differ much from the tentative diagram published by Seymour and Holden. (J. Metals, Oct., 1956, p. 1312.)

Contractor: Columbia University, New York, New York

Contract: AT(30-1)-1593

Brief Title: A STUDY OF INCLUSIONS IN URANIUM

Investigator: George L. Kehl

Scope of Work

The purpose of this research is to study and to identify metallographically inclusions in uranium metal. In brief, inclusions from selected specimens of high purity uranium will be mechanically removed and subjected to micro-beam X-ray diffraction analysis. Subsequently, techniques and procedures will be devised to enable unequivocal metallographic identification.

Specimen metal will be supplied by Argonne National Laboratory, and a portion of the X-ray diffraction work will be carried out at that laboratory.

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: STUDY OF THE TENSILE TRANSITION IN ALPHA URANIUM

Investigator: L. L. Marsh, Jr.

Scope of Work

Alpha uranium exhibits a tensile transition from a shear-type to a semicleavage-type fracture in the vicinity of room temperature. It has been observed that the hot rolling temperature, the annealing temperature, strain rate, and grain size influence the temperature range over which the transition is observed. Metallographic examination has revealed that twinning is the primary mode of deformation over the temperature range of the transition; twinning accounting for approximately 70 per cent of the plastic strain. At higher temperatures, slip assumes greater importance.

In line with this background information, the objective of the current program is to study the deformation processes prior to fracture in order to deduce the sequence of events resulting in the transition. On the assumption that there is a transition from slip to twinning with decreasing temperature, a quantitative metallographic study will be made of the relative amounts of slip versus twinning at various temperatures. Further, it is known that grain size and grain orientation affect the ease of twin and slip-line formation. Therefore, it would be appropriate to study these variables in detail.

There is another line of reasoning which will be investigated. Metallographic examination of fractured specimens has shown that the fracture path is often along twin boundaries. This suggests that the high-stress field associated with deformation twin boundaries is not relaxed sufficiently fast by secondary deformation or some other process. Accordingly, a study will be made of stress relaxation in uranium using internal-friction techniques.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: BASIC PLUTONIUM METALLURGY

Investigators: L. R. Kelman and B. Blumenthal

Scope of Work

Scope I - High Purity Plutonium

The preparation of high purity plutonium is the object of this program. A fundamental physical metallurgical research program on plutonium requires the availability of high purity metal, preferably of such composition that the residual individual contaminants have no significant effect on the measured property. In general, it is desired for high purity metal to contain no more than a few ppm of a small number of elements, be metallographically homogeneous, and as an ingot, have almost theoretical density.

The first approach utilizes the experience gained with the preparation of high purity uranium and uses both pyrometallurgical and high temperature electrolytic processes. The need for developing methods of controlling the hydrogen, carbon, nitrogen, oxygen, and heavy metal content of plutonium is anticipated, whereas the control of the lighter metals presently appears less difficult for thermodynamic reasons.

The study will require an adaptation of the chemical and spectrochemical analytical method to the very low concentration of contaminants of a pure metal. In addition, physical and metallographic methods will be used to determine the purity of the metal.

Scope II - Properties of Plutonium

The purpose of this research is to contribute to the fundamental knowledge of the metallurgy of plutonium. Plutonium is known to behave peculiarly in many respects; for example, it is unique as a metal in that it has six allotropic forms, the face center cubic phase has a negative expansion coefficient, and its thermal conductivity is extremely low. A study of all properties of plutonium should throw light on these peculiarities and thus assist a better understanding of the metal.

Recrystallization and grain growth as well as deformation mechanisms and the kinetics of transformations of the various allotropic forms are included in the program. This work will lead to a better understanding of the fabrication methods for this metal.

Another area of interest is the influence of impurities on the properties of plutonium.

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

Contract: W-7405-eng-82

Brief Title: THE METALLURGY OF SPECIAL METALS

Investigators: H. A. Wilhelm, O. N. Carlson, D. Peterson, P. Chiotti, B. A. Rogers, J. F. Smith, R. E. Rundle, G. H. Beyer, A. H. Daane, and M. Smutz

Scope of Work

Scope I - Extraction and Purification of Metals

Metal preparation studies and alloy investigations constitute a large part of the metallurgy program at Ames. Much of the work has some direct bearing on the search for special property materials of interest in the broad program on atomic energy by the U.S.A.E.C. Quite often the studies connected with a metal include investigations on ore treatment and on subsequent separation and purification processes. At this time, liquid-liquid extraction and ion-exchange are receiving most attention as means for obtaining purified compounds of the metals. These compounds are then employed in investigations on metal preparation.

Present investigations include work on opening of columbite, tantalite, zircon and rare earth containing ores. The removal of niobium from tantalum, of hafnium from zirconium and of the rare earths from one another and associated elements constitute most of the separation studies.

Methods have been developed for preparing many of the less common metals including all of the rare earths. Studies are continuing on a number of metal reduction processes in an effort to improve their quality and to reduce their cost.

Scope II - Phase Diagrams

A major effort has, been directed toward the study of many new alloy systems with emphasis on construction of the diagrams. With the large number of alloy systems to be investigated, the work has generally been directed along lines that, from primary considerations, might possibly yield an alloy that finds some practical application in the present atomic energy program. In addition to the practical aspects of these studies there is also the accumulation of information on alloy behavior which is valuable in the effort to build up a sound science connected with the nature of the metallic state.

Phase diagram studies are well under way or nearing completion on the U–Sb, U–Nb, U–Th–Al, U–Hf, Th–Mo, Th–V, Th–Hf, Zr–Zn, Zr–Ta, Nb–Ta and V–Ta systems. Investigations on several alloy systems involving rare earth metals are in progress. These include the La–Nd, Nd–Tm, La–Gd, Gd–Y, La–C, La–B, and LaO systems. Work has been initiated on the systems of V–Cr, Y–Ti and Y–V.

Scope III - Structure and Properties of Metals, Alloys, and Intermetallic Compounds

The physical properties of the rare earth metals and of the reactor materials such as uranium, thorium, and zirconium are under investigation. This work includes measurements of such properties as melting points, vapor pressures, crystal structures, allotropic transformations, heat capacities, thermal expansion coefficients, elastic constants, magnetic properties, and electrical resistivities. Some of these properties are particularly sensitive to the presence of impurities, and the effect on such properties of variation in type and quantity of impurity is being included in the investigations.

An investigation of the structure of intermetallic compounds and of the relationship of structure to the physical properties is also in progress. Representative compounds of various

types have been chosen for investigation. These include compounds whose structure seems primarily controlled by size considerations such as Laves' phases, compounds whose structure seems primarily controlled by the electron/atom ratio such as the gamma-brass structures, and compounds with highly directed valence bonds such as the silicides. Measurements include thermal expansion, elasticity, electric and magnetic properties, and the directional variation of these properties. Some thermodynamic studies are also being made.

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., E. B. Evans, and C. A. Barrett

Scope of Work

The investigation is aimed at attaining a fuller understanding of the peculiar behavior of zirconium when scaled at elevated temperatures. These pecularities include a change in nature of the scale product after different periods of oxidation, a sharply increased scaling rate after (sometimes) long periods of slow scaling, and, under some circumstances, large deformations and distortions in the metal again after some period of oxidation. The periods of time at which these three phenomena set in do not always coincide.

The dependence of these phenomena themselves and their nucleation times on gas composition, change in gas composition, previous surface treatment, shape and size of piece, temperature, are being studied. Micrographic, X-ray, electrical resistance and other studies are being made on the nature of the scales and the underlying layers. An adequate rationale of all these phenomena is being sought.

Contractor: Armour Research Foundation, Chicago, Illinois

Contract: AT(11-1)-315

Brief Title: ZIRCONIUM PHASE DIAGRAM STUDIES

Investigators: Robert F. Domagala and David W. Levinson

Scope of Work

During the past two years efforts have been directed to establish on a sound scientific basis the heat treatability of zirconium-base alloys. Eutectoid type systems are the only ones where alloys show any appreciable response to heat treatments.

The current work is a shift of emphasis to a definitive study of phase relationships in the Zr corner of the ternary system Zr–Fe–Sn. The compositional limits set for this work are 24 per cent Sn (Zr_4Sn) and 47 per cent Fe ($ZrFe_2$); the temperature range is from 600° to 1100°C. The binary Zr–Fe and Zr–Sn systems have been established. Approximately 100 25-gram ingots are being prepared by nonconsumable electrode arc melting. The investigation is proceeding for the most part by metallographic evaluation of heat treated and quenched specimens. Anneals are conducted at 100° intervals except where smaller increments are necessary to establish firmly the planes of four phase reactions. The solubilities of the addition elements in α and β Zr are being investigated, and the boundaries for the various phase fields will be laid out. The diagram is based on alloys prepared with iodide zirconium, but additional alloys will be prepared which contain a fixed (and deliberate) oxygen content to observe the shift in phase boundaries which may occur as a result of oxygen contamination.

Contractor: New York University, New York, New York

Contract: AT(30-1)-1902

Brief Title: ZIRCONIUM-OXYGEN ALLOYS

Investigator: Polykarp Herasymenko

Scope of Work

The aim of this work is the determination of thermodynamic properties including free energy, enthalpy and entropy of formation of zirconium-oxygen solid solutions in the range up to about 50 atomic % oxygen. The work will comprise:

1. Construction and testing of apparatus for equilibration of zirconium samples with gas of known partial pressure.

2. Actual measurements at various pressures of oxygen and at several temperature levels (900–1300°C).

Zirconium-oxygen alloys will be produced by equilibrating zirconium metal samples embedded in calcium oxide with calcium vapor of known partial pressure in evacuated molybdenum vessels. The composition of alloys will be determined from weight increases of metal samples after exposures at elevated temperatures.

Contractor: Horizons Incorporated, Cleveland, Ohio

Contract: AT(30-1)-1894

Brief Title: POTENTIAL METHODS FOR PREPARATION OF NIOBIUM METAL

Investigators: M. A. Steinberg, M. E. Siebert, A. J. Kolk, and C. J. Zelnik

Scope of Work

The over-all purpose of this contract is to evaluate a number of possible methods for preparation of niobium metal. The methods include fused salt electrolysis, active metal reductions and non-metallic reductions such as a carbon or hydrogen using appropriate niobium compounds. On conclusion of this laboratory scale investigation, recommendations will be made relative to the most feasible procedure(s) for a larger scale investigation.

A basic part of the program entails the preparation of a number of niobium compounds for use in the process evaluation. These include normal and reduced fluorides and chlorides, double fluorides and reduced oxides.

The thermal reduction portion of the work includes sodium, magnesium and possibly other metallic reductions of chlorides, fluorides and oxides.

The fused salt electrolytic phase of the work entails a study of the electrodecomposition of niobium fluorides and chlorides dissolved in melts of alkali or alkaline earth chlorides. Inert atmosphere graphite lined cells are being employed for this work.

In addition to the fused salt electrodecomposition work, impure niobium is being prepared by carbon or hydrogen reduction of the pentoxide. This is then employed as a soluble anode using a similar melt to give an electrorefining process.

Metal products prepared in these three phases of the work are subjected to suitable evaluations by analytical techniques, metallographic work and physical property determinations.

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: REACTION KINETICS OF HYDROGEN AND NITROGEN WITH NIOBIUM

Investigator: M. W. Mallett

Scope of Work

A fundamental investigation is being made of the kinetics and mechanisms of the reactions of nitrogen and hydrogen with high-purity niobium. This study includes the determination of the kinetics of the reaction over as wide temperature ranges as possible using manometric (Sievert's apparatus) and gravimetric (microbalance) techniques. The types of rate laws that are followed and the corresponding reaction-rate constants will be evaluated. From this, the activation energies for the reactions will be calculated. Various experiments to determine semiconductor properties, structure, and composition of reaction films will be made in establishing the reaction mechanisms. This will also include the determination of the diffusion species through the film. Further, the rate of diffusion of nitrogen and hydrogen in niobium will be studied. Diffusion gradients will be prepared in niobium cylinders and the gradients analyzed to evaluate diffusion coefficients. Terminal solubilities of each of the gases in niobium will be calculated from the diffusion data.

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

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

Brief Title: SELF-DIFFUSION OF NIOBIUM

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

Scope of Work

This research has the objective of determining the diffusion coefficient and activation energy for the self-diffusion of niobium. Radiotracer techniques are being used by virtue of the isotope Nb^{95}.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

Contract: AT(30-1)-1565

Brief Title: EFFECTS OF ALLOYING OF VANADIUM ON SUPERCONDUCTING PROPERTIES

Investigator: A. Boltax

Scope of Work

Recent experimental work has revealed that approximately one percent additions of such elements as Pd, Al, Ni, Cu, and Fe to V improves the superconducting properties as required in the fabrication of cryotron elements. A cryotron is a device used for amplification based on the destruction of superconductivity by a magnetic field. Briefly, the device is a superconductor at the boiling point of He (4.2°K) and can be changed to a nonsuperconductor by application of a magnetic field. The properties desired are a high residual resistivity (R_o) and a low threshold magnetic field (H_c) both measured at 4.2°K.

Preliminary measurements of both R_o and H_c have been made for several vanadium alloys, and further experiments are planned to study R_o and H_c as a function of composition. In particular, a series of V–Pd and V–Al alloys will be fabricated by arc-melting techniques and their superconducting properties studied.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

Contract: AT(30-1)-1565

Brief Title: BRITTLENESS OF BERYLLIUM

Investigator: A. R. Kaufmann

Scope of Work

Beryllium of commercial purity fractures easily on the basal plane and less readily on the 1120 prism plane. It is desired to determine the effect of transition element impurities, such as iron and nickel, on the 1120 fracture. Powdered beryllium will be mixed with small amounts of the impurity element and then extruded and cross-rolled to produce "ductile" sheet. The sheet will be heat-treated to get the added impurity into solution. The texture in this material is such that fracture on the basal plane can be avoided. It is hoped that a correlation between loss of ductility and impurity content will be found.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1825

Brief Title: THERMODYNAMIC PROPERTIES OF THE RARE EARTH CARBIDES

Investigator: C. L. McCabe

Scope of Work

The purpose of this work is to determine the free energy of formation of the rare earth carbides by vapor pressure and gaseous equilibria techniques. Vapor pressures are to be determined by the Knudsen orifice method. Methane–hydrogen mixtures are to be used for the gaseous equilibria measurements. Cerium and praseodymium carbides are being studied first.

Contractor: Syracuse University, Syracuse, New York

Contract: AT(30-1)-1910

Brief Title: ALKALINE EARTH EQUILIBRIUM SYSTEMS

Investigators: Aden J. King and Frank A. Kanda

Scope of Work

It is intended to complete the studies of alkaline earth alloy systems and their transitions in the solid state and to attempt to settle some thermal anomalies which have been observed for some of the "pure" metals and their alloys.

Due to their close family relationship and similar electronic structure, alloys within the alkaline earth group are of interest from the viewpoint of generalities in alloy systems. The calcium-strontium system is of special interest in solid state transformations because both metals display three allotropic forms, αF.C.C., βH.C.P., and γB.C.C. Both metals show thermal and dilatometric evidence of transitions in the solid state which are not revealed by high temperature x-ray technique. The mechanism of the β to α transition is not fully understood. X-ray evidence shows extra lines in the patterns which have not been explained satisfactorily but may be due to long range order-disorder processes.

Factors which effect solid state transitions such as impurities, cold working, etc., will be investigated for some of these systems. In connection with this we plan to investigate barium at low temperatures which, in contrast to calcium and strontium, shows only B.C.C. between room temperature and the melting point.

Investigations will also be made of systems involving other metals with the alkaline earths such as indium, aluminum, and alkali metals (lithium, sodium and potassium) which may shed additional light on the problem.

Contractor: Tufts University, Medford, Massachusetts

Contract: AT(30-1)1410

Brief Title: BASIC PROPERTIES OF LIGHT METAL HYDRIDES

Investigators: Charles E. Messer and Thomas R. P. Gibb, Jr.

Scope of Work

The research is concerned with the physical-chemical investigation of light metal hydrides, particularly thermodynamic properties, phase relationships, hydrogen dissociation pressures, and densities.

Study of the melting-freezing behavior of mixtures of lithium metal and lithium hydride has been extended from 91% down to 5% hydride. This work is being repeated on a new sample to determine the effect of slight impurity. Dissociation pressures will also be measured on this sample.

Melting-freezing behavior and dissociation pressure studies will be extended to mixtures of lithium hydride with related substances such as sodium hydride, calcium hydride, and lithium fluoride.

An ice calorimeter has been constructed to measure the high temperature heat capacities of lithium hydride, sodium hydride, and potassium hydride, to obtain chemical thermodynamic properties.

A furnace has been constructed for the growth of single crystals of lithium hydride from the melt, as part of the investigation of the production of color centers in hydrides by ionizing radiation.

X-ray crystallographic and density studies on hydride systems will be carried out as conditions warrant.

A supplement to Report NYO-3957, "A Survey Report on Lithium Hydride," will be issued, bringing this report up to date as of about January 1, 1957.

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

Solid state bonding processes used in the cladding of reactor fuel elements frequently involve diffusion with the formation of intermediate layers in the diffusion zone. Not enough is known about the process of layer formation during diffusion. Existing data indicate that intermediate layer formation conforms neither with the phase diagram nor classical diffusion theory. It has been found that pressure markedly influences layer formation and the strength of the diffusional bond.

A study is being made of layer formation during diffusion in order to ascertain the factors which control the formation and properties of the diffusional bond. Diffusion with the formation of intermediate phases is being investigated initially in the nickel-aluminum system, since this has been studied to some extent in the past and is known to exhibit a marked pressure effect. A thorough analysis of the diffusion processes taking place is being made over a selected range of temperatures and pressures.

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: BONDING FUNDAMENTALS

Investigator: H. R. Ogden

Scope of Work

The solid-state bonding of metals is a subject of considerable technical interest. Among others, the process of cladding is based on the bonding of two metals, both in the solid state. The mechanism generally is considered to involve two steps: (1) the bonding of minute irregularities, or asperieites, on the surface, and (2) growth of these areas, presumably by diffusion and/or recrystallization, to complete the surface bond.

Most of the previous research has been done on the macroscopic scale by bonding together relatively large flat surfaces. In the present work, the study is being made on a microscopic scale in which single asperity bonding is simulated. This is done by bringing a gold needle, having a very small tip radius, in contact with a polished flat gold surface. The applied load, temperature, loading time, and degree of cold work in the material are controlled variables. Bond strengths are measured as the normal force required to break the bond.

Recent experiments have confirmed the model, showing that above a critical temperature, bond strengths are increased with increasing time, even when the load is reduced to a low value. Below this temperature, the strengths do not show a time dependence, presumably because only the asperities have bonded.

Attempts are being made to relate the rate of increase in bond strength and area to a fundamental process, probably either diffusion of recrystallization. A relationship of this kind would be of considerable value in the establishment of bonding procedures.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

Contract: AT(30-1)-1565

Brief Title: FUNDAMENTAL STUDIES ON COEXTRUSION

Investigator: W. B. Nowak

Scope of Work

One of the major problems in the coextrusion of nuclear fuel elements arises from the incorporation of integral end seals. Unless the billet is properly designed, the extrusion will be excessively long or the fuel will pierce the cladding. Both for its own sake and as an aid in the design of billets, a study is being made of the deformations of an interface similar to that of an integral end seal. Various ratios of extrusion constants of fuel and cladding and various interface and extrusion geometries are under investigation.

During the past year, a "plane strain" extrusion press with a glass observation window was constructed for use with plasticene as the working material. Preliminary data including motion pictures were obtained on the deformation of non-orthogonal grids and on the deformation of a shaped interface.

Carefully controlled before-after experiments were also performed using circular rods of lead and lead-antimony alloys. The range of variables included: 0.5 to 2.0 for the ratio of extrusion constants of the two parts of a billet; 180°, 170°, 160°, and 150° for the included angle of the cone-shaped interface; 4, 9, 16, and 25 for the extrusion ratios.

The program for this year is to extend the plane strain investigations over a wider range of extrusion constants and interface shapes and to look into the behavior of corners and edges where separate pieces of billet material come together. The range of the lead experiments will also be extended to cover more acute cone angles and greater ratios of extrusion constants.

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 - Solubilities and Kinetics for Liquid Bismuth–Solid Systems

Iron and chromium are slightly soluble in liquid bismuth at moderate temperatures (300–700°C) and form no known intermetallic compounds with bismuth. The presence of either in the liquid metal affects the solubility of the other. A study of the thermodynamics of these effects is of value in the selection of a container material for a liquid bismuth-base reactor fuel. Further solubility effects due to the uranium fuel, titanium + magnesium, and zirconium + magnesium additives as corrosion inhibitors, and fission products generated in the fuel stream are also being investigated. Possible surface reactions between these same elements dissolved in the liquid bismuth and several steel or graphite containers are being investigated using radioactive tracer and x-ray reflection techniques. The effects of these same elements in the liquid metal on the kinetics of dissolution and precipitation of iron and chromium are also being studies in relation to the types of surface deposits identified.

The effects of the steel corrosion products, corrosion inhibitors, and fission products on the solubility of uranium in liquid bismuth are also being investigated.

Scope II - Solid-Liquid Equilibra in the Th-Bi-Pb System

The equilibrium diagram of the thorium-bismuth-lead system is being determined with particular attention being given to the low melting liquid-plus-solid portion of the system. Attempts are being made to establish the composition and crystal structure of the intermetallic compounds encountered in the system. The distribution of small amounts of other elements (uranium and protactinium) between the liquid and solid phases is also under investigation. The effects of cooling variables and minor additions of other elements upon the size and shape of intermetallic compound particles precipitating from solution in the liquid metals are being determined.

Scope III - Effect of Radiation on Materials

The radiation effects program is concerned with the evaluation of materials for the liquid metal fuel reactor (LMFR). Both static and dynamic tests are contemplated. Two materials are being studied, steel and graphite. The properties of concern in graphite are thermal conductivity, gross growth, c-axis change, and stored energy. These properties are being studied as a function of neutron flux and temperature in the presence or absence of a U–Bi solution. It is believed that the absorption of uranium on graphite is reduced by the formation of a ZRN–ZrC layer. The effect of temperature and radiation on this layer will be investigated as well as distribution of fission products and uranium in the graphite.

Steel will be used outside the LMFR core within which the U–Bi solution will circulate. The physical properties of this steel is being investigated as a function of fast neutron flux and the corrosive medium. Properties of interest are yield point, ultimate strength, and impact strength. The effect of fission fragments on the corrosion resistance of steels to U-Bi will also be investigated.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

Contract: AT(30-1)-1565

Brief Title: SOLUBILITY IN LIQUID METAL SYSTEMS

Investigator: S. Isserow

Scope of Work

The ultimate purpose of this investigation is the understanding of the factors determining solubility in liquid metal systems. The more immediate, practical goal is the development of a set of generalizations permitting the prediction of solubility on the basis of simple parameters (e.g., atomic number, melting point, heat of fusion) or parameters deducible from observed data.

Because of relative experimental ease and potential application for a liquid metal fuel reactor, bismuth is receiving prime attention. Data in the literature on metal solubilities in bismuth are being reviewed. In addition to checks of these data, the solubility of other metals will be determined. Some modification will probably be necessary in presently available experimental equipment, in which the saturated liquid metal is filtered at various temperatures. The experimental data will check generalizations from prior data. The new data will include other solvents and multiple solutes, such as those forming intermetallic compounds.

Contractor: New York University, New York, New York

Contract: AT(30-1)-1837

Brief Title: SODIUM–POTASSIUM ALLOYS

Investigator: Benson R. Sundheim

Scope of Work

The vapor pressure of both components of liquid Na–K alloys is being determined by measurement of the optical absorption spectra. From the vapor pressures and their variation with temperature and composition the significant thermodynamic quantities are to be computed.

Specially prepared glass membranes are being used to determine the activity of Na in certain Na–Hg solutions. The transference numbers of various ions are being determined in this glass so that it may be used in concentration cells for measuring the activity of the components of Na–K solutions at temperatures were the vapor pressure method cannot be used.

Contractor: New York University, New York, New York

Contract: AT(30-1)-1938

Brief Title: STUDIES ON MOLTEN SALTS

Investigator: Benson R. Sundheim

Scope of Work

A specially constructed spectrophotometer is being used to study the absorption spectra of molten salts. A systematic survey of the visible and ultraviolet absorption spectra of pure molten salts is in progress. Certain more complicated systems, such as solutions of cadmium in cadmium halides and solutions involving halide complexes in the transition metal series are also being studied. Highly precise measurements of electrical conductance are being made on the same systems. In addition to the purely descriptive information being obtained, it is expected that some generalizations about the structure of molten salt systems will result from this work.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1024

Brief Title: ELECTROCHEMICAL STUDIES OF NON-AQUEOUS MELTS

Investigator: G. Derge

Scope of Work

The general objectives of this contract are to expand knowledge of the chemical characteristics of non-aqueous melts as typified by fusions of salts, sulfides and oxides. This is required to understand and use them to advantage. The study is primarily by direct measurements of such properties as electrical conductivity and ionic transport phenomena, but supplemented by determination of such other properties as diffusion coefficients, surface tension, and density, which may contribute to interpretation of the data. These systems are characteristically of interest at high temperature and a variety of experimental procedures have been developed for work in the range from 1000°–1600°C.

The high electronic conductivity of molten cuprous sulfide changes little as the sulfur activity is reduced below that of the stoichiometric composition, but increases rapidly if the sulphur activity is increased. Measurements with radioactive tracer by the capillary-reservoir method have shown that the self diffusion of iron in molten iron sulfide is comparable to that in iron-carbon alloys and other liquids. However, the diffusional activation energy in the sulfide appears to be unusually high. The mobility of iron in silica saturated iron silicate has been measured at 1250°C as $\mu = 0.0009$ cm^2 per volt second.

The research will be continued to obtain a more extensive understanding of the influence of composition and activity variations on conductivity and diffusion in simple and mixed sulfide and oxide systems. The observations made to date are too limited to allow satisfactory generalization.

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 - Reactions in Fused Sodium Hydroxide

Three kinds of reactions are under investigation. These are self-decomposition, mass transfer, and leaching of reactive elements from alloys. This study is directed as determining the basic mechanisms by which fused sodium hydroxide reacts with metals and ceramic materials.

Scope II - Spectrophotometry of Fused Salts

Measurements are being made of the absorption spectra of fused salts in the spectral region of 200 to 750 mμ. The purpose of these measurements is to determine the presence of complex ions and other light absorbing species in fused salt melts. Measurements are made with a high resolution, recording spectro photometer of the flicker-beam type. Present investigations are largely concerned with solutions of various substances in alkali halide melts. These substances include transition metal ions and alkali metals.

Scope III - 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. Preliminary work consists of measuring the proton and sodium resonances in fused sodium hydroxide.

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

Contract: NBS Project No. 0506-11-3275

Brief Title: HEAT EFFECTS OF DYNAMIC ELECTROCHEMICAL REACTIONS

Investigator: J. M. Sherfey

Scope of Work

The use of electrochemical calorimetry to study the heat effects of dynamic electrochemical reactions offers a new approach to the investigation of polarization, half-cell processes and complex-ion equilibria and to the determination of heats of reaction. This approach shows promise of leading to significant contributions to the field of electrochemistry. Most of the work has been devoted to a study of the system $CaCN-NaCN-H_2O$. Preliminary values have been obtained for the two equilibrium constants which govern this system, for the heats of the reactions involved, and for the heats of formation of the various species present. A more accurate evaluation of these data is in process.

Contractor: Oregon, University of, Eugene, Oregon

Contract: AT(45-1)-535

Brief Title: ELECTROCHEMICAL AND POLAROGRAPHIC STUDIES ON THE CORROSION OF ZIRCONIUM

Investigator: George B. Adams, Jr.

Scope of Work

Interesting photogalvanic effects with anodic oxide films formed on zirconium have been observed, using ultra-violet light. A study of these effects in dilute aqueous sodium sulfate is now being undertaken.

Work is under way on a study of the activation energy of the anodic oxidation process on pure zirconium as a function of the electric field strength.

Work is being concluded on the current efficiency of the anodic oxidation process for pure zirconium in both the low and high potential ranges.

A study of the temperature dependence of the Tafel slope for the formation of very thin anodic oxide films on pure zirconium is contemplated. The Mott-Cabrera theory for very thin oxide film formation requires a direct proportionality of Tafel slope with absolute temperature. A direct test of this theory for very thin anodic oxide film formation on pure zirconium would be possible.

Contractor: Missouri, University of, School of Mines and Metallurgy, 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

In the first year and probably in the second, zirconium will be investigated, and the investigation will cover the following topics:

1. Rate of dissolution of purest Zr in HF of many concentrations.
2. Rate of dissolution of Hf-containing Zr in HF.
3. Passivation of Zr by HNO_3 and by water soluble fluorides.
4. Rate of dissolution upon addition of salts of more noble metals to HF.
5. Rates of dissolution in other acids.
6. The difference effect.
7. Potential measurements of the Zr electrode at a current = 0 and under current.
8. Preparation of ZrO_x alloys and their properties.
9. Determination of free Zr or of oxygen in ZrO_x alloys.
10. Rate of dissolution of ZrO_x in HF and other acids.
11. Electrochemical potentials of ZrO_x alloys.
12. Preparation of ZrN_x alloys.
13. Reaction of ZrN_x with HF and other acids.
14. Rates of dissolution and potentials of ZrN_x alloys.

The same problems will be carried out using Hf, Be and U after a complete investigation of Zr.

Contractor: Knolls Atomic Power Laboratory, Schenectady, New York

Contract: W-31-109-Eng-52

Brief Title: LIQUID METALS

Investigator: Leo F. Epstein

Scope of Work

Corrosion of solid metals by liquid metals has been shown to be essentially a solution process in many important cases. The rate determining step may be fixed by (1) the rate of diffusion of the solute in the liquid (example: iron in mercury); or (2) by some chemical reaction at the solid-liquid interface (example: iron in sodium).

In the first (diffusion limited) case, the exact functional dependence of the solution rate constant and the diffusion coefficient and such factors as flow velocities, the geometry of the system, etc. are still somewhat obscure, so that order of magnitude agreement between observed and computed corrosion rates is about as much as can be achieved currently. More data on this, as well as on the chemical reaction limited corrosion process are required in order to improve the theory of liquid metal corrosion and thereby provide a reliable working tool for engineers in the design of liquid metal systems. The initial investigations will be concerned with corrosion by liquid mercury of solid metals with relatively high solubility, such as copper, silver, and gold.

Contractor: Knolls Atomic Power Laboratory, Schenectady, New York

Contract: W-31-109-Eng-52

Brief Title: ALUMINUM CORROSION STUDIES

Investigator: R. M. Haag

Scope of Work

The objective of this program is a fundamental understanding of the process of corrosion of metals in high temperature water. This should contribute to the development of better, more corrosion-resistant materials. The work is concentrated largely on the nature of the corrosion product film and its role in protecting the base metal from further attack. At present, emphasis is being placed on aluminum and its alloys.

The products of corrosion of aluminum are hydrous oxides of greater or lesser degrees of hydration. The equilibrium properties of the $Al_2O_3-H_2O$ system are being studied. Decomposition pressures of the various phases are being measured as a function of temperature and composition.

The application of infra-red spectral analysis to corrosion studies is being developed. Measurements have been made of bulk oxide mechanically removed from corrosion coupons, of corrosion product removed by dissolution of the basis metal in a methanol-iodine solution and of corrosion products in situ. Measurements have been made of material pelletized in KBr, of the film itself when self-supporting and finally by reflectance from the basis metal. The latter technique allows non-destructive analysis of the corrosion product film.

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, S. Greenberg, and R. D. Misch

Scope of Work

The over-all purpose of this program is to determine mechanisms and controlling factors in the aqueous corrosion of reactor materials. Since all these materials rely on corrosion product oxide films for protection, the basic objective of the program is to learn the important features of the processes in which corrosion proceeds through such oxides. In general, all of the types of studies will be extended to all metals of interest. At this time, however, most measurements are restricted to single metals.

All phases of the kinetics of the corrosion of commercially pure aluminum (alloy 1100) are being studied in distilled water and very dilute solutions below the boiling point. This includes the determination of corrosion rates and the effects of solutes on same, the effects of dissimilar metals in contact with the aluminum, and the measurement of pH changes close to the corroding metal surface.

Further studies of the corrosion of the same metal in the same environments are being made by measurements of electrical potentials and the changes caused by corrosion currents. The techniques used in these polarization measurements were originally devised at Argonne.

The corrosion of aluminum, zirconium, and uranium are being studied at elevated temperatures. For aluminum and uranium and perhaps for zirconium, corrosion damage appears to be amplified by some of the hydrogen produced as a corrosion product. This type of damage is strongly reduced or eliminated by the formation and maintenance of small amounts of strongly cathodic second phases on the metal surface (to provide special sites for the liberation of hydrogen). This is conveniently done by careful alloying. Studies of special alloys from this point of view are being made for aluminum and uranium.

After prevention of hydrogen damage, it is desirable to provide lower corrosion rates, particularly for aluminum alloys. Mechanisms of inhibition of these metals are therefore being investigated.

The corrosion of zirconium and its alloys is being studied by means of the properties of the corrosion product oxide formed. The electronic resistance of oxide on metal surface is being investigated as related to the mode of oxide formation, and the composition of the base metal. Correlation with corrosion behavior will be attempted. Special alloys in this program are being made from the point of view of altering the defect structure of the corrosion product oxide.

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

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

Brief Title: RECRYSTALLIZATION AND SINTERING OF OXIDES

Investigator: Ivan B. Cutler

Scope of Work

The purpose of this research program is to investigate the phenomena of recrystallization in connection with the sintering of oxides. A main objective is the study of the rate of recrystallization as compared to the rate of densification of oxides. Nucleation of the recrystallized phase as well as the rate of nuclei growth is of interest to the investigation. Variables being studied are original grain size, final grain size, compaction, time, temperature, etc., on such systems as alumina, copper oxide, magnesia.

Additional research is being carried out on the effect of additive oxides on the systems outlined above. Additive oxides have been reported to alter remarkably the densification and recrystallization of sintered oxides. Resistivity measurements are being planned to augment the study of the mechanism of densification, recrystallization and the role of additives.

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

Contract: NBS Project No. 0901-11-4400

Brief Title: PROPERTIES OF CERAMICS AT ELEVATED TEMPERATURES

Investigator: M. D. Burdick

Scope of Work

One phase of this study is concerned with the development of the optimum strength and density, and an evaluation of the various factors affecting the strength and other physical properties of porous, brittle materials, as applied specifically to UO_2 and ThO_2.

Recent work on ThO_2 has shown the existence of a definite relationship between porosity, grain size, and flexural or compressive strength of these materials, and a general empirical equation relating these variables has been proposed. The present study is concerned with a more precise determination of the values of the empirical constants of the specific equations for the compressive and flexural strengths at room temperature and for the flexural strength at 1000°C.

The study of UO_2 is concerned with the same primary goal but, because of the non-stoichiometric nature of the so-called stable oxide, another method of attack to the problem is being explored. In this, a statistical solution is being used to evaluate the relative strength properties of some five varieties of UO_2 as related to their grain sizes and porosities. In addition, it is planned to determine the internal friction, modulus of elasticity, and the electrical resistivity of these various types of UO_2 specimens as a function of temperature to temperatures in excess of 1000°C.

The second phase of this study is concerned with the determination, by sonic methods, of the temperature dependence at temperatures in excess of 1000°C of the elastic constants—Young's modulus, shear modulus, Poisson's ratio, and bulk modulus—of a great variety of high-temperature, ceramic-type, materials including oxides, carbides, borides, silicides, cermets, and intermetallics that are potentially useful in nuclear reactors.

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 and A. G. Tharp

Scope of Work

Scope I - Sintering Behavior of Refractory Ceramic Materials

It is known that the sintering behavior of certain ceramic materials and the high temperature reactions between others are affected by the furnace atmosphere. For example, uranium oxide does not sinter well in air or oxygen; beryllia is affected at high temperatures by presence of water vapor; zirconia containing calcium oxide transforms to a cubic structure when heated in air but not in hydrogen. This study consists of a systematic investigation of the firing, individually and in binary combinations, of certain oxides, carbides and nitrides of interest. The density, strength, and stability of the sintered compact are correlated with the types of interaction as revealed by petrographic and x-ray diffraction examination to test current theories of the mechanism of sintering.

Scope II - Nature of Thorium Oxide Suspensions

The behavior of thorium oxide suspensions, particularly in water, is affected by the presence of very small amounts of various ions from impurities in the water and in the thorium oxide itself. Surface reactions of the thorium oxide with the water probably have an effect also. The first phase of this project is directed at determining, mainly by electro dialysis, the ions that are normally responsible for the anomalous behavior of thorium oxide suspensions. A later phase will involve an investigation of the effects of these and other ions individually and in combination.

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

Measurements are being made of the free energies and heats of formation of several compounds of thorium and uranium. Attention is centered at present on the silicides of these elements. Later, the thermodynamic properties of the sulfides, nitrides, and germanides will be determined. The data will be useful in predicting the possible course of high temperature reactions and in estimating the strength of the bonds formed in these compounds. High vacuum induction heating is used to determine the vapor pressure for the uranium and thorium silicides by the Knudsen effusion method.

Scope IV - Structure of Some Rare Earth Intermetallic Compounds

A study is being made of the methods of preparation and the structures of the silicides and carbides of samarium, europium, and gadolinium. From the theoretical point of view it is of interest to compare the lanthanide silicides and carbides with those of the Group IVA and the actinide series of elements. One may expect to get some correlation between the relative importance of atom size and the bonding directional properties of silicon and carbon is forming a compound of a given structure. The compounds are being prepared by reducing the rare earth oxide with either aluminum or carbon and at same time reacting it with silicon if the silicide is desired. The lower silicide is then prepared by volatilizing the silicon.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: CERAMIC RESEARCH

Investigators: J. H. Hardwerk, L. L. Abernathy, and G. C. Kuczynski*

Scope of Work

Scope I - Sintering of Ceramic Materials

This study is concerned with the determination of the mechanism or mechanisms by which ceramic materials sinter, i.e., density during firing at temperatures below their melting points. Work is now in progress studying the sintering together of alumina rods and spheres. Future work will be extended to study the sintering of silica, urania, thoria, magnesia, zinc oxide, and combinations of these materials.

Scope II - Properties of Oxide Single Crystals

Studies of the basic physical properties of oxide single crystals have been initiated. The mechanisms of plastic deformation, and measurement of creep in magnesia, urania, and thoria single crystals are being studied and measured. The measurement of stress-induced vitrification in alumina and silica single crystals is also receiving attention.

Scope III - Phase Equilibrium Studies of Oxide Systems

The orderly study of the high temperature reactions of the UO_2-ThO_2-MgO phase system is being investigated. This work will be extended to include other systems such as UO_2-ThO_2-CaO and UO_2-ThO_2 and rare earth oxides.

*Consultant, University of Notre Dame.

Contractor: Knolls Atomic Power Laboratory, Schenectady, New York

Contract: W-31-109-Eng-52

Brief Title: HIGH TEMPERATURE REACTIONS IN CERAMICS

Investigator: L. G. Wisnyi

Scope of Work

The high temperature behavior of ceramic materials is being investigated with emphasis on melting points, decomposition ranges, phase transformations and chemical reactions.

Various methods are being employed by which the above phenomena are studied. For the determination of melting points and decomposition ranges a refractory metal ribbon furnace has been used successfully up to 3000°C. Temperature is controlled manually and measured with an optical pyrometer while any significant changes are observed visually. With reference to melting point determinations the work has included study of refractory oxides of interest to nuclear technology with special emphasis on uranium dioxide (2760 ± 30°C). In the investigation of the thermal stability of various uranium fuel compounds the decomposition temperatures as well as the decomposition products were observed and detected for the alkali and alkaline earth uranates.

Turning to the more difficult problem of phase formations and chemical reactions at high temperature a large vacuum tungsten tube furnace suitable for thermal analysis work has been constructed and is being used with a fair amount of success up to 2000°C. Temperatures have been measured by utilizing a tungsten-molybdenum thermocouple; it is planned to go to higher temperatures with more refractory metal thermocouples such as a tungsten-tantalum couple. Calibration work has been completed and the melting point of strontium aluminate has been determined; in addition single crystals were obtained for subsequent X-ray analysis and petrographic studies.

Contractor: (The) 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.

Benzene, toluene, xylenes, methane, biphenyl, naphthalene, pyridine, and thiophene have been carbonized between 900° to 1400°C., using heating times from 10 to 68 seconds. The various carbons produced are then graphitized up to 3000°C. and their X-ray parameters compared.

The physical structure of carbons determined by X-ray scattering and gas absorption are compared. The diffusion of gases through porous carbon bodies as a function of temperature, small carbon burn-offs, and initial carbon porosity are being investigated. Mercury porosimetry, electrical resistivity, and helium displacement are also used to characterize the carbons.

The reactivity of carbons to carbon dioxide and oxygen are being investigated in the low-burn-off (up to three per cent), low-temperature range (500 to 900°C.) using a microbalance and in the high burn-off (up to 50 per cent), high-temperature range (850 to 1400°C.) using a flow reactor. Quantitative and qualitative data on gas release from carbon as a function of carbon type and temperature is being determined by gravimetric and mass spectrometric techniques.

Contractor: Buffalo, University of, Buffalo, New York

Contract: AT(30-1)-1440

Brief Title: BASIC PRINCIPLES OF MANUFACTURE OF CARBONS

Investigator: Stanislaw Mrozowski

Scope of Work

The purpose of this research is to discover and to study any physical laws involved and any physical changes occurring in the manufacturing process of carbons, in order to understand the nature of processes occurring and to be able to use this information for preparation of carbons with required properties. At present the main effort in this project is being directed to the study of the dependence of the physical properties of carbons on the type of porosity introduced in the manufacturing process (particle size, binder content, etc.) in order to check the applicability of theoretically derived relationships.

Additional studies include: changes occurring in the length of samples during the heat treatment process; the thermal expansion coefficient in relation to the composition of a carbon; and the elastic modulus of carbons in relation to the same variables.

Section II

ALLOY THEORY AND THE NATURE OF SOLIDS

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

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

Title: ELECTRON ENERGY STATES IN THORIUM, URANIUM, AND PLUTONIUM

Investigator: John E. Hove

Scope of Work

The nature of the electronic structure of thorium, uranium, and plutonium is being determined from measurements of the electrical conductivity, the Hall coefficient, the magnetoresistivity, and the thermoelectric power. These data are being obtained as a function of temperature, down to 1.1°K, and for the cases involving magnetic properties, as a function of field, up to 33 kilogauss. A concurrent theoretical program is being carried out on the electron band structure of these metals. The approximation method used in these calculations is a Slater-type interpolation scheme derived on the basis of a variational treatment of the cellular method.

One objective of this study is to understand the factors which contribute to the striking structural changes that occur from one member of the series to another, and that occur in a given member as a function of temperature. In addition, it is hoped to understand the unusual behavior of uranium at low temperatures (less than 50°K) where the Hall coefficient drops by a factor of about three, the volume thermal expansion coefficient is negative, and the thermoelectric power shows a reverse in the slope with temperature. A particular attempt will be made to understand the allowing characteristics of the three metals in those systems of scientific and engineering interest.

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 J. Korringa

Scope of Work

The purpose of the project is to make experimental and theoretical studies of the characteristic emission and absorption spectra of the four elements titanium, zirconium, hafnium, and thorium, and their alloys. This work is being coordinated with that in the metallurgy department of ORNL, which is furnishing the pure metals and such alloys as are of significance.

The scope of the work in the experimental direction is to make spectral measurements under the highest resolving power and accuracy of intensity measurement that are feasible in the intermediate wavelength range ($5 < \lambda < 15$ Angstroms), and to plan and construct an instrument in the long wavelength region ($50 < \lambda < 1000$ Angstroms).

The scope of the work in the theoretical direction is to correlate the experimental findings with established theory where possible, in an effort to understand the effect of alloying on the electronic band structure. Conversely, an understanding of the electronic band structure should lead to better understanding of mechanical, thermal, and electrical properties of these alloys.

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, T. Riste, R. Nathans, B. C. Frazer, G. Shirane, H. R. Danner, and R. Pepinsky

Scope of Work

Neutron Diffraction Research

The diffraction program has centered around the use of neutrons for studies of magnetic, ferroelectric and antiferroelectric crystals.

Scope I - Magnetic Crystals

The ordering of magnetic spins has been studied in antiferromagnetic CoO. Above the transition temperature, T_c, the magnetic Bragg peaks, which occur as superlattice lines, are absent. The diffuse magnetic peaks are located around the corresponding Bragg peaks and can therefore be studied without the presence of the much stronger nuclear peaks, as is the case for ferromagnetic substances. The diffuse peaks behave qualitatively in the same way as in ferromagnetics. At T_c they reach maximum intensity and minimum width in agreement with the theory of Van Hove. In the static approximation one finds that the short range part of the correlation has a very weak temperature variation. The pronounced increase in the diffuse intensity at T_c is caused by an increase in the long range part of the pair correlation.

In a number of alloy systems discontinuities have been found in the magnetic moment vs. composition curve at the onset of a superlattice. Preliminary neutron diffraction studies on ordered Fe_3Al indicate that the value of the moments of the iron atoms depend on the nearest neighbor population. For those iron atoms surrounded completely by iron nearest neighbors a value of approximately 2.2 μ_B is observed, whereas the other iron atoms in the lattice, with 50% aluminum and 50% iron as nearest neighbors, possess roughly half the moment.

A neutron diffraction study of the magnetic structure of Fe_4N has been completed. This crystal consists of a nitrogen atom at the body-center position in a face-centered cubic cell of iron atoms. Denoting the Fe magnetic moments at the corner and face positions by μ_1 and μ_2 respectively, the magnetic structures is $\mu_1 = 3$, $\mu_2 = 2$.

Scope II - Ferroelectric Crystals

The study of ferroelectric $BaTiO_3$ has been continued. Additional single crystal neutron data have been collected for the tetragonal phase and several refinement calculations have been carried out. Somewhat more satisfactory thermal vibrational parameters have been obtained, but the atomic positional parameters have remained nearly the same as those reported previously. When measured relative to Ba, the shifts (from cubic positions) along the ferroelectric c-direction are about 0.06 A for Ti, -0.09 A for the O above Ti, and -0.06 A for the O's on the side faces of the perovskite cell. The atomic positions of ferroelectric $BaTiO_3$ in its orthorhombic phase were also determined by a single crystal neutron diffraction analysis. Pillar shaped crystals with their major axes along the cubic (110) direction were maintained as single domains by the application of an electric field. The structure obtained can be viewed as a framework of slightly distorted oxygen octahedra in which the central Ti ions are displaced towards one of the octahedron edges (polar axis) by 0.13 A. The Ba ions are also shifted in the same direction but by the smaller amount of 0.07 A. The similarity between this structure and that of the tetragonal phase indicates that Ti plays an essential role in the ferroelectricity of this crystal, as already suggested by Slater.

The structure of $NH_4H_2PO_4$ has been determined in a single-crystal neutron study. There are definite N–H...O bonds in the structure, but the (N–H)'s are about 10° off the N,O line of centers. The oxygens are exerting sufficient influence to distort the NH_4 groups, but not enough to result in linear N–H...O bonds. As expected, the O–H...O system is very similar to that found in neutron studies of KH_2PO_4.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: NEUTRON DIFFRACTION STUDIES

Investigators: S. S. Sidhu, L. Heaton, and N. W. Gingrich*

Scope of Work

Scope I - Structure of Solids

The purpose of this research is to study problems of the solid state of matter by structure determinations using neutron and X-ray diffraction methods which depend on the nuclear and the atomic properties of elements. For example, the positions of light atoms among heavy atoms have been determined by studying metal-hydrogen systems; the existence and the nature of antiferromagnetic structures in copper–manganese alloys and in $Fe_{1-x}S$ have been shown and their effect on the crystallographic structures of these materials is under investigation; binary alloys have been developed by employing positive and negative nuclear scattering amplitudes of metallic elements for which the structure factors are zero and currently the same principles are being extended to the development of isotopic alloys.

Scope II - Structure of Liquids

This work was undertaken to determine the distribution of atoms in the liquid state. Alkali metals were chosen because theoretical considerations concerning liquid structure and interatomic forces have dealt largely with alkali-like atoms. Neutrons offer the advantages over x-rays of low absorption and isotropic scattering amplitude for liquid diffraction studies.

Cesium and rubidium have been investigated in the temperature range from their melting points (28.5°C and 390°C respectively) to 575°C and their atomic distribution functions determined by Fourier analysis. Elemental potassium, sodium and the isotope six of lithium are under study at the present time.

*Resident Research Associate during summer of 1956 on leave from the University of Missouri.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W31-109-eng-38

Brief Title: ALLOY THEORY

Investigators: M. V. Nevitt, A. E. Dwight, and S. S. Sidhu

Scope of Work

Scope I - Structurally Analogous Intermediate Phases

A program devoted to the problem of intermediate phases in transition metal systems is underway, with the CsCl, beta-tungsten, and sigma phases under current scrutiny. These phases exemplify

(a) a simple structure in which an ionic bonding component appears operative, (CsCl),

(b) a structure in which covalent bonds appear active, (beta-tungsten), and

(c) a complex structure in which the nature of bonding is more obscure, (sigma).

The occurrence and range of stability of these phases in a large number of binary systems is being explored to establish the rationale of their stability in terms of size factor and position in the periodic table. Paramagnetic susceptibility and ferromagnetic saturation moment measurements will be made to clarify the nature of the electronic interactions involved.

The effect of various transition element solutes on the course of the beta plus gamma/gamma boundary in certain ternary uranium base alloy systems is also to be studied and correlated with the effect of each solute element on the lattice parameter of gamma uranium.

Scope II - Solubilities and Intermetallic Structures in the Gold–Beryllium and Iron–Osmium Systems

The atomic radii and the valencies of gold and beryllium metals do not favor extended solubility of one metal in the other. Consequently, one would expect, in general, that the two metals would form a number of intermetallic phases. On the other hand, the electronic structures of 3d and 4s shells of iron are identical with those of 5d and 6s of osmium. In the "free" state, each d-shell contains 6 electrons and each s-shell contains 2. The metallic radii of iron, for coordination numbers of 8 and 12, are 6.1 and 6.7 per cent smaller than those of osmium. These factors favor extended solubility of one metal in the other. X-ray diffraction and metallographic studies of the two systems show that the solubility of beryllium in gold is extremely limited, if any. The two metals form 5 intermetallic phases, whose crystal structures are being worked out. Iron and osmium form two solid solutions; iron dissolves in osmium to the extent of 80 atomic per cent, and osmium in iron to the extent of 10 atomic per cent. There are no intermetallic phases in this system.

Contractor: Ames Laboratory, Ames, Iowa

Contract: W-7405-eng-82

Brief Title: SOLID STATE STUDIES

Investigators: G. C. Danielson, P. H. Sidles, F. H. Spedding, S. Legvold, and D. E. Hudson

Scope of Work

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

The general purpose of this investigation is to increase our knowledge of the thermal properties of metals and alloys at high temperatures. The first specific objective is 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 entire temperature range 20°C to 1000°C.

A modified Angstrom method has been developed for measuring the thermal diffusivity to a precision of two percent over the temperature range 20°C to 1000°C. The method has been applied to the pure metals: uranium, thorium, iron, nickel, and copper. Discontinuities associated with Curie temperatures and phase changes are clearly discernible and can be examined quantitatively. A pulse-heating method is being developed for measuring the specific heat of metals from 20°C to 1000°C. The method has been applied to the pure metals: platinum, nickel, and iron. A precision better than two percent seems possible but has not yet been achieved. Densities at high temperatures have been obtained from densities at room temperature corrected for thermal expansion. Expansion coefficients have been determined by X-ray diffraction and by optical methods. Thermal conductivities of nickel and uranium from 100°C to 600°C have been measured by a conventional method in which the temperature gradient in the sample of unknown thermal conductivity is compared with the temperature gradient in a standard iron sample; but neither the temperature range nor the accuracy of this method appears to be adequate.

Scope II - Physical Properties of Semiconducting Metallic Compounds

The general purpose of this investigation is to increase our understanding of electronic conduction in all solids and especially in semiconducting metallic compounds. The first specific objective is to grow single crystals of compounds having a cubic structure similar to that of fluorspar and to investigate the temperature dependence of both electron and hole mobilities.

Single crystals have been grown of Mg_2Si (both n and p type), Mg_2Ge, Mg_2Sn, Mg_2Pb, and Cu_2Se. Electrical resistivities, Hall coefficients, and thermoelectric powers are being made over the temperature range 1°K to 1000°K. Hall mobilities have already shown anomalies in their temperature dependence and a theoretical explanation of such anomalies will be attempted. Thermal, optical, and magnetic measurements are also planned.

Scope III - Physical Properties of Rare-Earth Metals and Alloys

The general purpose of this investigation is to increase our knowledge of solid-state physics by examination and correlation of properties in the closely related elements known as the rare-earth metals.

Measurements of the electrical resistivity of rare-earth metals have revealed that these metals are poor conductors. Hall effect measurements indicate that at least two bands are involved in the conduction process and that the magnetic properties of the shielded 4f electrons have a strong influence on the electrical conductivity. Many of the metals have been found to exhibit paramagnetism, antiferromagnetism, and ferromagnetism as the temperature

is lowered. This is unusual behavior for a metal. Recent measurements on a single crystal of dysprosium reveal anisotropy which is very pronounced at temperatures below 85°K where the metal is ferromagnetic. Parallel to the optic axis of the hexagonal crystal, dysprosium is extremely hard magnetically while perpendicular to the optic axis the metal is readily saturated in modest magnetic fields. Dilatometric and electrical measurements have shown that crystallographic transformations occur just below the melting points and that the metals exhibit plastic flow in the high temperature phase. In alloy work it has been possible to alter the magnetic properties of gadolinium by alloying it with yttrium and to vary the superconducting transition of lanthanum by alloying it with yttrium. A mass spectrometer has been used to measure accurately the relative vapor pressure of a substance as a function of temperature. In this way the heats of sublimation and heats of vaporization of several rare-earth metals have been determined.

Scope IV - Special Topics in Solid State Physics

The general purpose of this investigation is to increase our knowledge of solid-state physics by exploiting unusual properties of materials.

The electrical, optical and counting properties of diamond have been studied in order to determine impurity and trapping levels in diamond. The electrical characteristics of boron single crystals have shown that the mobilities of the charge carriers are very low and that the hole mobility is greater than the electron mobility. 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; but the anomalous minimum in the curve of resistivity versus concentration is still unexplained. 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.

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: C. S. Smith

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.

The system of research by projects is not viewed as the most suitable in these areas of basic research; also, the training of highly competent research personnel is better accomplished in the more free and fluid atmosphere which prevails at present in the Institute. We therefore conduct a rather broad area of basic research in metals, solid-state physics, and physical chemistry. Present activities include work on diffusion in metals and ionic crystals, antiferromagnetism at low temperatures, structural transformations, plasticity and fracture in metals, the thermodynamics of liquid metallic systems, studies on the superconducting state, and experimental and theoretical work on semi-conductors, particularly InSb. Theoretical interest in the properties of the Group V semi-metals is expected to continue.

Contractor: Colorado, University of, Boulder, Colorado

Contract: AT(11-1)-377

Brief Title: RESEARCH ON METALS AND ALLOYS AT LOW TEMPERATURES

Investigator: William F. Love

Scope of Work

The work under this contract is concerned with the investigation of magnetic effects in metals and alloys at low temperatures. Measurements of the Hall effect in copper and copper–gold alloys down to liquid helium temperatures and in magnetic fields up to 9,500 gauss are in progress. Future work on the Hall effect will be done on oriented single crystals of pure metals. New experimental apparatus for the investigation of magnetic effects in solids at very large transient magnetic fields and low temperatures is being constructed. The fields will be produced by the discharge of a bank of large capacity, high voltage capacitors through a specially constructed solenoid. The power supply is a large industrial spot welder control manufactured by Westinghouse. This supply has a bank of capacitors of total capacitance 2640 μf rated at 3000 volts. Fields of the order of one megagauss are being planned using a modification of the power supply to utilize it as a Marx surge generator. The experiments initially planned are to measure the magneto-resistance of metals and alloys at these large magnetic fields and at low temperatures.

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 silver, cadmium, indium, tin, and antimony from Period 5 and with lead from Period 6. Experimental studies using iodide zirconium are being made of the phase boundaries, the axial ratio of the close-packed hexagonal phase, and the electronic specific heats at helium temperatures. The interrelationship of these properties will provide a test of a current theory of alloying based on electron concentration. Purification of zirconium by zone refining promises to provide a source of alloys of still greater purity.

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 rearrangements 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 function of amount of deformation, to determine the paths of movement of the reference axes toward stable end 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.

Scope III - Low and High Temperature Crystal Structures

X-ray diffraction facilities for studies of structures in the range 75°K to 1800°K are available. Equipment under construction will extend the range down to 4°K. 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. Thorium shows an anomalous change in lattice parameter near 75°K. Investigation of the supposed allotropic transformation in vanadium is in progress. The elevated temperature program is currently concerned with the structure of cerium and the hydrides of titanium and zirconium.

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

Contract: W-7405-Eng-26

Brief Title: OXIDE FILM FORMATION ON METALS

Investigator: 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 may be used as a basis for predicting the protective or non-protective nature of an oxide. The inadequacy of this theory has been demonstrated by studies of the oxidation behavior of the alkali metals and niobium. The former exhibit protective oxide formation, whereas the latter oxidizes nonprotectively, both in contradiction to theoretical predictions. Investigation of these metals is being continued with emphasis being placed on an electron optical study of the details of the oxidation process in an effort to determine the general conditions necessary for protective oxide formation.

Scope II - Radiation Effects on Oxidation

This research consists of a phenomenological study of the changes induced in the oxidation process by a radiation field. The first phase of the work is directed at separating the effects of neutron, beta, and gamma radiation.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-Gen-359

Brief Title: LATTICE IMPERFECTIONS

Investigator: 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 in use and under development are those of small angle X-ray scattering studies and fine-focus studies of individual Bragg reflections.

During the past year the former technique has been successfully applied to imperfections which correspond to surface irregularities. It has also been used to study the effects of irradiation and subsequent annealing on NaCl crystals, the conclusion arrived at being that such treatments do not give rise to defect clusters larger than 20 Angstroms. Fine-focus equipment has been developed to give a high resolving power such that dislocation densities of about $10^6/cm^2$ should be observable.

It is planned to apply the small angle technique to "pre-melting" phenomena, to zinc single crystals containing small angle dislocation boundaries, and to irradiated lithium-containing materials where fission may give large regions of local damage. The fine-focus equipment is to be used to obtain quantitative data on polygonization in various metals and to examine the interaction between dislocations and ferromagnetic domain walls in iron–silicon single crystals.

Contractor: Virginia, University of, Department of Chemistry, Charlottesville, Virginia

Contract: AT(40-1)-1768

Brief Title: GROWTH AND CHEMICAL PROPERTIES OF NEARLY PERFECT CRYSTALS

Investigator: 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.

By using several methods of growing crystals—that is, from the vapor, the melt, and from solution—and by controlling accurately such variables as purity, temperature, and the degree of supersaturation, it is hoped to be able to control the number of screw dislocations and to eliminate or to control other types of imperfections such as vacancies. Emphasis will first be placed on trying to reduce to a minimum dislocations in copper crystals grown from the melt. It is believed that screw dislocations can best be controlled in metals by growing small crystals from the vapor, and this will be attempted. Most of the studies will be carried out with copper.

Contractor: California, University of, Berkeley, California

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

Brief Title: ORIGIN AND EFFECTS OF DISLOCATIONS

Investigator: J. Washburn

Scope of Work

The purpose of the project is to obtain information concerning the origin of dislocation networks in crystals, and to correlate mechanical properties with dislocation density and distribution.

As a start toward this broad objective, two kinds of experiment are being developed. The first is an investigation of the dependence of dislocation density on the rate of solidification and direction of growth relative to crystallographic direction. A furnace has been constructed which by virtue of a steep temperature gradient (1000°C/cm) permits growth of crystals at varying rates (1 to 100 cm/hr) keeping a substantially constant temperature gradient across the liquid solid interface.

The second phase of the program is a study of the effect of growth conditions on the propagation of a known array of dislocations existing in a seed crystal into new layers of crystal solidified from the melt. Single crystals of sodium chloride and zinc which have been plastically deformed so as to introduce known arrays of edge or screw dislocations are partially melted and then resolidified under conditions such that nucleation of new crystals does not occur. The structure of the melted and resolidified part of the crystal can then be compared with that of the unmelted part.

Dislocation density and distribution will be determined by etch-pit methods, high resolution X-ray techniques, and optical examination of cleaved surfaces.

Contractor: Pittsburgh, University of, Pittsburgh, Pennsylvania

Contract: AT(30-1)-647

Brief Title: APPLICATION OF CHEMICAL THERMODYNAMICS TO THE STUDY OF ALLOYS

Investigator: W. E. Wallace

Scope of Work

The program has as its goals (1) the devising and development of suitable experimental procedures for high precision thermochemical studies of alloy systems and (2) the accumulation of a body of data to serve as a basis for the study of bonding in metallic systems. The ultimate objective is the understanding of the nature of the forces acting between the particles in solids and the interpretation of structure and energetics in terms of these forces.

Studies involving four systems constitute the bulk of the present program. These systems are Mg–Cd, Ta–H, $MgNi_2$–$MgCu_2$, and KCl–KBr. Calorimetric investigation of the kinetics of disordering of $MgCd_3$ and MgCd is under way. In addition measurements are nearing completion which will give the zero point entropy of the intermetallic compounds $MgCd_3$, MgCd and Mg_3Cd. Calorimetric determination of the residual entropies of KCl–KBr will shortly be begun. Electrical conductivities of KCl–KBr solid solutions are being measured to ascertain the concentration of vacancies in this system. Specific heats through the Curie point and saturation magnetic moments are being measured for the pseudo-binary systems $MgNi_2$–$MgCu_2$. Hydrogen vapor pressures over the Ta–H system are being measured to permit the heats, free energies and entropies of formation of this system to be evaluated. In addition, the phase boundaries between the terminal solid solution and the intermediate phase Ta_2H, which precipitates out below 52°C, are being located.

Contractor: Tennessee, University of, Knoxville, Tennessee

Contract: AT(40-1)-1068

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

Investigator: E. E. Stansbury

Scope of Work

A program utilizing an adiabatic calorimeter for operation from 50°C to 1000°C for the measurement of certain thermodynamic properties of metals and alloys is in progress. Specimens (50-200 grams) are heated internally and continuously to yield energy absorption-temperature data. Isothermal and other techniques of operation are possible under certain circumstances. Measurements which have been made include specific heats, heats of transformation, heats of solution in the solid state, and heat effects associated with non-equilibrium states. The precision of the measurements is generally better than one percent, at least below 700°C.

Investigations recently completed include specific heats of pure copper and nickel to 650°C; specific heats of iron, zirconium, and titanium to 950°C; specific heats of several copper–nickel alloys; specific heats of several zirconium base and iron base alloys. Heats of transformation have been measured for iron, zirconium, titanium, and several of the alloys.

The objectives of the research are to provide thermodynamic data of general applicability and specific data for theoretical studies of kinetics of phase transformations and of alloy formation.

Contractor: California, University of, Berkeley, California

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

Brief Title: THERMODYNAMIC FUNCTIONS OF THE METALLIC STATE

Investigator: R. Hultgren

Scope of Work

This project has the aim of collecting from the scientific literature the thermodynamic data of alloy systems, critically examining them, and selecting and publishing the most reliable values of the thermodynamic functions.

From diverse data, such as vapor pressures, electrochemical potentials, chemical equilibrium constants, heats of formation, and heat capacities, the thermodynamic functions are calculated. Self-consistency and consistency with the work of others and with the phase diagram are carefully examined. Contradictions are frequently found and unreliable data discarded. Best values are selected and probable accuracy estimated. Tabular values and a summary of reasons for the selections are multilithed and distributed.

Since June 1956, the systems Au, Mg, Mn, Ag–Cd, and Cd–Mg have been completed. Substantial progress has been made on the systems Cr, Na, Zr, Cu–Zn, Au–Ni, Cu–Pt, and Fe–Mn.

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

The energy effects associated with plastic deformation and solid-state reactions are being investigated by means of a calorimetric technique of considerable precision which employs tin as a solvent. The energy changes are correlated with other measurements such as x-ray diffraction, microhardness, and electrical resistivity to enhance the understanding of the phenomena.

Investigation of the following topics is planned:

1. Determination of the stored energy of cold work as a function of strain and strain rate for gold-silver alloys deformed at room temperature and at −195°C.
2. Investigation of the effect of thermal cycling on the stored energy of cold worked metal.
3. Determination of the energy stored in such processes as impact, low temperature creep, and deformation at a temperature slightly above room temperature.
4. Study of the effects of cold work and annealing on ordered and disordered Cu_3Au and the energies of formation and ordering of $CuAu_3$ and CuAu.
5. Measurement of the heats of formation of III-V compounds.
6. Exploration of a new calorimeter solvent, specifically bismuth and lead.

An associated study (G. Scatchard) is concerned with vapor pressure measurements in binary and ternary alloys in the Ag−Cd−Zn system and the zinc vapor pressure in the Cu−Ni−Zn ternary.

Scope II - Thermodynamics and Structure of Solid Solutions

The atomic arrangements and the sizes of the atoms in solution are being observed in solid solutions, and the thermodynamic properties of these same solid solutions are being determined. The combined information has been very revealing, and has indicated the inadequacy of the existing theories of solid solutions. It now appears that the sizes of the atoms in solution cannot be predicted from a knowledge of the lattice parameters, and the contribution of the strain energy associated with the close packing of atoms with disparate sizes has not been calculated in a satisfactory manner.

Considerable emphasis has been placed during the last year on the development of a new x-ray method of measuring the mean displacements of atoms from the average lattice sites. For the past two years, data on atomic sizes have been derived in this research program from the size coefficients obtained from an analysis of the diffuse x-ray scattering. The new method involves the determination of the static displacements of the atoms from the average lattice sites using measurements of the integrated intensities of the diffraction lines. Data on the same systems using both methods have now been obtained, and the atomic displacements calculated from each set of measurements are quite consistent. The new x-ray technique has some important advantages since atomic displacements can be observed in alloys such as those of copper−zinc, where the difference in scattering power is small.

Scope III - Fundamentals of Cold Work and Recrystallization

The types of imperfections and the mechanisms of plastic deformation and of subsequent recovery and recrystallization processes are being studied by x-ray methods. The principal x-ray technique involves the careful measurement of diffraction line shapes and the representation of these shapes by means of a Fourier series. The coefficients of this series are subject to interpretation, and measurements of subgrain sizes, local strains and the prevalence of stacking faults are accomplished without introducing another model of the cold worked structure a priori. The line shape method allows observations on heavily deformed metals. Highly deformed metals are being studied by means of double crystal rocking curves. Single crystals of zinc are being bent at several temperatures down to liquid nitrogen temperature, while in the parallel position in a double crystal spectrometer, and the subgrain formation is being observed on bending and after annealing.

The line shape analysis has also been applied to the study of imperfections in phases formed by martensitic transformations. In the case of iron–nickel martensite, the imperfections are found to be of the same order as those observed in heavily cold worked metals. Similar types of imperfections are being observed in cobalt and cobalt–nickel sites and it is expected that the mechanical properties of these transformation products may be greatly dependent on the character of these imperfections.

Contractor: Illinois, University of, Urbana, Illinois

Contract: AT(11-1)-67, Project #15

Brief Title: STUDY OF DIFFUSIONLESS PHASE CHANGES IN SOLID METALS AND ALLOYS

Investigator: T. A. Read

Scope of Work

This research on diffusionless phase changes in non-ferrous metals has been concerned primarily with the gold–cadmium system since its transformation exhibits the following attractive features: (1) the transformation has a high degree of reversibility such that a single crystal can be heated and cooled through the transformation temperature indefinitely without loss of its single crystal character, and (2) the inhomogeneous shear involved in the transformation occurs on a scale of dimensions sufficiently coarse for it to be readily observable and accurately measurable.

A prominent feature of the orthorhombic and tetragonal phases of Au–Cd alloys is the mobility of the twin and domain interfaces under the influence of applied stress. This phenomenon and the stabilization effect of aging are being studied by a combination of stress-strain measurements, observations of interface motion by optical microscopy, and Laue x-ray diffraction patterns. The influence of impurities and previous thermal history is also being studied.

It has been observed that substantial shifts of diffraction line positions in patterns obtained with powdered samples of Au–Cd alloys occur with time at room temperature after transformation, as well as with repeated transformation. The possibility of lattice imperfections generated by plastic deformation or some other process is being investigated by means of a recording x-ray spectrometer.

The theory of the crystallography of transformation in this system leads to the prediction that the mode of transformation of a single crystal in the form of a thin foil should be basically different from a bulk single crystal specimen. Experiments are being performed to check the expectation that the thin foil will transform to a single crystal of the low temperature phase rather than a twinned crystal.

Contractor: Minnesota, University of, School of Mines and Metallurgy, Minneapolis, Minnesota

Contract: AT(11-1)-477

Brief Title: SOLID SOLUBILITY OF INTERSTITIAL ELEMENTS IN TRANSITION METALS

Investigator: Morris E. Nicholson

Scope of Work

It is generally believed that the most important factor which limits solid solubility of interstitial elements in metals is the strain energy associated with introducing an interstitial element into the lattice. Recently it has been found that the solid solubility of carbon in nickel–copper alloys changes abruptly at about 30 atomic per cent copper. This is not readily explainable if strain energy of solution is the only important factor. One explanation of this is that the solid solubility is of interstitial elements in transition elements is influenced very strongly by the density of states of the 3-d electrons in this system. In order to determine whether or not the theory is a satisfactory one, the study of solubility of interstitial elements will be extended to the Co–Ni alloy system in which the solid solubility of both C and N will be determined.

The solid solubility of carbon in cobalt–nickel alloys will be studied by exposing cobalt nickel alloys of several compositions to a CO/CO_2 atmosphere. In the case of the study of solid solubility of nitrogen in cobalt–nickel alloys, the several alloys will be nitrided in an ammonia atmosphere. The solid solubility limits will be determined chemically.

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

Investigator: J. E. Hove

Scope of Work

This work embodies an effort to understand the basic mechanisms whereby thermal energy and matter are transported through a crystalline solid. The emphasis, at least for initial phases, will be on the high temperature processes, which are not well known and which also have obvious technological importance. Such items as the high temperature thermal conductivity in ceramics (above 1500°C) will be investigated, particularly with regard to explaining the anomalous increase in some material at these temperatures, as well as the dependence of the thermal conductivity on porosity and type of atomic structure. The electrical resistivity will be simultaneously measured to separate electronic from lattice heat conduction. In addition, studies of atomic structure and dynamics will be made as needed to help understand the high temperature behavior. Another phase of the program deals with mass diffusion, especially in metals and alloys, under the influence of a strong temperature gradient. The initial part of this work is a study of thermal self-diffusion in a pure metal and later work will be concerned with binary systems.

Contractor: Illinois, University of, Urbana, Illinois

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

Brief Title: DIFFUSION IN METALS

Investigators: Frederick Seitz and David Lazarus

Scope of Work

The program is concerned with experimental and theoretical studies of the fundamental mechanisms for diffusion in metallic systems. The experimental techniques involve precision sectioning experiments using radioactive tracers. To date, particular attention has been devoted to studies involving the noble metals, copper, silver, and gold, and their binary solid solutions. Measurements have been completed of self-diffusion in each of the noble metals, and of diffusion of neighboring electronegative impurities (Cd, In, Sn, Sb) in pure silver and electropositive impurities (Fe, Co, Ni) in pure copper. Electronegative impurities diffuse uniformly more rapidly than the host atoms with lower activation energies, the change in activation energy being proportional to the valence difference between solute and solvent atoms. These data have been interpreted successfully in terms of the screening of the impurity atoms by the free electrons of the host lattice. Electropositive impurities diffuse with higher activation energies and generally smaller diffusion coefficients than the solvent atoms. The screening effects are evidently much more complicated, in this case, since the defect valence results from missing electrons in an otherwise closed d-shell. Studies have been made of diffusion in alpha and beta-brasses, the latter showing a strong dependence on long-range order. Work has been undertaken for the alpha solid solutions of silver with zinc, cadmium and indium, to study the effects on diffusion of varying chemical composition and composition gradient.

Contractor: Rensselaer Polytechnic Institute, Troy, New York

Contract: AT(30-1)-1044

Brief Title: ANISOTROPIC DIFFUSION

Investigator: H. B. Huntington

Scope of Work

Self-diffusion in single crystal antimony is under investigation, using the tracer Sb_{124}. Because of the brittleness of the substance sections are obtained by grinding. Specimens are annealed in pairs in the diffusion furnace, one with trigonal axis parallel, the other perpendicular to the specimen axis. Specimens with axis parallel give the greatest difficulty, not only in their preparation but also in their subsequent handling because of the danger of fracture on the cleavage planes perpendicular to the specimen axis during the grinding of the lateral surfaces. Similar studies are being performed with indium. Here the main effort up to now has been devoted to crystal orientation. The 1.07 tetragonality ratio of this material makes it an interesting one to investigate the influence of small changes in geometry on the jump energy.

A somewhat novel aspect of anisotropic diffusion is being explored in observing the motion of markers in the presence of high currents. Preliminary experiments with currents of the order of 5×10^3 amp/cm^2 through copper wires are under way to observe motion, if any, of markers inscribed on the wire surface. It is hoped that eventually this study may give some direct indication of the interaction between electrons and lattice defects.

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

Contract: AT(40-1)-2036

Brief Title: RESEARCH IN INTERMETALLIC DIFFUSION

Investigator: Lawrence Slifkin

Scope of Work

This work deals with the diffusion of metal atoms in metal crystals, using radioactive tracers to follow the process. The experiments are aimed at increasing our understanding of the mechanism of the phenomenon. The results should be intrepretable on an atomistic basis in terms of crystal imperfections. We hope thereby to learn more about these imperfections and the physics of the metallic state.

The diffusion of various solutes in a given type of crystal has been interpreted from two different points of view. One considers only the valence of the solute and explains experimental results in terms of electrostatic interactions. The second approach considers only the size of the diffusing atom. An assessment of the relative significances of these two factors will be attempted in the present work by measuring the diffusion of rare earth tracers in a given metal. Here, the effects observed may definitely be attributed to the size effect.

Another fundamental problem is the effect of a gradient of chemical concentration on the diffusion coefficient. This will be investigated by comparing the diffusion of tracers in the presence of a concentration gradient with that observed in crystals of the same composition but with zero gradient.

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: F. E. Jaumot, Jr. and R. L. Smith

Scope of Work

This work is concerned with various problems of diffusion in solids. This work can be divided roughly into three categories: (a) a study of the effects of the valence and atomic size of the solute atom on diffusion in solid solutions; (b) an investigation of diffusion of the constituents in defect alloys and compounds; and (c) surface diffusion.

The work on the first item is substantially complete. Specifically, the rates of diffusion of the elements of Group IA and Group IIA of the periodic table in single crystals of silver were studied. The results are described in two previous papers and in a paper to be submitted for publication in the near future.

The interest in defect alloys stems from the need for more detailed and direct evidence for the vacancy mechanism and the need for information concerning the mobilities of defects or vacancies as well as the atoms in such materials. In addition, this study offers the possibility of obtaining separate activation energies for formation and movement of vacancies in alloys.

Surface diffusion is important because of its close relationship to sintering phenomena. Two techniques, a sectioning and a microradiographic technique are being used. The work has progressed to the point that some confidence can be placed in the valuable microradiographic method.

Contractor: Pennsylvania, University of, Philadelphia, Pennsylvania

Contract: AT(30-1)-1879

Brief Title: EFFECT OF PLASTIC STRESSES ON DIFFUSION

Investigator: R. Maddin

Scope of Work

The research is concerned with the effect of stress on the self-diffusion coefficient in silver. The self-diffusion coefficient will be determined in silver single crystals for calibration purposes. Following this, the self-diffusion coefficient will be determined as a function of the amount of strain in torsion for which suitable equipment is now being arranged. It is contemplated also to investigate the effect of strain rate on the self-diffusion coefficient since this variable is believed to be important.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1852

Brief Title: ATOM MOVEMENTS IN CERAMIC OXIDES AT ELEVATED TEMPERATURES

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

Scope of Work

Oxygen ion diffusion will be measured in stable refractory oxides in the temperature range 1300-1900°C by means of exchange measurements with O^{18}. Mass spectrometer techniques will be employed to determine distribution of O^{18} in the system. Exchange will be determined both by decrease of O^{18} in the gas phase and by analysis of the solid phase for O^{18} pickup. Measurements of ionic electrical conductivity will be carried out by utilizing a protective electrolyte to suppress electronic conduction. Transfer numbers will be determined if feasible. Initial measurements will be undertaken for Al_2O_3, MgO, and CaO.

Contractor: Maryland, University of, College Park, Maryland

Contract: AT(40-1)-2068

Brief Title: PROCESSES OF DIFFUSION AND ELECTRICAL CONDUCTION IN SOLIDS

Investigator: Homer W. Schamp, Jr.

Scope of Work

The purpose of the research program 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.

Contractor: Purdue University, Purdue Research Foundation, Lafayette, Indiana

Contract: AT(11-1)-359

Brief Title: DIFFUSION IN LIQUID ALLOYS

Investigator: Richard E. Grace

Scope of Work

The purpose of this project is to study chemical and self-diffusion in liquid mercury and liquid lead alloys by the capillary-reservoir technique. By eliminating concentration gradients and viscosity gradients the applicability of the Einstein and Eyring diffusional mobilities can be evaluated. An added feature of the experiments is that no volume change occurs within the diffusion zone. The viscosity of liquid alloys used in the diffusion experiments is to be measured. The capillary-reservoir technique is being used in order to evaluate whether the Kirkendall effect is operative in liquid alloys. Both the diffusion coefficient of the solvent and that of the solute are to be measured. Experiments on rates of extraction of metallic solutes from dilute liquid alloys by liquid metal solvents are planned in order to investigate diffusional processes in series in heterogeneous liquid alloy systems. An evaluation of thin films operative at liquid metal interfaces is planned.

Contractor: General Electric Company, Research Laboratory, Schenectady, New York

Contract: W-31-109-Eng-52, RD-1

Brief Title: FUNDAMENTAL METALLURGICAL RESEARCH

Investigator: D. Turnbull

Scope of Work

The first part of the project is the carrying out of thermodynamic measurements designed to test critically the theories for metallic solutions. On this program the heats of formation of several alloys as a function of composition and state (i.e., solid vs liquid, ordered vs disordered) are being measured accurately. Some of the systems being investigated are Au–Ni, Cu–Ni, Ag–Cu, AgAu, Au–Cu, Cu–Pt and Al–Ag.

The object of the second part of the project is to determine the effectiveness of dislocation lines and interstitial atoms in short-circuiting the diffusion of interstitial solutes and to obtain information on the interaction energy of interstitial solutes with dislocations and grain boundaries. Specifically the effects of large and small angle grain boundaries on the diffusion of carbon in alpha-iron at low temperatures is being investigated. Also the effect of alpha-iron grain size on carbon solubility will be measured, and the segregation of carbon during long time anneals will be investigated by high resolution autoradiography.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1826

Brief Title: FUNDAMENTAL STUDY OF THE EARLY STAGES OF SINTERING

Investigator: F. N. Rhines

Scope of Work

The objective of this research is to obtain an improved understanding of the course and mechanism of the early stages of sintering as it occurs in metallic powder. Two methods are used to study the structural changes which take place. The fracture technique consists of measuring the size and relative number of interparticle welds as observed on fractured cross sections through the sintered mass. The planar sectioning technique, through the use of a newly devised tangent count, permits quantitative measurement of the curvature of the pore surface.

Progress so far has shown that the number of interparticle welds, measured on unpressed −170 + 200 atomized oxygen-free copper shot sintered in hydrogen at 1011°C, does not increase appreciably in sintering times up to 160 hours. During this time the number of existing weld contacts grow in size but with a diminishing rate.

The radius of the average surface arc of the pore surface passes through a maximum which corresponds in time with the early reduction in density. Moreover, the amount of pore surface area per unit volume reflects this effect.

Emphasis is now upon the development of a "rate curve" in which the sintering process will be defined in terms of available surface energy and curvature of the pore surface.

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 and A. L. Pranatis

Scope of Work

A study is being made of the sintering of pure metals and oxides in order to determine the rate controlling mechanisms under various conditions. An effort is being made to determine the influence of structure on the sintering of metals, oxides, and metal oxide mixtures. It has been shown that grain boundaries play an important role in the sintering of metals. An effort is being made to determine the influence of grain boundary orientation on metal sintering rates. The influence of grain boundaries upon the sintering rate of oxides is being studied. An investigation is being made of the scale factor in sintering and the influence of atmosphere upon sintering rates of metals and oxides.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1192

Brief Title: STUDY OF METAL-CERAMIC INTERACTIONS AT ELEVATED TEMPERATURES

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

Scope of Work

This research is designed to investigate the fundamental factors of importance in the interaction of metals and ceramic materials at elevated temperatures, with particular reference to factors affecting the fabrication of metal-ceramic composites. These factors include chemical reactions at interfaces, interfacial and surface energy, wettability, effects of heat treatment, and physical properties of composites.

Contractor: California, University of, Berkeley, California

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

Brief Title: MECHANICS OF METAL-CERAMIC BONDS

Investigator: J. A. Pask

Scope of Work

Studies on wettability of platinum by sodium silicate glass have shown poorer wetting at an intermediate pressure which has been related to changes in interfacial tensions with varying vapor pressures. A continuation of more quantitative studies under controlled vapor phases has led to a system containing an experimental volume in which the vapor phase may be varied over wide ranges of pressure, temperature and composition. Experiments performed during the past year have provided additional data indicating the complex nature of the wetting of metals by glass even in relatively simple systems. Attempts to determine all the affecting variables in the platinum–sodium silicate system are continuing.

Experiments on wetting of iron by sodium silicate glass and platinum by sodium lead silicate glass have been made at a pressure of 10^{-5} mm of Hg. Modifications of the experimental system have resulted in lack of duplication of earlier reported results. As a result the complexity of the wetting problem has been further emphasized. Consequently studies on system design, metal purity, surface treatment, prior metal history, and glass stability at low pressures are being made with reference to their role in surface wetting studies.

Contractor: California, University of, Berkeley, California

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

Brief Title: THE GASEOUS SPECIES ABOVE HIGH MELTING SOLIDS

Investigator: A. Searcy

Scope of Work

Little is known about the compositions and stabilities of gaseous species formed by refractories upon vaporization or reaction with their environments. Knowledge of such species may be of extreme importance in prediction of utility of refractories under various conditions of service and can add much to the body of data from which our theories of the nature of chemical bonding are derived. The immediate purpose of this project is to investigate the nature of gaseous species of some metal-metal carbide and metal-metal oxide systems.

The existence of gaseous carbide or oxide molecules will be determined from chemical and X-ray analyses of sublimates and from the change in evaporation rate from a sample with changed oxide or carbon content. Molecular weights of gas species will be determined by use of a mass spectrometer or by use of an apparatus for simultaneous determination of the weight of molecules in a molecular beam and the force exerted by the beam. From the force of the beam, vapor pressures and free energies of formation of the gas molecules will be calculated.

Attempts will be made to quench vapors of unusual compositions to form solids of abnormal oxidation states. Such properties of the solids as chemical reactivity, magnetic susceptibility, and crystal structure could then be investigated.

Contractor: Illinois, University of, Urbana, Illinois

Contract: AT(11-1)-67, Project #9

Brief Title: ANNEALING OF COLD WORKED METALS

Investigator: Paul A. Beck

Scope of Work

The conditions for the formation of substructures during plastic deformation and annealing in slightly bent zinc single crystals and subgrain growth in these as well as in high-purity aluminum single crystals after heavy reduction by rolling will be investigated using special X-ray diffraction techniques and electron transmission microscopy, whenever appropriate. The correlation between subgrain growth and mechanical property changes in rolled aluminum single crystals will be studied. The mechanism of nucleation in coarsening and in strain annealing will be studied by means of the modified Schulz reflection Laue technique. The correlation between reorientation and grain growth on annealing in 70-30 brass will be investigated by means of quantitative texture methods and accurate grain size measurements. It is also planned to investigate the possible effects of variations in the amount of inhibition present during recrystallization and grain growth on the resulting texture in high-purity Al–Mn alloys.

Contractor: New York University, New York, New York

Contract: AT(30-1)-1754

Brief Title: SECONDARY RECRYSTALLIZATION

Investigator: John P. Neilsen

Scope of Work

A study of specimens containing two-dimensional grain structure (i.e., all the grain boundaries are more or less perpendicular to the surface of the sheet specimen) in zinc and aluminum has revealed the occurrence of a dormant stage of grain growth followed by a rapid growth stage during isothermal annealing. There is a possibility that this sequence repeats in some periodic manner. Sheet specimens containing about 15 grains/in.^{2} were annealed close to the melting point and continuously observed at that temperature. For a very considerable elapse of time, almost no changes in grain configuration were observed. This was then followed by a period of very rapid grain growth during which the number of grains was reduced to 3, 4, and occasionally 1/in.^{2} The time interval of annealing prior to this rapid growth varied from specimen to specimen for the same prior history.

It is intended to study this phenomenon in great detail with the aid of time-lapse photography. Apparatus is being constructed which will take one picture a minute for eight hours of a specimen of tin held at 200°C in a vacuum system. The object of this part of the investigation is the following:

(a) To obtain quantitative data on the dynamics of this phenomenon.

(b) To obtain the effect of impurities on the process.

Contractor: Pennsylvania, University of, Philadelphia, Pennsylvania

Contract: AT(30-1)-1893

Brief Title: EFFECT OF STRESS ON RECOVERY

Investigator: N. Brown

Scope of Work

The purpose of the research is to determine whether an elastic stress changes the rate of recovery in pure metals.

High purity single crystals will be plastically deformed in known fashions such as pure shear or in torsion. The temperature of deformation will be sufficiently low so that appreciable recovery does not take place during the initial deformation. The specimen will be allowed to recover at a given elevated temperature for various times and during recovery an elastic stress will be imposed on the specimen. The experiment will be designed to determine what effect the magnitude and the type of elastic stress has on rate of recovery. The type of elastic stress during recovery may be different from the type of stress producing the original plastic deformation. For example, if the specimen is deformed plastically in shear in one direction, the elastic stress during recovery may be a shear in the opposite direction.

The initial experiments will use zinc single crystals deformed by simple shear in the slip directions. The recovery will be first measured by changes in the yield point after various recovery times. The recovery temperature, the time of recovery and the plastic strain prior to recovery must be closely controlled in order to determine the effect of an elastic stress on the rate of recovery.

Contractor: California, University of, Berkeley, California

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

Brief Title: CREEP OF ALLOYS

Investigator: E. R. Parker

Scope of Work

Emphasis during the past year has been concentrated on establishing the nature of substitutional solid solution hardening of alloys. Sufficient evidence has been collected to establish the fact that at least two mechanisms of solution hardening operate. One of these is definitely structure sensitive. Since such hardening is at least in part structure sensitive, no simple general laws of solid solution hardening can be established. The apparently structure insensitive mechanism is currently being investigated as well as the nature of solid solution hardening at high temperatures where creep is important and room temperature relationships do not apply.

It is an experimental fact that large-grained materials creep slower at low stresses and high temperatures than do the same materials with a fine-grain structure. The reason for this grain size effect has heretofore been unknown. However, recent investigations have led to the conclusion that this difference in creep rate is not due to the grain size "per se" but is due to the nature of the substructure within the grains.

This study has led to the prediction that for a given grain size the creep rate will decrease as the degree of preferred orientation of the grains is increased.

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. Fleischer

Scope of Work

During the past year, discontinuous deformation of face-centered cubic metals over the temperature range of 4°K to 15°K was the principal subject of investigation. Such behavior is regarded as the result of localized heating and softening in slip regions when flow stress is highly temperature dependent and specific heat is low. It was established that the discontinuous deformation may occur in the absence of twinning. Studies of flow stress temperature dependence down to liquid helium temperature were carried out. A criterion for the incidence of discontinuous deformation has been formulated.

Work during the coming year will be concerned with the behavior of grain boundaries in tension specimens tested to liquid helium temperature. In tests on polycrystalline aluminum at liquid helium temperature, intercrystalline cracks have been observed to form as a specimen was being plastically deformed. Supression of dislocation climb along boundaries at the low temperatures may be an important contributing cause. To explore this phenomenon further, current experiments with polycrystalline samples of high purity aluminum are concerned with the grain size, orientation and temperature dependence of the cracking. Later experiments are planned with bicrystals and possibly tricrystals.

Contractor: Columbia University, New York, New York

Contract: AT(30-1)-1817

Brief Title: STRUCTURE OF METALLIC LIQUIDS

Investigator: Robert B. Gordon

Scope of Work

Scope I - Ultrasonic Measurements

The objective of this research is the application of physical acoustics to the study of the structure of metallic liquids. That acoustical measurements can yield important information on the structure of liquids has been frequently demonstrated in studies of organic liquids, liquid mixtures, and electrolytes. There is reason to believe that such measurements will be equally useful in the investigation of metallic liquids, liquid alloys in particular.

The experimentation involves measurement of the velocity and attenuation of sound as a function of temperature and frequency in liquid alloys. Apparatus utilizing the pulse technique has been constructed which permits determination of sonic velocities to within one part per thousand and the temperature coefficient of the velocity to one per cent accuracy in a temperature interval of 40°C or less. With the quartz crystal transducer in direct contact with the melt, this apparatus has been operated continuously at temperatures as high as 350°C with no deterioration of signal quality.

Velocity measurements at 5 mc on alloys of the Pb–Sn system are now being completed. These will be followed by attenuation and dispersion measurements on the same alloys. It is believed that these results will suggest the alloy systems on which further measurements can most profitably be made.

Other properties needed in the interpretation of the ultrasonic data are being determined as required: At present apparatus is being set up for precise density measurements on liquid alloys.

Scope II - Galvanomagnetic Properties

This research is concerned with the problem of the electronic structure of liquid alloys. The principal experimental work will be the measurement of the Hall effect in metallic liquids. No reliable Hall data are available for liquids. It is believed that such data will be of value in attempting to understand the electrical properties of certain liquid alloys, e.g., the alloys of Cd and Zn where the addition of solutes does not affect the resistivity. Hall coefficient measurements should also be useful, for example, in explaining the minima observed when the resistivity of liquid Cd and Zn is measured as a function of temperature.

The experimental program consists of, first, design and construction of apparatus for Hall coefficient measurements on both solid and liquid alloys at elevated temperatures. This apparatus will be used to investigate the galvanomagnetic properties of the monovalent metal Na as a function of temperature in both the solid and liquid states. The measurements will be extended to alloys of monavalent metals and then to the divalent metals and their alloys where it is anticipated that the theoretical interpretation of the results will be more difficult.

Contractor: Yale University, Hammond Metallurgical Laboratory, New Haven, Connecticut

Contract: AT(30-1)-1857

Brief Title: PHYSICAL PROPERTIES OF LIQUID METALS AND ALLOYS

Investigator: W. D. Robertson

Scope of Work

In connection with a study of the heat capacity of liquid metals and alloys, it was decided to employ an adiabatic calorimeter and to investigate the temperature dependence of heat capacity of alkali metals with particular reference to the change with temperature in the liquid state near the melting point. The necessary apparatus has been obtained for the construction of the calorimeter.

In addition, the program has been extended to the resistivity of liquid alkali metal solutions. It is anticipated that this work will provide information on the effect of atomic size on resistivity of liquids, and on the configurational state of liquids. The necessary apparatus and equipment has been designed and is currently being assembled.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1888

Brief Title: ACTIVITIES IN LIQUID AND SOLID BINARY METAL SYSTEMS

Investigator: 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 components. 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.

The initial step will be to develop an experimental procedure whereby an electrode potential cell can be operated successfully with the metal systems of interest between 600 and 1100°C. A typical cell would be

$$\mathrm{Me} \left| \mathrm{Me^{++}(chloride\ Melt)} \right| (\mathrm{Me + Bi})_{\mathrm{liquid}}$$

where Me is the less noble element. A study of the sources of error inherent in this method will be made. The lead–silver system and a well known system such as the lead–antimony system will be studied first. Results will be compared with information from the literature and the results of calculations to establish the validity of the experimental technique.

Subsequently, work will be begun on systems such as:

Silver–aluminum
Bismuth–aluminum
Bismuth–magnesium
Bismuth–gold
Bismuth–manganese
Lead–nickel

Contractor: Harvard University, Cambridge, Massachusetts

Contract: AT(30-1)-1956

Brief Title: REACTIONS BETWEEN SOLID AND LIQUID METALS AND ALLOYS

Investigator: B. Chalmers

Scope of Work

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

(1) Solidification of a metal from its own melt and from a series of melts in which it is in liquid solution, at various concentrations, in a liquid metal solvent. This should include a very dilute solution as the extreme case. The interface characteristics and, if possible, dislocation content, will be studied to find out whether there is an abrupt transition or a continuous one between growth from a pure melt and growth from a dilute solution.

(2) The solution of a metal or alloy in a molten alloy will be studied, with a view to determining which of the various processes (solid diffusion, liquid diffusion, or interface reaction) limits the rate of solution, and to determine the sensitivity of the rate to temperature, crystallographic orientation and chemical composition.

(3) A study of the process of dendritic freezing in alloys. In a pure metal, the rate of advance of a dendrite is controlled by the amount of supercooling, which is determined only by the temperature of the melt and the disposal of the latent heat released by the growing dendrite. In the case of an alloy, the liquidus temperature at the growth interface depends on the composition and therefore on the rate of growth of the dendrite and the diffusion of solute away from the growing point. Chemical diffusion and thermal diffusion are therefore both effective in determining the actual amount of supercooling that exists at the growing point, and therefore the rate of growth.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1903

Brief Title: CORROSION OF SOLID ALLOYS IN LIQUID METALS AND SALT MELTS

Investigator: Carl Wagner

Scope of Work

Theoretical considerations indicate that the interface between a solid metallic solution and a liquid corroding medium has the tendency to become rugged if one constituent of the alloy is dissolved preferentially. Under these conditions, severe localized attack of solid alloys may occur. A basic study of the following types of attack is suggested.

(1) Attack of solid alloys by liquid metals under conditions where one constituent of the solid alloy is dissolved preferentially, e.g., dissolution of copper in liquid silver from solid copper–nickel alloys.

(2) Anodic dissolution of the less noble constituent of a solid alloy at well defined electrochemical potentials, e.g., dezincification of brass in a salt melt.

Contractor: Michigan State University, East Lansing, Michigan

Contract: AT(11-1)-400

Brief Title: THERMAL PROPERTIES OF SEPARATED METALLIC ISOTOPES

Investigator: D. J. Montgomery

Scope of Work

The effect of isotopic mass on certain thermal properties of separated metallic isotopes is to be determined. The immediate objective is the measurement of the temperature dependence of the specific volume of the separated isotopes of lithium-6 and lithium-7, and the measurement of their melting points. If appreciable differences between isotopes are found, the measurements will be extended to "alloys" of different proportions of isotopes. Similar measurements should be made on magnesium-24 and magnesium-26, and on calcium-40 and calcium-44, after the techniques have been developed with the more plentiful lithium isotopes. Other thermal properties to be considered are specific heat, latent heat, and thermal conductivity. Analysis of the data will permit testing of theories of solids and theories of melting. If substantial differences between isotopes are found, the experiments should be fairly conclusive in deciding between theories for the equation of state of solids and for melting phenomena, since the atomic wave fields will be almost exactly the same, the mass alone changing.

The purpose of the work then is: (1) to obtain basic physical data on an important class of substances; (2) to utilize isotopic mass as a probe for the investigation of matter in the aggregate; and (3) to examine the possibility of isotopic enrichment by fractional crystallization.

Contractor: Wichita, University of, Wichita, Kansas

Contract: AT(11-1)-203

Brief Title: PERMEABILITY METHOD OF DETERMINING SURFACE AREAS OF FINELY DIVIDED MATERIALS

Investigators: L. L. Lyon and G. R. Crocker

Scope of Work

The primary objective of this program is the delineation of the various factors influencing the transport of a fluid through a porous medium across which a pressure differential has been established. The factors influencing the transport of a gas through a porous medium on which it is not adsorbed are fairly well understood. However, if the gas is adsorbed to any appreciable extent, transport can occur in the sorbed phase. The experimental work of this project is concerned with the delineation of the various factors influencing surface flow of various adsorbable gases through porous media that have a wide range of surface areas or pore diameters.

To date the transport of carbon dioxide through several porous glass tubes has been investigated over a wide temperature range. This type of experimental work will be continued on porous glass using sulfur dioxide as the adsorbable gas. Attempts are also being made to collect similar data on the flow of an adsorbable gas through an unconsolidated porous medium such as a powder pack of high surface area carbon black. The internal structures of the porous media will be carefully studied by use of helium permeability and low temperature nitrogen adsorption-desorption techniques.

Section III

RADIATION EFFECTS ON MATERIALS

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, J. J. Antal, A. Golend, D. Keating, A. C. Damask, L. Porter, P. W. Levy, W. L. Kosiba, G. H. Vineyard, and J. B. Gibson

Scope of Work

One of the major research activities in the solid-state physics program is the study of defects in crystals. While the major effort is concerned with radiation effects in solids, other departures from perfect periodicity, e.g., order-disorder phenomena, are under active investigation. The problems are being attacked by many diverse techniques both experimentally and theoretically.

Scope I - Neutron Transmission Experiments

Long wavelength neutron transmission experiments on irradiated graphite, described in 1955, led to an absolute determination of the concentration of lattice defects in this crystal. The technique is being applied to other types of solids of low neutron absorption: α-Al_2O_3, a high melting ionic oxide, and beryllium, a metal. A single crystal of α-Al_2O_3, which had been irradiated at room temperature, showed a measurable, but very small effect on the transmission. The value predicted by theory is some 6 times greater. In order to minimize annealing effects samples will have to be irradiated and examined at low temperature, and the equipment is now being rebuilt for this purpose.

Scope II - X-ray Studies

Displaced atoms, and particularly the associated strains or structural changes, can be studied by X-ray techniques. Diamond is under extensive investigation because radiation effects in this crystal are pronounced and of considerable theoretical interest. Upon heavy irradiation (4×10^{20} fast nvt) diamond exhibits an X-ray diffraction pattern characteristic of an amorphous material in which many features of the diamond structure are retained. There is also pronounced small angle scattering. The density of the irradiated diamond is lower by 14% as determined by hydrostatic methods. The radial distribution curve obtained by Fourier analysis is strongly characteristic of the diamond structure. The amorphous structure may be due to strains in the diamond caused by the presence of interstitial atoms, or to structural changes more akin to partial transformation to graphite. It is hoped that a definite model will evolve from a detailed analysis of the data combined with annealing investigations.

Another material in which radiation effects are being studied with X-ray is sodium azide, (in cooperation with Picatinny Arsenal). The compound is known to decompose under gamma-ray and reactor irradiation. Sodium azide is a layer structure with the cubic close packed stacking sequence. X-ray studies have shown that both gamma-rays and reactor irradiation definitely produce stacking faults in this packing sequence. This is an unusual manifestation of radiation damage since a stacking fault involves the cooperation of many atoms to shear layers with respect to each other. Mechanical deformation of sodium azide also produces stacking faults, the number of which is found to increase with increased mechanical deformation irradiation. The principal difference between the mechanically deformed and irradiated states is that the former state lacks the color centers. Annealing studies on the two states are in progress.

Scope III - Effect of Irradiation on Solid State Reactions

A series of experiments, partly described in 1955, have shown that diffusion rates may be increased by irradiation to such an extent that the short range order of alloys is increased at a temperature where normal reaction rates are negligible. These conclusions were based primarily on resistivity studies. As a by-product of this study, the resistivity of alpha-brass was found to decrease with increasing short range order. The relation between resistivity and short range order has been further investigated by measurements on very carefully quenched samples of $AuCu_3$ which have shown an increase in electrical resistivity with increasing short range order. This result is supported by theoretical studies. Changes in short range order are being used as a sensitive tool to measure certain radiation effects in alloys, namely, the migration of defects through which a change in short range order results from the accelerated atomic diffusion.

It has been shown that prior reactor irradiation facilitates the white to gray tin transformation by decreasing drastically the induction period. Further work is being done on this problem with the hope of arriving at the basic mechanism. The transformation of irradiated specimens was found to start at the specimen surface and primarily from a single nucleus. The rate of growth of the radius of a gray tin nucleus at a given subcritical temperature is linear, and therefore, rate measurements can be most conveniently made by determining the rate of advance of the interface between the white and gray tin along a wire specimen. Using 99.999% pure tin, seeding experiments and linear rate measurements indicate that irradiation does not effect the linear rate of growth of the gray tin, and the rates obtained are in good agreement with those of other investigators. Irradiation merely decreases the induction time. The radiation effect may not be directly associated with the defects produced, but rather with the over-all strain energy produced in the specimen by the defects.

Scope IV - Optical Properties of Insulators

The effect of radiation on the optical absorption of solids is of inherent interest and, in addition, can become a useful tool for the determination of the number of displaced atoms produced by fast particles. Several different approaches can be used: (1) search for absorption bands produced by fast particles and not by ionizing radiation and demonstrate that one or more of these bands arise from vacancies or interstitials; (2) attempt experiments with materials, such as the alkali halides, in which the absorption bands associated with vacancies are well known, and produce changes in the vacancy concentration by radiation; (3) look for damage in materials like the azides in which the crystal may be decomposed, and correlate the decomposition with the radiation damage. The first of the above approached has been used with Al_2O_3, and fused and crystalline silica. These high melting oxides are very resistant to coloration by ionization radiation but color easily upon neutron irradiation. The growth of the absorption bands in Al_2O_3 was studied as a function of irradiation and it was found that the growth may be resolved into a linear part and an initial part which saturates exponentially. The problem is being investigated theoretically as well as by additional experiments at low temperatures.

Coloring of the alkali halides by gamma-rays has been beset in the past by difficulties of obtaining reproducible results. Recently, it has been shown that when these materials are colored at room temperature (with the crystals always kept in total darkness) by gamma-rays, the F centers which are produced decay with a fast and slow component. The fraction of fast decay is large when the total coloring is small and the fast component becomes small as the total F center density approaches saturation. Taking proper cognizance of this observation a very considerable improvement in reproducibility is obtained for F center coloring as a function of gamma-ray dose. The coloring of the various materials discussed above can be described in terms of the absorption bands produced and in terms of the growth of these bands as a function of dose or time.

Scope V - Chemical Effects of Irradiation

Irradiation can also effect the chemical reactivity of solids either via ionization effects or by means of displaced atoms. Several examples of ionization induced reactions have been under study in cooperation with the Nuclear Engineering Department of Brookhaven and the Polytechnic Institute of Brooklyn with emphasis on gamma-ray induced solid state polymerizations. It has been clearly established by now that these reactions proceed via a free radical mechanism in the solid state without any local fusion. Some structural X-ray work is essential for further understanding of the mechanism. A clear cut example of changes in chemical reactivity due to displaced atoms is the reaction between graphite and oxygen whose rate is influenced in the following ways by displaced atoms and ionizing radiation. 1) Prior reactor irradiation increases greatly the oxidation rate of graphite in the 250 to 400°C range. The increase in the reaction rate is a catalytic effect in the sense that there are about 1% displaced atoms present at the reaction temperatures. The displaced atoms are not themselves being oxidized preferentially but facilitate the over-all oxidation. 2) Ionizing radiation (gamma rays) present during oxidation also increases the rate of oxidation of unirradiated graphite but by a much smaller factor. Results lead to the conclusion that the gamma ray effect is probably due to the ionization of oxygen molecules since gamma rays have not been observed to have any effect on the properties of graphite at these exposures. 3) The activation energy for the oxidation of unirradiated graphite is independent of the oxidant with a value of approximately 50 kcal/mol for both oxygen and air. Neutron irradiation, however, resulted in a considerably lower activation energy for the reaction i.e. 36.1 kcal/mol. This may be interpreted as an actual lowering of the activation energy or as a superposition of two reactions, the normal thermal reaction plus the defect induced oxidation. This point cannot be decided until the reaction is followed over a much wider temperature range.

Scope VI - Order-Disorder Problems

A simple "chemical" theory of the kinetics of order-disorder transformations, published recently, has been expanded into a more elaborate theory based on basic statistical mechanical principles and the simple theory was shown to be a special case of the more general one. An infinite array of distribution functions, introduced to express the occupation of all possible sets of lattice sites, determine all the kinds of order in the lattice, including the usual long and short range order. The mathematical treatment has been explored in detail for two mechanisms of atom movement, direct interchange and vacancy interchange, and for two common lattice types, AB b.c.c. and AB_3 f.c.c. Present evidence suggests predominance of the vacancy mechanism, at least in close packed systems. The simple vacancy model has been applied to experiments of Burns and Quimby on electrical resistivity in Cu_3Au with reasonably good agreement between theory and the observed relaxation times over a range of temperatures below the critical temperature.

Theoretical work has also been done on the effect of short-range order on the electrical resistivity of binary alloys with calculations made for Cu_3Au and alpha-brass. Both the type of short range and the angular dependence of an electron scattered by an impurity enter in the calculation. The final results were found to depend sensitively on these factors. Good agreement with experiment was obtained for the case of Cu_3Au, poorer for alpha-brass.

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

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

Title: CRYSTAL DEFECTS AND MECHANISM OF DAMAGE

Investigator: C. Eugene Dixon

Scope of Work

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

Recent work on this project has included measurements of the threshold displacement energy in nickel near 4°K, the rate of resistivity increase in copper and nickel at 4°K and 80°K due to 1.25 Mev electron irradiation, and subsequent annealing studies.

Future work will include measurements of displacement cross section as a function of electron energy and studies of the influence of impurities, dislocations and electron energy on resistivity recovery. Very low temperature irradiations (near 4°K) will be made in most of this work; however, recent interesting effects of 150°C irradiation have been discovered and these will be explored further.

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

This project on irradiation damage aims to determine the kinds and amounts of various lattice defects produced in crystalline solids by nuclear irradiation. It also aims at identifying the types of thermal recovery processes which occur at various temperatures and at determining the results of the annealing.

To date most of the work has been on deuteron irradiated copper, silver, and gold. An annealing process at about 30°K has been discovered in copper and silver. An appreciable fraction of the resistivity and the length change anneals out in this process. The damage produced by deuteron irradiation at 10°K is about five times less than predicted by theory. The annealing from 78°K to about 220°K involves a range of activation energies. A well defined second order annealing process occurs at about 250°K. In copper, this process is associated with an energy of motion of 0.68 e.v. Quenching experiments on gold give the energy to form a vacancy to be 1.05 ± 0.2 e.v. The energy to move a vacancy is 0.68 ± 0.06 e.v. A theoretical description of the processes leading to damage, of the temperature spikes, and of the annealing processes has been given. The theory has been compared with present experiments, and additional experiments to clarify matters have been suggested.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1193

Brief Title: RADIATION EFFECTS IN SOLIDS

Investigator: R. Smoluchowski

Scope of Work

This program is devoted to the study of radiation effects on the fundamental properties of solids. Work on alkali halides has been on the electrical and optical properties, stored energy, and density. Work on metals has been on electrical conductivity. The radiations used have been protons in the energy range 90-410 Mev, and gamma rays from a one curie cobalt 60 source.

During the last year measurements were completed on the heat released upon warming up of sodium chloride crystals exposed to 350 Mev protons. Considerable heat is released, part of which can be accounted for by the disappearance of color centers. But a good bit of the heat is not connected with any changes in optical absorption.

Work was completed on the room temperature resistance changes in tungsten irradiated with different energy protons. From the magnitude of the effect and its dependence on energy, it appears that in this energy region the radiation effect is due mainly to the high energy fragments produced in nuclear reactions involving the incident protons. Some theoretical calculations also indicate this.

The future program includes measurements of the density changes in sodium chloride exposed to high energy protons, and the annealing of these changes at various temperatures. A careful study of the annealing of conductivity changes in alkali halides will be made. It is also planned to make some irradiations at liquid nitrogen temperature for alkali halides and metals.

Contractor: Purdue University, Lafayette, Indiana

Contract: AT(11-1)-125

Brief Title: BASIC RADIATION DAMAGE STUDIES

Investigator: K. Lark-Horovitz

Scope of Work

To study lattice defects, electrons at 2.5 Mev and 5 Mev from the Linear Accelerator have been used to irradiate various semiconductors—germanium, silicon, indium antimonide and indium arsenide. The effects of electrons of various energies have been studied to determine the scattering cross-section, and compare it with theory, taking into account effects of geometry, multiple scattering and scattering of particles out of the foil.

In silicon, never investigated with electrons before, a number of n-type and p-type samples of various conductivities have been investigated: the bombardment introduces levels close to the valence band and close to the conduction band besides deep lying levels.

Before irradiation photoconductivity in silicon can only be observed up to 1.1 μ. After electron-irradiation photoconductivity is found out to 1.4 μ.

In InSb and InAs effects of purely elastic scattering without transmutations can be observed. It was found that n-type indium antimonide irradiated at low temperature is converted to p-type, but upon heating to room temperature reconverts to n-type. P-type InAs converts to n-type.

By measuring Hall Effect, conductivity, thermoelectric power and photoconductivity on electron irradiated silicon, germanium, and other semiconductors the production of defect levels as produced by irradiation will be studied and their annealing observed.

Studies of the threshold energy for displacement in semiconductors will be made.

Contractor: Northwestern University, Evanston, Illinois

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

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

Investigator: John W. Kauffman

Scope of Work

Radiation damage in metals is to be studied using electrons in the range 0.5 to 5 Mev. The Northwestern University Van de Graaff generator is being completed and will be used to produce the desired electron beam. The purpose of this work is by proper choice of experimental procedures and methods to separate effects and determine concentrations of the various types of lattice imperfections resulting from the irradiation as well as to study interactions between them and to account for the kinetics of the recovery process of the irradiation induced damage.

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.

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.

Contractor: Brown University, Providence, Rhode Island

Contract: AT(30-1)-1880

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

Investigator: Philip James Bray

Scope of Work

Radiation damage in solids is being investigated by techniques of nuclear magnetic resonance. The shape, intensity, and spin-lattice relaxation time of nuclear resonances in ionic crystals will be studied as a function of the crystal purity and strain condition, type and intensity of incident radiation, color-center density and distribution, and temperature. Similar studies will be made on glass after a search for nuclear resonances from special glass constituents such as boron and aluminum.

A broad examination of metals will begin with investigation of quadrupole interaction splitting of the nuclear magnetic dipole resonance in copper powder, as induced by irradiation. The results will be compared with effects induced by cold working. Nuclear resonance investigations will be extended to other pure metals, to radiation disordering of ordered alloys such as copper–gold, and to precipitation–hardening of alloys such as copper–beryllium. Studies will also be made in metals of those resonances properties, such as Knight shift and exchange broadening, that depend primarily on the conduction electrons.

The line width and structure of proton nuclear resonances in irradiated and unirradiated polymers will be studied for information on the effects of cross-linking and bond rupture caused by irradiation. Investigations will be made of various nuclear resonances in other irradiated organic materials.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-858

Brief Title: X-RAY STUDY OF RADIATION DAMAGE

Investigator: B. E. Warren

Scope of Work

The aim of the program is to develop x-ray diffraction methods for measuring and evaluating the nature of damage in materials, and to apply the methods to typical samples. Because the problem is basically similar, and samples are easier to prepare, considerable emphasis is placed on parallel studies of cold work distortion in metals, and the effects of interstitials and solid solution distortions.

Huang type distortions can be produced by either interstitials or solid solutions involving atoms of different size. During the present year the Huang type diffuse scattering has been measured for the first time. Deformation faulting in a FCC metal has been measured by the peak shifts which are produced. Twin faulting produces a peak asymmetry, and the effect has now been observed in cold worked α-brass. Methods for measuring separately the probability of deformation and twin faulting in FCC metals are being developed. A thorough analysis has been completed for the effect of deformation faulting, twin faulting, and production of small incoherent domains in the cold working of a BCC metal. The theory has been applied to a pattern of cold worked β-brass.

This type of thorough study of the x-ray diffraction effect of each of the possible types of damage or imperfection will be continued.

Contractor: Knolls Atomic Power Laboratory, Schenectady, New York

Contract: W-31-109-Eng-52

Brief Title: X-RAY STUDIES OF RADIATION DAMAGE

Investigator: C. W. Tucker, Jr., and P. Senio

Scope of Work

This program is an experimental and theoretical study, using all X-ray diffraction techniques, of radiation damage in ionic, covalent, and metallic crystals. Its purpose is to discover (a) the nature of the defects produced by heavy particle irradiation, (b) the lattice distortions produced by the defects, and (c) the crystal chemical basis for variations in damage among various ionic, covalent, and metallic crystals.

A set of X-ray scattering effects due to point defects in irradiated crystals has been discovered and interpreted qualitatively by theory. Further experimental and theoretical work is in progress to interpret quantitatively some of the effects, namely, diffuse scattering around the reciprocal lattice points and the artificial temperature factor. A theoretical connection has been derived between the interstitial content of damaged metals and lattice parameter changes using an elastic model for point defects. Exploratory irradiations of a wide variety of ionic, covalent, and metallic crystals have shown a great variation among these crystals in the X-ray effects shown on irradiation. Low cleavage strength coupled with brittleness seems to be involved in producing broadening of the Bragg reflections, while very high melting point, tightly bonded crystals generally show the X-ray effects arising from point defects. Work is continuing on exploratory irradiations to obtain a systematic explanation for the production of the X-ray effects in various crystal structure types as well as analysis of the effects themselves.

Contractor: Rutgers University, New Brunswick, New Jersey

Contract: AT(30-1)-1730

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

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

Scope of Work

The crystal imperfections resulting from pile-irradiation is being investigated by means of the special x-ray diffraction methods developed in this laboratory. Significant differences have been found in the scattering between irradiated and unirradiated copper and nickel wires. Studies of partially annealed copper and nickel wires will be carried out before and after irradiation to test the validity of the thermal spike theory of radiation damage.

X-ray rocking curve studies were made of unirradiated and irradiated germanium single crystals (resistivity 35 ohm-cm, impurity content 1 in 109). The slow neutron flux of the irradiated germanium crystal was 1.43×10^{18} nvt and the temperature of irradiation was 160-200°C. A characteristic broadening of the (1 1 1) reflection of the irradiated germanium crystal was observed. Careful measurements of the (1 1 1) rocking curves disclosed a marked anisotropic effect with regard to the rocking direction. It is proposed to investigate this observed anisotropic effect in greater detail.

The x-ray studies will be supplemented by a thorough analysis of the basic factors that contribute to diffuse scattering. It is proposed to adapt the methods developed by H. Bohr [1], N. Wiener[2], A. Kolmogorov[3], and others in the study of almost periodic and random functions to the problems of lattice imperfections and thus to make a new mathematical approach through the field of generalized harmonic analysis.

An application of these methods has been made by Slade and Nanni to problems of detection of electrical signals [4].

(1) "Almost Periodic Functions" by Harold Bohr, Chelsea Publishing Co., New York (1947).

(2) N. Wiener, "Generalized Harmonic Analysis," Acta Math. 55, 117-258 (1930).

(3) A. Kolmogorov, "Interpolation and Extrapolation von Stationären Zufälligen Folgen," Bull. Head. Sc. U.R.S.S., Sc. Math. 5, 3-14 (1941).

(4) "Effect of Noise on the Temporal Moments of a Pulse," Technical Report No. 2, Signal Corps Project No. 17-132B-0.

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, with special emphasis on the copper–beryllium and nickel–beryllium systems, and copper-base solid solution alloys. It has been found in the first case that fast neutron bombardment has enhanced 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%) alloy 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.

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

Contract: W-7405-eng-26

Brief Title: LOW TEMPERATURE RADIATION EFFECTS IN METALS

Investigators: T. H. Blewett, R. R. Coltman, T. S. Noggle, J. K. Redman, C. E. Klabunde, D. O. Thompson

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 will be extended to include tensile strength studied on a variety of metals. Experiments on the growth of uranium metal during low temperature bombardment are also planned.

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.

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 R. 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, 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°K and 20°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. Recently significant differences in etch behavior in fast neutron bombarded specimens of Ge and Si have been discovered, and studies of stored energy are being planned.

Contractor: Knolls Atomic Power Laboratory, Schenectady, New York

Contract: W-31-109-Eng-52

Brief Title: RADIATION DAMAGE TO STRUCTURAL METALS

Investigator: C. A. Bruch

Scope of Work

Specimens of polycrystalline metals representing the three major crystallographic systems are being studied to determine changes in some mechanical and physical properties due to neutron irradiation. The metals include copper, nickel, titanium, zirconium, iron, molybdenum, and Type 347 stainless steel. The neutron exposures at temperatures of 60 to 100°C range from approximately 8×10^{18} to 2×10^{22} thermal nvt, the fast neutron exposures (greater than 1 Mev) being approximately one-tenth the thermal nvt's. A limited number of metals were irradiated at 270 to 300°C. Some of the more significant observed effects of neutron irradiation are as follows:

1. The ductile to brittle transition temperature in molybdenum was increased from −30 to +70°C. (J. Metals, Feb., 1955, p. 281.)

2a. The per cent change in a particular property was greatest in metals which had the lowest value of the property prior to irradiation.

b. There are indications that the crystal system is of importance. (J. Metals, Oct., 1956, p. 1362.)

3. Radiation effects in titanium and zirconium did not reach saturation after an exposure of 2×10^{22} thermal nvt. Saturation occurred in the other metals studied after an exposure of approximately 5×10^{20} thermal nvt.

4. Neutron irradiation at 60 to 100°C did not produce any changes in microstructure. In addition when these irradiated metals were given annealing treatments sufficient to restore the metal to the pre-irradiation hardness level, there were no changes in microstructure.

5. It is indicated that the effect of neutron flux is small.

6. Exposure temperatures above 60 to 100°C, decreased the radiation damage in all of the metals except molybdenum. In this metal the hardening was greater at 300°C than at lower temperature, whereas the changes in electrical resistivity were lower at the high temperature.

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

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

Brief Title: MECHANISM OF DIMENSIONAL INSTABILITY

Investigator: L. L. Seigle

Scope of Work

The growth of fissionable materials under pile irradiation is being studied both theoretically and experimentally. An explanation of shape changes in alpha-uranium under irradiation has been proposed, based upon the anisotropic diffusion of interstitial atoms and lattice vacancies generated by fission fragments. In order to test the theory, an effort is being made to determine the anisotropy of self-diffusion of alpha-uranium. Irradiation tests of uranium are under way to ascertain the influence of structure upon dimensional changes of fuel materials in the reactor.

Studies are continuing of the generation and mechanism of annihilation of lattice defects in metals, and the dimensional and structural changes associated with these processes.

Contractor: Knolls Atomic Power Laboratory, Schenectady, New York

Contract: W-31-109-Eng-52

Brief Title: STATISTICAL MECHANICS OF GASES

Investigator: Leo F. Epstein

Scope of Work

The irradiation of nuclear reactor fuel elements produces substantial amounts of the gases xenon and krypton. To evaluate the importance of this internal gas production on the damage phenomena observed in irradiated solids requires a knowledge of the temperatures, pressures, etc., existing in the gases. To determine these factors, reliable equation of state and transport property (viscosity, diffusion coefficient, thermal conductivity, etc.) data are needed in temperature and pressure regions which are not readily accessible to experimental observations. The use of statistical mechanical methods as a tool for extrapolation and prediction is a possible technique for examining these properties.

Using the digital computer equipment, the problem of the effect of intermolecular potentials upon the second and third virial coefficients and upon the transport properties is being examined. Work in progress includes (1) the computation of the third virial coefficient of a Lennard-Jones 6-S gas as a function of temperature and the repulsive parameter S; and (2) the preparation of tables of the transport integrals for the 6-9 gas.

Contractor: Argonne National Laboratory, Lemont, Illinois

Contract: W-31-109-eng-38

Brief Title: IRRADIATION EFFECTS ON METALS

Investigators: S. H. Paine, J. H. Kittel, and F. R. Taraba

Scope of Work

Scope I - Power Distribution in Uranium Specimens Under Irradiation

The distribution of neutrons in cylindrical fuel specimens under irradiation is quite sensitive to the efficiency of the metal as an absorber. In general, the neutron flux within the specimen is smaller than at the surface because of absorption of the neutrons in fission and capture events. This effect is known as "self-shielding." It is negligible for small thicknesses of normal uranium, but becomes very appreciable at higher U^{235} contents.

The rate of power production in uranium is proportional to the neutron flux, and its distribution in any particular specimen, therefore, is affected by self-shielding. In accordance with this fact, the internal temperature, the buildup of radiation damage, the total atom burnup and the distribution of fission products within the metal are also affected by the neutron distribution. Strictly speaking, then, an irradiated fuel specimen may be considered as a concentric series of different fission product alloys, all irradiated at different temperatures to different burnup levels and having different degrees of structural damage which result in different physical properties. Any effort to understand the fundamentals of radiation damage in fuel requires an understanding of these interrelated variables. Experimental work on neutron distributions in specimens of various sizes and degrees of absorptivity is being conducted at Argonne with this objective in mind. Present goal is limited to a complete treatment of cylindrical geometry, including the Wilkins end effects.

Scope II - Irradiation Induced Changes in Single Crystals

Because uranium is radically anisotropic in its reaction to irradiation and suffers gross dimensional changes, the immediate interest in this investigation lies in the kinetics of atom transport in the so-called fission product and fast neutron "thermal spikes." A correlation is being sought between dimensional change rate and the major radiation variables of temperature, neutron flux, and total dose. Single crystals of uranium are being used in this investigation to simplify the interpretation of experimental results and obtain fundamental information concerning the mechanism of damage.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1828

Brief Title: IRRADIATION EFFECTS ON SURFACE REACTIONS OF METALS

Investigator: R. Smoluchowski

Scope of Work

Radiation damage can affect metal surface reaction in two ways. It may alter the properties of the metal being irradiated in a pile or a cyclotron, or the radiation emitted by the radioactive atoms may alter the system under study. The objective is to carry out a quantitative study of the influence of irradiation upon the surface reaction of metals. This will include the effect of the irradiation on the metals, the surface reaction products and on the ambient solution or gas. The experimental methods used will include: (1) determination of the electrode potentials of the metals; (2) measurements of the solution rates of the metals and solid metal compounds in various solutions; and (3) kinetic studies of metal-gas reactions.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

Contract: AT(30-1)-1565

Brief Title: RADIATION DAMAGE IN METALS–SURFACE EFFECTS

Investigator: W. Lees

Scope of Work

To predict the effects of fast-neutron irradiation on bulk phenomena in metals, one must account theoretically both for atomic displacements arising in the dissipation of neutron energy and for the influence of such displacements on the observed phenomena. Observations at a metallic surface can test directly the process conceived for energy dissipation, since the flux of atoms displaced from the metal—with a measurable evaporation-threshold energy—can be measured and compared with the neutron flux. Simple energetic considerations set a very low upper bound to the amount of evaporation to be expected with a practicable irradiation. A few atomic species, among which iridium is outstanding, permit very high sensitivity of detection because of the high specific activity induced by concomitant thermal-neutron capture. Evaporated iridium is to be collected during irradiation, separated from the source on return, and measured by comparing its total activity with that of an aliquot from the known mass of the source. To enhance counting sensitivity, the evaporated material will be isolated and collected chemically, if possible. The specific activity induced fixes the local magnitudes of neutron flux in a specimen, if the energy distribution is known. The absolute distribution of neutron energies, with the amount of metal evaporated, provides the desired relation.

Contractor: Nuclear Metals, Inc., Cambridge, Massachusetts

Contract: AT(30-1)-1565

Brief Title: RADIATION EFFECTS ON PRECIPITATION-HARDENING ALLOYS

Investigator: A. Boltax

Scope of Work

Samples of copper–iron and nickel–beryllium alloys are being prepared to study the resolution of precipitate particles by fast neutron irradiation. The alloys are to be made in a specially constructed vacuum furnace. Measurements before and after irradiation will be made by means of magnetic, electrical resistivity, and electron microscopy techniques. The magnetic measurements will include hysterisis loss and saturation induction on copper–iron samples and Curie temperature on nickel–beryllium samples. From these measurements, semi-quantitative values of the number of atoms displaced or transferred across the matrix-precipitate interfaces as a function of the average precipitate particle size can be calculated. Results of this type should be useful in advancing the current theories of displacement spikes.

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

Contract: NBS Project No. 0509-11-3271

Brief Title: THERMOCHEMICAL STUDY OF IRRADIATION EFFECTS

Investigator: E. J. Prosen

Scope of Work

The objectives of this project are: to develop methods and make calorimetric measurements of the increase in energy content of quartz, silicon carbide, and other insulators resulting from exposure to radiation in nuclear reactors; and to determine the total energy increments and the effect of annealing at different temperatures. It is expected that the data obtained will aid in the understanding and the interpretation of charges which occur in the exposed substances and will also contribute to the understanding of the chemical bonding in crystalline materials. A current phase of the work is the determination of the heat of combustion of silicon carbide. Fairly complete combustion has been obtained using titanium ribbon as the ignitor and auxiliary substance. Measurements have not yet been made on irradiated silicon carbide. A gold calorimeter is being constructed for the determination of the heats of solution of unirradiated and irradiated quartz and vitreous silica in aqueous hydrofluoric acid at 80°C.

Contractor: Brown University, Providence, Rhode Island

Contract: AT(30-1)-1772

Brief Title: STUDY OF RADIATION EFFECTS BY ULTRASONICS

Investigator: Rohn Truell

Scope of Work

The purpose of this work is to determine whether ultrasonic attenuation and velocity or moduli measurements can contribute any new and significant information in the field of radiation effects in solids. The extent to which dislocations and dislocation changes may enter into radiation effects has received special attention.

It has been definitely established that directional bombardment of single crystal silicon with fast neutrons is clearly observable by high frequency ultrasonic methods. It has been found from ultrasonic double refraction measurements and from accurate velocity measurements that the anisotropy (induced by the directional fast neutron bombardment) can be expressed in terms of a difference in moduli for transverse waves. The effects observed have produced appreciable and easily measurable changes at relatively low irradiation levels and it is expected that changes can be detected at still lower levels. The effects observed here appear to be connected primarily with displaced atoms but not with dislocation behavior.

A second area of investigation is that of neutron and gamma irradiation of crystalline quartz. The effects observed ultrasonically of gamma irradiation of crystalline quartz are very small compared with the neutron effects which cause large attenuation and velocity changes.

A third area being investigated is that of the gamma irradiation effects in alkali halide single crystals. Large attenuation changes have been observed as a function of irradiation; these changes appear to be definitely connected with dislocation damping as contrasted with the effects observed in silicon and crystalline quartz.

Other semi conductor and insulating materials are being used in addition to those mentioned.

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

Contract: AT(30-1)-1312

Brief Title: IRRADIATION DAMAGE TO GLASS

Investigators: N. J. Kreidl, J. R. Hensler, and G. E. Blair

Scope of Work

Five non-browning optical glasses were developed with excellent stability to the target dose of 10^6 roentgens and which were still usable at 5×10^8 roentgens. Investigation of the mechanism of coloration of glass by radiation as well as its prevention in the visible region by incorporation of cerium into the glass led to the discovery of the identical action of the other transition elements of shifting and intensifying the absorption bands produced.

A study of the absorption band structure in the ultraviolet as well as the visible region was made possible by use of a phosphate base glass which had good transmission out to 200 mμ.

Results of electron spin resonance measurements of irradiated phosphate glasses associated and the electrons giving rise to coloration with the phosphorus nucleus and showed some influence of aluminum, also present in the composition.

The chemically simpler pure GeO_2 glass was found to be relatively insensitive to coloration than binary, ternary, and quaternary GeO_2 base glasses with Na_2O, CaO, and Al_2O_3 which were qualitatively similar in their coloration properties to the more common silicate analogues; the germanate glasses were in general less sensitive, however.

The discovery of the sensitivity and stability of the absorption bands produced in glasses containing transition elements led to the development of a cobalt-containing borosilicate glass which could be used as a dosimeter up to 10^7 roentgens. Work is underway to extend the useful range of this glass in dosimeter work.

Contractor: Canisius College, Buffalo, New York

Contract: AT(30-1)-1810

Brief Title: INVESTIGATIONS IN IRRADIATED VITREOUS SILICA

Investigator: Herman A. Szymanski

Scope of Work

The first phase of this investigation was to anneal five different samples of radiation damaged vitreous silica using isothermal annealing and also the method described by Overhauser in the Physical Review, page 393 (1953), and to subject these results to the theoretical treatment suggested by Primak and this author in the Physical Review of Feb. 15, 1956, page 1268. These anneals have been completed and are being prepared for publication. X-ray spectra have also been determined and will be included in this analysis.

Future work is to consist of analyzing these results and completing the five anneals planned. It is planned to expand the annealing facilities and then to diversify this study further to include the new types of materials and experimental methods.

Contractor: (The) Pennsylvania State University, University Park, Pennsylvania

Contract: AT(30-1)-1858

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

Investigator: J. 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°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 coming year, it is planned to explore the effects of neutron and gamma irradiation in greater detail. In addition to studying the changes in dynamic mechanical properties, and the changes in nuclear magnetic resonance line shapes, it is hoped also to investigate the thermal and dielectric properties of polymer samples in both the irradiated and non-irradiated conditions. Apparatus for making thermal and dielectric measurements over a broad temperature range is now being constructed. Effort will also be devoted to obtaining or preparing polymers having unique structural features so as to allow more definite assignment, in terms of molecular structure, of the observed dispersions.

SUBJECT INDEX

PRODUCTION, TREATMENT, AND PROPERTIES OF MATERIALS

ALLOY THEORY AND THE NATURE OF SOLIDS

RADIATION EFFECTS ON MATERIALS

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