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SUMMARIES OF PHYSICAL RESEARCH PROJECTS IN METALLURGY

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FOREWORD

In addition to conducting research in-science 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.

The summaries for Metallurgy included in this directory have been prepared principally by the senior 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 TIS may be abstracted in the journal of *Nuclear Science Abstracts*. The journal is published bi-monthly by the Technical Information Service and is available on subscription at six dollars a year from the Superintendent of Documents, Government Printing Office, Washington 25, D. C.

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Section I

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PRODUCTION, TREATMENT, AND PROPERTIES OF MATERIALS ----



Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1024

Brief Title: ELECTROCHEMICAL STUDIES OF NON-AQUEOUS MELTS

Investigator: G. Derge, G. M. Pound, M. T. Simnad, and Ling Yang

Scope of Work

The immediate 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 being accomplished primarily by direct measurements of such properties as electrical conductivity, and ionic transport phenomena. Special circuits and experimental techniques have been developed for work at temperatures in the range from $1000^{\circ}-1500^{\circ}C$.

A good understanding of the iron-silicate system has resulted. It changes from an electronic behavior for melts with more than 90% iron oxide to ionic behavior for silica saturated compositions. For the latter, mobility and transport numbers have been measured. The work will be extended to more complex slags.

The molten sulfides of different metals have shown a wide range of electrochemical features, including ionic, and electronic characteristics comparable to both metallic and semiconductor behavior. This leads to new concepts of liquid structure.

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Contractor: Columbia University, New York, N. Y.

Contract: At(30-1)1135

Brief Title: PHYSICAL-CHEMICAL PROPERTIES OF SALT MELTS CONTAINING ZIRCONIUM HALIDES

Investigator: H. H. Kellogg and L. J. Howell

Scope of Work

In view of the possibility that fused-salt electrolysis will prove to be a successful commercial method for the recovery of pure zirconium metal, it is believed that the study of the physical chemical properties of such melts will provide the necessary basic data for process development. The systems deemed to have the best promise for commercial application are the low-melting all-chloride systems containing $ZrCl_4$, lower-valence zirconium chlorides, and one or more of the following: NaCl, KCl, TiCl, MgCl₂, and CaCl₂.

Studies of the phase diagram, vapor pressure, activity relationships, and electrical conductivity are in progress on the following systems:

1. $ZrCl_4$ + NaCl

2. $ZrCl_4 + KCl$

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3. Initially $ZrCl_4$ + NaCl, but equilibrated with zirconium metal, in order to develop the equilibrium concentrations of lower-valence zirconium chlorides.

Studies of the ternary system, ZrCl₄-NaCl-KCl are also being made.

Contract: NBS Project No. 3262

Brief Title: ELECTRODEPOSITION OF BERYLLIUM FROM NON-AQUEOUS MEDIA

Investigator: Abner Brenner

Scope of Work

Since beryllium cannot be deposited from aqueous solutions, because hydrogen is preferentially evolved, the possibility of its deposition from non-aqueous media is being investigated. Although metallic beryllium flake has been obtained commercially from molten salt baths, the deposits have not been thick and coherent. It is felt that such deposits of pure beryllium or beryllium alloys, as well as ductile beryllium, might be obtained by electrodeposition from organic solvents. Moreover, emphasis is being placed on studies using plating solutions of organic solvents, since these baths have the advantage of being operable at room temperature, or slightly above. Investigations have also been initiated to study electrodeposition of coherent deposits of beryllium from fused salt baths.

In the electrodeposition of Be, several types of solutes are being used, including halides, dialkyls, diaryls, borohydrides, and hydrides of beryllium. These compounds have to be prepared, since they are not commercially available.

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Contract: NBS Project No. 3275

Brief Title: HEAT EFFECTS OF DYNAMIC ELECTROCHEMICAL REACTIONS

Investigator: Abner Brenner

Scope of Work

A new method of investigating the polarization phenomenon involved in electrochemical reactions is proposed, to be applied mainly to the electrodeposition of metals. Methods now used involve the measurement of electrode voltage under different conditions of electrolysis. It is proposed to study polarization by measuring the heat evolved in electrolysis. This method is direct, since polarization represents wasted energy which appears as heat.

The work will involve carrying on electrolytic processes in a calorimeter so that the electrical energy consumed may be compared with the heat evolved. An energy balance will be computed for each experiment, and the anode and cathode potentials will also be measured to note any differences in the irreversibility of the reaction, as indicated by the readings of potential and also by the heat effect.

Contract: NBS Project No. 4100

Brief Title: BINARY URANIUM-ALLOY SYSTEMS

Investigator: R. W. Buzzard

Scope of Work

This program has as its primary objective the establishment of the constitutional diagrams of binary uranium alloys. Much attention has been devoted to the Au-U and Ag-U systems, and investigations of the U-Pt and U-Ru systems are under way. Alloys of U with Pd, Ir, Rh, and Os, and also with rare earth elements, such as La, are to be studied.

Attention will also be given (1) to the development of a potentiometric method for the study of transformation kinetics and (2) to an analysis of the alloying characteristics of uranium.

Analysis of the alloying characteristics of uranium based on the Hume-Rothery theory is unsatisfactory. Analysis of the published uranium binary systems has shown that a rather simple correlation of the uranium alloying characteristics may be made, based on an effectivesize/valency factor. Contractor: Columbia University, New York, N. Y.

Contract: AT(30-1) 1593

Brief Title: A STUDY OF INCLUSIONS IN URANIUM

Investigator: George L. Kehl and Hyman Steinmetz

Scope of Work

This research is part of a long-range, systematic, cooperative effort among seven AEC installations to study and to identify metallographically inclusions in uranium metal. The program at Columbia University pertains to identification of magnesium and calcium inclusions. Specimens for this purpose, including those distributed to the cooperating laboratories, were prepared by the Argonne National Laboratory. High purity uranium ingots were prepared that contained a selected second element in an amount just sufficient to form a second phase.

After metallographic preparation, the included phase is removed for appropriate analysis (X-ray and electron diffraction) by means of a supersonic "jack hammer." The apparatus was developed as a part of the contract scope and consists of a 47 kc oscillator that drives a transducer crystal mounted on a micropositioner. The transducer, in turn, drives an electrolytically pointed stylus. While observing the operation microscopically, individual inclusions are broken up by appropriately positioning the stylus with respect to the inclusion. A specimen for analytical purposes is secured by picking up the inclusion debris on a greased quartz fibre mounted on a second micropositioner.

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: METALLOGRAPHY OF URANIUM AND THORIUM

Investigator: H. A. Saller

Scope of Work

This research is part of a long-range, systematic, cooperative effort among seven AEC projects to study and to identify metallographically inclusions in uranium metal. The program at BMI pertains to the identification of inclusions caused by the presence of nitrogen, and other non-metallic inclusions.

This work has required the development of adequate metallographic techniques. The hot stage microscope has been used to study grain boundary behavior in uranium during etching and other stages of sample manipulation. Another phase of this work involves the development of definitive etchants.

In the future, the study will be extended to include thorium.

Contractor: Massachusetts Institute of Technology, Cambridge, Mass.

Contract: AT(30-1)-981

Brief Title: PROPERTIES OF METALS AND ALLOYS (SPECIFIC PROJECTS ARE IDENTIFIED BELOW)

Investigator: A. R. Kaufmann

Scope of Work

Alloying Behavior of Beryllium. The alloying behavior of beryllium will be investigated with specific reference to the solubility of different elements in the beryllium. Of particular interest will be the solubility of the elements of the first transition series and of the noble metals in beryllium. It is planned to use x-ray, resistivity, and magnetic measurements in establishing the solubility as a function of temperature and also in establishing the role that the electrons play in the determination of the solubility limits.

Fractional Crystallization in a Liquid Metal System. Metals A and B form stable intermetallic compounds in bismuth (m.p. 271°C). The individual solubilities of A and B in liquid bismuth are of the same order of magnitude -1 wt. % at 600°C, falling to 0.05 wt. % at 300°C. Attempts are being made to crystallize pure compound $A_x Bi_y$ from melts composed of about 1% A, 10 ppm B, and the balance Bi by slowly cooling a liquid of such composition from 600°C to 300°C. Successive samples of the liquid phase are taken through filter samplers during cooling to determine the progress of precipitation of compound $A_x Bi_y$ and to determine the extent of co-precipitation of metal B. Metal B is being introduced into the system in the form of a radioactive tracer to permit accurate measurement of its concentration in the 1-10 ppm range.

Precision Lattice Parameter Measurements of Zirconium-Oxygen Solutions. Using precision x-ray techniques, the lattice parameters of zirconium-oxygen solutions from 0 to 1 wt. % oxygen will be measured at room temperature. Considerable care will be taken to ensure purity of constituents and homogeneity of solutions. The temperature during exposure will be controlled to 0.1°C. It is hoped that the results may be used for analytical determination of oxygen in zirconium.

<u>Two-Dimensional Ductility in Be Sheet</u>. Be sheet having two-dimensional ductility (of the order 30-40% elongation) should be obtainable by the hot rolling of sintered, pressed powder, as it has been obtained by the cross rolling of extruded powder. The various factors involved are being investigated.

Radiation Damage in Cu-Fe Alloys. An investigation of the effects of radiation damage on Cu-Fe, the precipitation hardening alloy, is in progress. A series of alloys between 0-5 wt. % Fe in Cu have been studied using magnetic, electrical resistivity, and hardness measurements. Neutron irradiations in fast flux regions have been carried out in the reactors at Brookhaven and Arco.

Deformation Processes for Zr. A study of the deformation processes occurring in good quality, single crystals of zirconium has been in progress. The slip and twin systems have been determined at room temperature, and a value for the critical resolved shear stress for slip has been obtained. Further study of the twinning systems is in progress.

Abnormal Grain Growth in Uranium. It is found that an extremely coarse grain size develops in uranium when extruded under certain conditions. The purpose of this study is to determine the reasons for this phenomenon.

Magnetic Susceptibility Studies. The purpose of this work is to learn more about the metallic state in metals and alloys through observation of magnetic behavior. Unusual metals and alloys on which there is little reliable magnetic data are being measured.

Contractor: General Electric Research Laboratory, Schenectady, New York

Contract: W-31-109-Eng-52

Brief Title: DEVELOPMENT OF ZIRCONIUM ALLOYS

Investigator: W. R. Hibbard, Jr.

Scope of Work

The aim of this project is to develop an understanding of the relationship between the structure and properties of zirconium alloys for the purpose of obtaining principals on which to base the development of improved structural alloys for nuclear reactors. Work has been completed on the mechanical properties of unalloyed zirconium as a function of grain size, impurities, strain rate, strain, temperature, preferred orientation, and other variables. The mechanical properties, particularly tensile, up to high temperatures have been studied for a variety of binary alloys including the interstitials (oxygen and nitrogen), the elements which form eutectoids (Fe, Mo, Cr, Nb, Ta), and those which form peritectoids, such as aluminum. The more interesting of these materials are being examined in stress-rupture tests at temperatures up to 500°C. Similar studies are in progress on ternary alloys based on zirconium-aluminum.

Much effort is devoted to a study of the effect of size, shape, and distribution of precipitated particles on the properties of zirconium base alloys. These correlations are being interpreted and compared with the modern theories for hardening by a second phase. Contractor: Armour Research Foundation, Chicago, Illinois

Contract: AT(11-1)-315

Brief Title: A STUDY OF THE MECHANISMS OF HEAT TREATMENT OF ZIRCONIUM-BASE ALLOYS

Investigator: D. J. McPherson and R. F. Domagala

Scope of Work

During the past three years, Armour Research Foundation has established the phase relationships in nine zirconium-base binary systems. Current research is directed toward an investigation of the mechanisms of heat treatment of such alloys, the logical second prerequisite for development work.

Three prototype systems have been selected for study. TTT curves will be established for three alloys in each of the systems Zr-Mo (eutectoid), Zr-Sn (peritectoid), and Zr-Ti (α and β isomorphous). The curves will be delineated by a metallographic and resistivity study of isothermally transformed specimens. Tensile and impact tests will be performed to document the TTT curves with mechanical properties corresponding to the various conditions of transformation. A second year's work will be directed towards a study of TTT curves established for quenched and reheated specimens, also to be correlated with tensile and impact data. These curves and accompanying data should then serve as a firm basis for the selection of the heat treatment of any zirconium-base binary alloy to produce optimum structures and properties. Contractor: Case Institute of Technology, Cleveland, Ohio

Contract: AT (11-1)-258

Brief Title: SCALING OF ZIRCONIUM AT ELEVATED TEMPERATURES

Investigator: W. M. Baldwin, Jr., D. J. Garibotti, and H. M. Green

Scope of Work

The purpose of this investigation is to gain a full understanding of the nature and cause of the following unusual features, observed in the scaling of zirconium in air: (1) the nature of the scale (black and compact, or white and porous) is temperature and time dependent; (2) zirconium strip undergoes large dimensional changes during scaling; and (3) the scaling rate increases tremendously after a critical time during isothermal runs.

The problem has been attacked by scaling studies of high purity zirconium in air, oxygen, nitrogen, and mixtures of oxygen and nitrogen. The unusual features observed in air were not found with pure oxygen or nitrogen, but were found with mixture of these two gases. Studies of other atmospheres have firmly established that the joint presence of oxygen and nitrogen is necessary for the unusual scaling behavior of zirconium.

As a result of the considerable attention directed to the cause of dimensional instability of zirconium, one of two possibilities is suggested: (1) the surface layers of the metal deform, thus cracking the scale, or (2) the scale expands and 'drags' the metal along with it. Experiments involving metallographic, X-ray, electrical conductivity, and microhardness tests are under way in an attempt to correlate the scale and metal properties with the oxidation rate in various atmospheres.

Contractor: Oregon, University of, Eugene, Oregon

Contract: AT(45-1)-535

Brief Title: POLAROGRAPHIC STUDIES ON THE CORROSION OF ZIRCONIUM

Investigator: Pierre Van Rysselberghe

Scope of Work

The work under this project covers the electrochemical and polarographic studies on the corrosion of zirconium. A part of this work relates to the study of the rate of oxide film formation in the anodic polarization of zirconium over a range of temperature from 18° to 85°C using different alloys corroded by various aqueous media. The data obtained from this portion of the studies will be used to evaluate the fundamental constants (barrier height and half width in the metal oxide interface) of the Mott equation and their change with medium temperature and alloying constituents. An attempt will be made to show that the results obtained at unit current efficiency, working at potentials below one volt, can be extrapolated into the results obtained by Charlesby above 10 volts. By making all measurements at potentials below that of oxygen evolution, essentially only anodic potential-time measurements and their temperature dependence are necessary to evaluate the Mott constants. Basic information on the corrosion characteristics of the metal samples studied will be developed from the changes in the Mott constants with polarizing medium, temperature, and alloy constituents.

Another portion of this work relates to an examination of the behavior of zirconium in media where free radicals are produced, e.g., through a two-step reduction of hydrogen peroxide on mercury cathodes which has been adapted to the formation of OH and HO₂ radicals.

The polarographic part of the work will include a search for an indirect method (by differences after precipitation of zirconium by reagents such as cupferron) of determining millimolar concentrations of zirconium in aqueous media. Accompanying this will be a search for a direct method of determination based on the formation of a zirconium complex whose wave would correspond to the complex agent reducing at a higher cathodic potential than it does in the absence of zirconium. It is also planned to reinvestigate both the uncomplexed zirconium wave and the wave obtained with the oxalate complex by means of a differential polarograph.

Contract: NBS Project No. 4400

Brief Title: PROPERTIES OF CERAMICS AT ELEVATED TEMPERATURES

Investigator: R. F. Geller

Scope of Work

The purpose of this study is to develop optimum mechanical properties in pure UO_2 and ThO_2 , and to determine the elastic properties of ceramics at elevated temperatures.

Fused UO₂ and ThO₂ with a maximum addition, or impurity, of 2% will be used. Particlesize distribution, fabrication procedures, and conditions of heating will be controlled to obtain specimens with reproducible properties. These factors will be varied to produce maximum density specimens and also specimens with optimum strength in compression, bending and tension, and elasticity. Properties will be measured at 1000°C, and possibly at 1400°C.

The sonic method will be used to measure the purely elastic properties (Young's modulus, shear modulus, and Poisson's ratio) of carbides, cermets, intermetallic compounds, and oxides of interest.

Contractor: Battelle Memorial Institute, Columbus, Ohio

Contract: W-7405-eng-92

Brief Title: CERAMICS FOR HIGH-TEMPERATURE USE

Investigator: W. H. Duckworth

Scope of Work

The purpose of this project is to investigate unexplored compounds for new and useful ceramics having potential high-temperature applications. At present, few ceramic materials are available or can even be considered for some applications of AEC interest because of limitations in mechanical and thermal properties. It is proposed to synthesize refractory ceramic compounds and mixtures of compounds to determine the suitability of the resultant properties to meet program requirements.

Properties of ceramic bodies based on vanadates have been determined, and those based on tantalates are being studied. It is also planned to examine the properties of oxides of lanthanum, yttrium, and scandium. Contractor: Massachusetts Institute of Technology

Contract: AT(30-1)-960

Brief Title: THE MEASUREMENT OF THERMAL CONDUCTIVITY OF REFRACTORIES

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

Scope of Work

The purpose of this research is to investigate the factors affecting thermal conductivity of refractories, reliable methods of measurement, and to determine satisfactory values for refractory materials of interest.

Various methods of determining thermal conductivity in the temperature range from room temperature to 2000°C have been investigated. Procedures have been worked out to give reliable results. Data have been obtained over a wide temperature range for a number of pure refractory oxides, carbides, nitrides, and glassy materials. Additional materials are being tested as they become available.

Various factors affecting the thermal conductivity of refractories are being investigated. These include the temperature dependence of thermal conductivity, the effect of porosity, the effect of translucency and transparency on heat transfer through refractories by a radiation mechanism, the effects of combining two phases, the effects of solid solutions, and the effects of grain boundaries. At elevated temperatures, the emissivity of refractories becomes an important factor, and this is being investigated. The high temperature thermal conductivity of refractory powders and fibers is also being considered.

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Contractor:Chicago, University of, The Institute for the Study of Metals, Chicago, IllinoisContract:AT (11-1)-96Brief Title:RESEARCH ON THE STRUCTURE AND PROPERTIES OF GRAPHITEInvestigator:Lothar Meyer

Scope of Work

It was found that cold gas molecules such as helium, methane, ethane, and neon show an extremely small accommodation coefficient on hot graphite surfaces. Methane and ethane do not even decompose on graphite surfaces heated up to 2300°C as long as the gas pressure is so low that the mean free path exceeds the dimension of the vessel. A systematic study of this effect has been started using an electronic device which keeps the resistance of a graphite filament constant, and records the change in current necessary to keep the resistance constant when one of these gases is admitted to the chamber containing the filament. The accommodation coefficient of several gases will be measured as a function of the temperature of the graphite surface, the temperature of the gas itself, and the pressure of the gas.

The ultimate aim is to grow graphite by decomposition of hydrocarbons in a controlled manner at very low pressure by preheating the gas so far that it just does not decompose homogenously but will do so when impinging on the still hotter graphite surface. Contractor:Alfred University, Alfred, N. Y.Contract:AT(30-1)-1297Brief Title:GRAPHITIZATION OF CARBONInvestigator:V. D. Frechette

Scope of Work

The project includes the study of methods for forming graphite compacts, with special attention to the density, strength, and microscopic structure of the products. Compacts will be formed (1) especially by cold compaction of synthetic polycrystalline graphite and chemically purified natural flake graphites with and without prealignment of the grains and with various degrees of previous wet oxidation and (2) by partial wet oxidation and peptization to form a dense sol, condensation, and hydrostatic compaction. In both cases, the compacts will be subsequently heated to graphitizing temperatures (1) without pressure and (2) with pressures up to 5,000 pounds per square inch.

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

Contract: AT(30-1)-1710

Brief Title: FACTORS AFFECTING THE MECHANISM OF GRAPHITIZATION AND THE HETEROGENEOUS GAS REACTION OF GRAPHITE

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

Scope of Work

The purpose of the work is two-fold. First, factors affecting the mechanism of graphitization are being studied in an attempt to lay a fundamental basis for predicting the graphitizability of carbonaceous materials. Second, the heterogeneous gas reactions of carbons are being investigated with the aim of successfully predicting the relative gasification rate of a particular carbon from its physical and chemical properties.

The mechanism of graphitization is being studied, using pure organic compounds as starting materials. The early stages of the cracking and condensation or polymerization of these starting materials are being studied in detail and are being correlated with structure studies on the chars and graphitized carbons subsequently produced by further thermal treatment.

Reactions of graphites and carbons, both in particle and extruded shape forms, with carbon dioxide are being studied over the temperature range 500°C to 1400°C. Their gasification rates are being correlated with unit cell dimensions and crystallite sizes, surface areas, pore size distribution, true and apparent densities, porosity, quantitative and qualitative mineral matter content, internal gas diffusion rates, and resistivity. Changes in these properties of the residual carbon upon gasification are also being investigated.

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 project is to study in a most general way the influence of all variables involved in the processes of manufacture of carbons on the properties of the final product, in order to clarify the nature of the fundamental physical relationships and of the transformations taking place at each step of the manufacturing process. The ultimate goal of this research is an exact formulation of conditions necessary for manufacture of carbons and graphites with definite physical properties. It is planned, therefore, to study the structure of cokes and their characteristic differences, which lead to differences in the final carbon products, as functions of heat treatment, state of subdivision, porosity, etc. Furthermore, it is planned to investigate such physical properties of cokes, green, baked, and graphitized carbons, as density, elasticity, mechanical strength, thermal expansion, thermal conduction, and others as functions of the above variables. These properties will also be studied as functions of the temperature, calcination temperature of the coke, formulation of the mix, temperature and time of mixing, binder type, shape and size of particles, and the influence of the flow conditions in molding and extrusion (temperature, pressure, and speed of extrusion). Contractor: (The) Ohio State University, Columbus, Ohio

Contract: AT(11-1)-265

Brief Title: INVESTIGATION OF FLOW OF GASES THROUGH PERFORATED MEMBRANES Investigator: Edward Mack, Jr.

Scope of Work

This work is concerned with the porosity or flow characteristics of perforated membranes, as determined by the size, number, and spacing of the holes, and by the thickness of the thin foil or sheet in which the holes are made. These studies amount to being an extension of the work, reported in many papers in the literature, on the Brown-Escombe effect.

This effect was discovered by the English botany professor, H. T. Brown, and his colleague, F. Escombe, while they were studying the diffusion of gases through the stomata of leaves. Their findings, checked also with perforated membranes, established the fact that the rate of diffusion through a set of properly spaced tiny holes is as great, or almost as great, as though the whole leaf or membrane were an open aperture. This surprising result follows from the dependence of the rate of diffusion on the diameter, or perimeter, rather than on the area of the small hole.

Thus far, the smallest hole diameter for which studies are reported is 8-10 microns. Hole diameters in stages down to 1 micron or smaller are being used, and work is planned with foil or sheet thin enough at every stage so that the holes will give orifice flow. By working with sheets of several thicknesses, one would expect to be able to extrapolate to the flow at zero thickness of sheet.

Contractor: Wichita, University of, Wichita, Kansas Contract: AT(11-1)-203, Supplement No. 3 Brief Title: THE PERMEABILITY METHOD OF DETERMINING SURFACE AREAS OF FINE-

LY DIVIDED MATERIALS

Investigator: L. L. Lyon and G. Crocker

Scope of Work

The primary objective of this program is the delineation of the various factors influencing the rate of flow of a fluid through a porous medium across which a pressure differential has been established.

The flow of a non-adsorbable gas, such as helium, through a porous medium can be accounted for in terms of viscous and diffusive effects. In the case of an adsorbable gas, flow may also occur in the sorbed phase. This type of transport is referred to in the literature as surface flow. Thus, the conductance (F) of a gas through a porous medium can be described by the equation:

$$\mathbf{F} = \mathbf{F}_{\mathbf{v}} + \mathbf{F}_{\mathbf{d}} + \mathbf{F}_{\mathbf{s}}$$

F is the total conductance, F_v is the conductance due to viscous effects, F_d is the conductance due to the diffusive effects, and F_s is the conductance due to surface flow effects.

From the available information, two limiting cases of surface flow may exist. At low coverages (less than a monolayer of adsorbate present), surface flow should be due solely to mobility in the sorbed layer. At greater coverages, some of the pores of the porous medium may be filled by condensation, in which case the flow probably takes place by a mechanism quite different from mobility in a sorbed layer.

The experimental work of this project will investigate the conductance of various adsorbable gases through porous media that have a wide range of surface areas or pore diameters. The internal structures of the porous media will be carefully studied by the techniques of helium permeability and low-temperature nitrogen adsorption-desorption experiments. ____ Section II

ALLOY THEORY AND THE NATURE OF SOLIDS



Contractor: North American Aviation, Inc., Downey, California

Contract: AT(11-1)-Gen 8

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

Investigator: Sidney Siegel and Dwain B. Bowen

Scope of Work

The nature of the electron structure of Th, U and Pu is being determined from measurements of the electrical conductivity, the Hall coefficient, the magneto-resistance, the susceptibility, the thermal conductivity, and the thermoelectric power as a function of temperature. A concurrent theoretical program is being carried out to provide a model of the electronic bond structure of these complex metals. The principal approximate methods employed are the tight binding and the Fermi-Thomas statistical model.

It is believed that studies of this nature will help in understanding the striking changes in structure that occur from one member of the series to the next and the changes in phase that occur with variations in the temperature and/or pressure. A particular attempt will be made (1) to correlate the fundamental properties with the properties and characteristics of interest for applications of the metals as reactor-fuel materials, and (2) to obtain an understanding of the alloying characteristics of the three metals in those systems of interest for improved radiation stability and/or phase stability.

Contractor: North American Aviation, Inc., Downey, California

Contract: AT(11-1)-Gen 8

Brief Title: HIGH TEMPERATURE EFFECTS IN THORIUM, URANIUM, AND PLUTONIUM Investigator: Sidney Siegel and Dwain B. Bowen

Scope of Work

The purpose of this investigation is to determine the influence of macroscopic elastic strain on the band structure and the spectrum of lattice oscillations of Th, U, and Pu as a function of temperature, with a view toward understanding the changes in electronic, thermodynamic, and rate-dependent properties that are induced by precipitate strain. Measurements of Th, U, and Pu physical properties as a function of elastic strain and temperature will be correlated with corresponding measurements on alloys with internal strains introduced by a second-phase precipitate. Knowing the relative free energy in various conditions of strain, it should be possible to infer the texture and form of internal precipitates that provide the greatest dimensional stability. The influence of these strains on transformation kinetics will also be evaluated.

An attempt will also be made to evaluate the effects of strains produced by large quantities of fission products on the properties of the pure metals and their alloys, under conditions of high burnup.

Contractor: Ohio State University Research Foundation, Columbus, Ohio

Contract: AT(11-1)-191

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

Investigator: C. H. Shaw

Scope of Work

The metals of particular interest in this work are titanium, zirconium, hafnium, and thorium and their alloys. One of the determining factors in the fine structure of the emission lines and absorption edges is the density of electronic states in the conduction band of the metal. It is the purpose of this research to measure such structure under high resolving power and with adequate precision to allow band structure studies to be made from the data.

So far, data have been completed for the emission lines of zirconium. Unexpected differences in the structure of certain of the lines are being examined theoretically.

Equipment has been completed for the preparation of thin films of these metals by sputtering, and a thin film sputtered on thin beryllium metal is being prepared at present. These films will be used for absorption work.

A major consideration in the apparatus continues to be the contamination of the sample when under electron bombardment. Although data are obtained with a registered vacuum of 10^{-6} mm Hg, the rate of target contamination is still too high. Modifications in pump design are under consideration to improve the ultimate vacuum.

Contractor: Carnegie Institute of Technology, Pittsburgh, Pennsylvania

Contract: AT(30-1)-1596

Brief Title: PHYSICAL PROPERTIES OF RARE METALS AND THEIR ALLOYS

Investigator: J. E. Goldman

Scope of Work

The objective of this research investigation is to determine the influence of the properties of the electron gas in certain metals and their alloys on the phase transitions that are observed. More specifically, it is known that the metals titanium, zirconium, and hafnium have allotropic modifications that become stable at higher temperatures. Small amounts of alloying elements, e.g., silver, aluminum, etc., affect the stability of the phase very appreciably and, in that way, alter the temperature at which the transformation is observed. It appears that this could be a manifestation of the theory originally promulgated by Jones which suggests that this is attributable to the changing number of electrons in the "Fermi Distribution" caused by the addition of electropositive elements. It is desirable to attempt to confirm this. There are three types of measurements which give fairly direct information about the electrons. These are specific heat at very low temperatures $(2^\circ - 4^\circ K)$, magnetic susceptibility, and Hall effect. All three types of measurements are being made. Contractor: Illinois Institute of Technology, Chicago, Illinois

Contract: AT(11-1)-90, Project No. 5

Brief Title: INVESTIGATION OF IMPERFECTIONS IN SOLIDS

Investigator: Theodore J. Neubert

Scope of Work

The subjects of principal concern are lattice vacancies, dislocations, color centers, impurity ions, and order-disorder transitions in ionic crystals. Although the research must ultimately be extended to crystals more complicated than the alkali halides, work for the present is to be limited to face-centered cubic crystals. In non-cubic crystals, the color centers produced by x-irradiation are likely to be so different from those observed in alkali halide crystals that little analogical use can be made of alkali halide information. By investigation of "alkali halide type" crystals in which there are more-complex anions or cations, or accessible phase transitions introducing relatively minor alterations in crystal structure, it is hoped that the new information obtained will be helpful in the understanding of phenomena in more complicated crystals. Some work has been done on NaCN, and work on KPF₆ is being started.

The general interest in lattice vacancies and order-disorder transitions is being carried forward by an investigation of the dielectric properties of silver tetraiodomercurate which may involve study of the kinetics of the ordering and disordering process.

Lattice vacancies, dislocations, and effects of impurity ions are, pending the resolution of certain experimental difficulties, to be investigated by study of the mechanical properties (elastic modulus and internal friction) of single crystals and polycrystalline aggregates.

Contractor: Pittsburgh, University of, Pittsburgh, Pa.

Contract: AT(30-1)-647

Brief Title: APPLICATION OF CHEMICAL THERMODYNAMICS TO THE STUDY OF ALLOY-ING PROCESSES

Investigator: W. E. Wallace

Scope of Work

The general aim of the present work is (1) to develop experimental methods for obtaining highly precise thermodynamic quantities for substitutional solid solutions, and ordered or disordered metallic solutions (intermetallic compounds, double salt solid solutions, etc.), (2) to apply the experimental methods developed to selected alloy systems, and (3) to employ the resulting data to attain a better understanding of alloying processes. The thermodynamic quantities being measured include (a) heats, entropies, and free energies of formation of substitutional solid solution or, more specifically, of alloying, (b) low-temperature specific heats by adiabatic calorimetry, (c) changes in configurational energy and entropy associated with orderdisorder transformations, and (d) volume vs temperature, and volume vs composition relationships in substitutional solid solutions.

Measurements under way at present seek (1) to better the residual entropy measurement made in this Laboratory for the superlattices $MgCd_3$ and MgCd, (2) to obtain a residual entropy measurement for superlattices Mg_3Cd and Na_2K , (3) to relate the residual entropies to the observed number of Schottky defects, (4) to develop an automatic control for the adiabatic shield in the low-temperature heat calorimetry, and (5), by x-ray diffraction measurements, to establish the nature of certain thermal anomalies observed in the $MgCd_3$ superlattice. Contractor: Iowa, State University of, Iowa City, Iowa Contract: AT(11-1)-72, Project No. 4. Brief Title: THE STRUCTURES OF INTERMETALLIC COMPOUNDS Investigator: Norman C. Baenziger

Scope of Work

The purpose of the research program is to gain more information on the interaction of metals with one another to form intermetallic compounds. The research is concerned primarily with the intermetallic compounds formed between the metals of group II-B (Be, Zn, Cd, Hg) and alkali, alkaline earth, and rare earth metals.

Additional compounds of the $NaZn_{13}$ type were found in rare-earth beryllium systems; the magnetic properties of the $NaZn_{13}$ type compounds were studied. The crystal structures of $BaCd_{11}$, $SrCd_{11}$, $LaZn_{11}$, $PrZn_{11}$, $CeZn_{11}$, NaHg, Na_3Hg_2 , $NaHg_2$, KHg, KHg_2 , $CaBe_{13}$, $LaBe_{13}$, $PrBe_{13}$, $NdBe_{13}$, $ZrBe_2$, ZrBeSi, $BaZn_5$, $SrHg_{11}$, and KHg_{11} have been determined.

The structural work on additional compounds in the K-Hg, Na-Hg, Ba-Zn, and Sr-Zn systems will be carried on, as well as investigations of ternary systems, to explore the transition between the $NaZn_{13}$ type to the $BaCd_{11}$ type. A diffraction study of the NH_4 -Hg amalgam structure is also being made.

Contractor: Massachusetts Institute of Technology, Cambridge, Mass.

Contract: AT(30-1)-1002

Brief Title: FUNDAMENTALS OF SOLID SOLUTIONS AND RECRYSTALLIZATION PHENOMENA

Investigator: B. L. Averbach, Morris Cohen, and Michael B. Bever

Scope of Work

Scope I - Metallurgical Thermodynamics - M. B. Bever

The major effort of this project is devoted to investigations of energy effects associated with plastic deformation and solid state reactions, and their correlation with changes in other physical properties. A calorimetric technique employing tin as a solvent has been developed and is now capable of considerable precision. The energy stored during cold working of a goldsilver alloy has been measured for various deformation processes. Currently, this investigation is being extended to deformation at 77°K and to the effects of strain rate. The energy changes associated with order-disorder transformations in gold-copper alloys and heats of formation and precipitation in copper-tin alloys are also under investigation. An incidental result of the work is the measurement of heats of solution of various metals in tin.

In one investigation, the vapor-solid equilibria of alloys are determined by spectroscopic measurement of line intensities. The vapor pressure of zinc over solid silver-zinc alloys has been measured, and current work is concerned with silver-cadmium and silver-cadmium-zinc alloys. In another investigation, activities are measured electrochemically with solid electrolytes.

Scope II - Solid Solutions and Grain Boundaries - Morris Cohen

Thermodynamic and x-ray studies of solid solutions have been undertaken as an aid to the understanding of solid solutions and the nature of precipitation phenomena which occur on cooling. Data have been obtained for the gold-nickel and the aluminum-zinc systems. The work is being extended to copper-gold, cobalt-platinum, and aluminum-silver alloys. Diffuse scattering x-ray methods have been developed for evaluating the short-range order and the sizes of atoms in certain solid solutions. Electronic changes are observed by measurement of the Hall effect. Grain boundary energies are determined in copper-gold and copper-nickel alloys by means of an interferometric method which measures the groove angles that equilibrated grain boundaries make with a polished surface.

Scope III - The Fundamentals of Cold Work and Recrystallization - B. L. Averbach

X-ray methods, involving the Fourier analysis of the shape of diffraction lines, are being applied to the study of cold worked metals and to the basic phenomena involved in the recovery and recrystallization processes. Direct measurement of the particle sizes, local strains, and stacking faults are obtained. It is also possible to derive the total interfacial area from the particle size determinations, and the calculated interfacial energy is being correlated with stored-energy measurements. Among the materials being studied in various ways are alpha brass, cobalt and cobalt-nickel alloys, copper-silicon, aluminum, and zinc. Contractor:Illinois, University of, Urbana, IllinoisContract:AT(11-1)-67, Project No. 3Brief Title:DIFFUSION IN METALSInvestigator:F. Seitz and D. Lazarus

Scope of Work

Experimental and theoretical studies of the basic mechanisms of diffusion in metallic systems are undertaken. To date, particular attention has been devoted to precision radioactive tracer measurements of the migration of solvent and solute atoms in substitutional alloy systems in the terminal solid solutions. Silver alloys have provided a very rich field for investigation. It has been found that earlier measurements of diffusion coefficients in these systems are greatly in error, for reasons which remain unresolved. The new precision work shows that solute atoms diffuse faster than the solvent atoms in cases in which the former lie to the right of the solvent in the periodic chart (i.e. Cd, In, Sn, and Sb in silver), whereas they diffuse slower when they lie to the left. The effect is associated principally with a variation in the activation energy for diffusion, although the work shows that the D₀ coefficient in the expression $D = D_0 \exp(-E/RT)$ for the diffusion coefficient also depends in a systematic way upon the difference in atomic number of solvent and solute. Lazarus has developed a theory of the dependence of D₀ and E upon atomic number which gives a good account of the experimental facts and represents a quantitative refinement of the qualitative "Johnson-Wagner" theory.

One of the significant innovations of the research program has been the development of precision grinding techniques which permit the removal of very thin layers of material from the diffusion zone and hence permit the determination of very small diffusion coefficients, as well as accurate measurements using isotopes of short half-life.

Contractor: Rensselaer Polytechnic Institute, Troy, New York

Contract: AT(30-1)-1044

Brief Title: ANISOTROPIC DIFFUSION IN METALS

Investigator: H. B. Huntington and J. R. Neighbours

Scope of Work

The primary purpose of this project is to investigate diffusion in those metals where the effect is anisotropic. Measurements of the diffusion coefficients and activation energies for directions parallel and perpendicular to the hexagonal axis in zinc, cadmium, and thallium have been completed and published. A similar study on antimony is now under way, and, depending on these results, studies may be initiated on diffusion in other rhombohedral metals such as arsenic and bismuth. Further, it is hoped to study the anisotropy of diffusion in uranium and iridium.

These experimental results are being interpreted by a theoretical treatment of the entropies of lattice defects. In addition, a preliminary attempt is being made to gain an insight into the observed differences between the activation energies for self-diffusion parallel and perpendicular to the hexagonal axis in zirconium and cadmium; this to be accomplished by employing a semi-empirical Bond-Mayer force law representing the ion-core repulsion to estimate the energies of the saddle points for vacancy diffusion. Contractor: (The) Franklin Institute, Philadelphia, Pennsylvania

Contract: AT(30-1)-1484

Brief Title: STUDIES IN SOLID STATE PHYSICS

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

Scope of Work

The first phase of the work, now completed and published, was devoted to an experimental study of carbonyl iron including (1) strain and crystallite size in the untreated powders, (2) annealing kinetics, and (3) isothermal annealing studies. The other aspect of the project is a study of diffusion in metals on which work has been completed for a comparison of diffusion techniques employing radioisotopes, and an investigation of diffusion in single and polycrystals of zinc at low temperatures. In the current program, the latter aspect is now being continued and extended by investigating the effects of lineage boundaries on diffusion in zinc single crystals. Experiments are planned with single crystals in which the basal plane is parallel and perpendicular to the direction of growth so as to observe the influence of dislocation pipe orientation on diffusion. Studies are also planned to investigate the effect of purity on the formation of lineage boundaries and their behavior in diffusion, and, further, it is hoped to grow more perfect single crystals by the strain-anneal technique for comparison of the diffusion behavior.

A long-range program has been initiated on the effects of various physical parameters in diffusion. Specifically, these parameters are the atomic size, valence, and crystal structure of both solute and solvent. The work is intended to demonstrate the effects of changes in these parameters on the diffusion coefficients and the activation energies and compare these results with the current theories for diffusion in dilute solid solution.

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Contractor: Carnegie Institute of Technology, Pittsburgh, Pa.

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

Brief Title: PROPERTIES OF GRAIN BOUNDARIES AND SMALL-ANGLE X-RAY SCATTERING

Investigator: R. Smoluchowski

Scope of Work

Scope I - Grain Boundaries

The main results obtained during the last year are as follows: A study of anisotropy of the diffusion of silver along grain boundaries of columnar bi-crystals of copper using a radioactive tracer technique has been completed. The results show a high diffusion anisotropy which was predicted on the basis of the previously proposed model. This anisotropy is highest at low angles between grains decreasing to zero at 45° where the uniform slab model applied. Another result obtained in a completed study of self-diffusion along grain boundaries in columnar bi-crystals of iron (containing about 3% silicon) is that there is no simple relation between the energy of the grain boundary and the depth of diffusion, whereas there is a good correlation between the density of atoms of misfit in grain boundaries (at cusp positions) and the depth of diffusion. The immediate future program is to complete the study of grain boundary diffusion in the copper-aluminum system, which should clarify the role of atomic size in grain boundary diffusion. Another part of the program which is under way is the study of properties of pure twist grain boundaries, i.e., made out of screw dislocations; this will be then extended to more general grain boundaries, such as occur in random polycrystalline materials. Diffusion paral-lel and perpendicular to an array of edge dislocations is also being studied.

Scope II - Small-angle X-ray Scattering

During the last year the construction of the high intensity camera has been completed and applied to the study of imperfections in diamond, revealing surface defects of the order of one micron and the absence of proton-irradiation effects. A theory of x-ray scattering by surface defects and by dislocations has been developed. The future plan is to study small-angle scattering by an array of parallel edge dislocations and to compare it with the theoretical predictions. It is intended to study formation and clustering of vacancies in metals upon heat treatment. Similar studies will be made by means of a fine-focus Guinier type x-ray tube.

Contractor: Sylvania Electric Products, Inc., Bayside, N. Y.

Contract: AT(30-1)-Gen 366

Brief Title: GRAIN BOUNDARY EFFECTS IN THE SINTERING OF METALS AND OXIDES Investigator: L. L. Seigle

Scope of Work

A study will be made of the effect of grain boundaries in the sintering of pure metals and oxides in order (1) to evaluate the role of grain boundaries in the sintering of pure oxides, (2) to determine the influence of structural factors on the sintering of metal-oxide mixtures, and (3) to continue investigation of the effects of grain boundaries on sintering rates in metals.

It appears that grain boundaries play a decisive role in the removal of porosity from metal compacts, since once grain boundaries have been sufficiently eliminated by grain growth, the process of densification practically ends. Thus, the kinetics of metal sintering can scarcely be explained on the basis of models which assume an isotropic material.

It seems quite possible that grain boundaries play a role in the sintering of crystalline oxides similar to that in metals. Moreover, metal-oxide interfaces may also behave in this manner.

Contractor: Massachusetts Institute of Technology, Cambridge, Massachusetts

Contract: AT(30-1)-1192

Brief Title: METAL-CERAMIC INTERACTIONS AT ELEVATED TEMPERATURES

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

Scope of Work

The purpose of this research is to investigate fundamental factors of importance in the interaction of metals and ceramic materials at elevated temperatures, with particular reference to factors which may affect the fabrication of metal-ceramic composites. These factors include chemical reactions at interfaces, interface and surface energy, wettability, effects of heat treatment, and properties of metal-ceramic composite bodies.

The main factors involved in combining metals and ceramic materials in composite bodies are the surface tensions, interfacial energies, and wettability of the phases in contact. The major area of research on this program has been investigation of these effects. Suitable methods have been developed, and it has been found that small additions of other materials (as little as 0.01%) have a pronounced affect on these properties. The effects of these minor additions to nickel and iron in contact with Al_2O_3 are being investigated. In conjunction with this, the surface energy of additional pure metals and ceramic materials is being studied. In addition to the direct measurements of surface and interface energy, studies of the effect of these variables on the sintering process and on metal-ceramic microstructure and properties are being investigated. Contractor: California, University of, College of Engineering, Berkeley, California

Contract: AT(11-1)-34, Project No. 7

Brief Title: MECHANICS OF METAL-CERAMIC BONDS

Investigator: J. A. Pask

Scope of Work

The objective of this research program is an investigation of the nature and strength of the bonds existing at the contact or interface of a metal phase and a glass or ceramic phase, such as those existing in metal-ceramic seals and protective coatings on metals.

Preliminary studies have indicated that all metals, except gold, are completely wetted by binary glasses in air and that various degrees of wetting are exhibited in vacuum. Also, there was evidence that reactions were occurring between some of the metals and the glasses. Further work has shown that (1) the interfacial energy between simple glasses and non-reactive metals in vacuum is dependent upon temperature, and (2) in vacuum, certain metals can reduce Na₂O in a sodium silicate glass to form sodium gas and a sodium-metal oxide compound.

Experimental studies now under way and contemplated for the coming year are the determination of (1) the effect of metal-ion additions to the glass on the wetting of metals by sodium silicate glass, (2) the thermodynamics of glass-metal reactions in vacuum to form new compounds, (3) further studies on the temperature dependence of the interfacial energy between glass and metal in vacuum, and (4) the effect of oxygen pressure on the wettability of certain metals by certain glasses. 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 research is divided into two phases, as follows:

1. The present program of measuring the creep of sintered alumina will be continued to include data on the effect of additives (impurities) on the strength and creep properties of alumina. In addition, it is hoped that the effect of crystal size on the rate of creep and on the tensile strength may be established.

2. The sintering process is often accompanied by grain or crystal growth. The purpose of this phase of the work will be to differentiate between sintering and the concurrent crystal growth. The determination of the rate of recrystallization will be made at selected temperatures on selected oxides by measuring the variables of time and crystal size. For large crystals, a thin-section technique will be employed to designate size. It is hoped that small crystals may be recognized with the aid of the electron microscope.

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

Contract: AT(30-1)-Gen 366

Brief Title: MECHANISM OF DIFFUSIONAL BONDING OF ALUMINUM TO NICKEL

Investigator: L. L. Seigle

Scope of Work

Investigations of the solid state bonding of aluminum to nickel in cladding operations indicate that the size and nature of the diffusion zone are significantly affected by variations in applied pressure. It has been found that increasing pressure decreases the thickness of intermediate layers in the diffusion zone and markedly increases the tensile strength of the bond.

Apparently, there has been no prior appreciation of the effectiveness of applied pressure in inhibiting the formation of intermetallic layers during diffusion. Accordingly, it is proposed to investigate this phenomenon in greater detail in the Al-Ni system to determine the underlying courses and ascertain whether this may be a general effect.

It is proposed to conduct a thorough analysis of the diffusion processes taking place during the formation of the Ni-Al bond at atmospheric pressure and at one elevated pressure, for example 30,000 p.s.i.

Contractor: General Electric Research Laboratory, Schenectady, New York

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

Brief Title: FUNDAMENTAL METALLURGICAL RESEARCH

Investigator: D. Turnbull

Scope of Work

This project consists of two principal phases: research on the thermodynamics of solids and solid solutions, and investigations of diffusion in metals. In the thermodynamic work a study of the order-disorder transformations in copper-gold alloys is being continued. Measurements of activity coefficients are being correlated with the structure of the alloys. Measurements are being made of the thermoelectric power, electrical resistance, and Hall coefficients for these alloys to provide data for an interpretation of ordering on the basis of the development of lattice strains. The heats of solution for copper-gold alloys are being measured for both the liquid and solid states to isolate the strain energy turn. In addition, some supplementary lowtemperature specific heat measurements are being made for the alloys in the ordered and disordered states.

Another aspect of the thermodynamic work is the study of the specific heat of anisotropic lattices. The previous work is being extended to measurements on graphite and selenium in the liquid helium temperature range where, as shown previously, there are departures from the Debye law for anisotropic crystals.

In the diffusion studies, the two important factors being investigated are: the influence of orientation on grain boundary self-diffusion and the effect of impurities on self-diffusion in metals. In the study of grain boundary diffusion, an investigation is being made of the influence of relative crystallographic orientations of adjacent grains on the rate of atom mobility. These measurements are interpreted in terms of the concentration of dislocations in the grain boundaries and have shown that dislocation channels increase the rate of self-diffusion in metals. Additional measurements on the rate of self-diffusion in directions parallel and perpendicular to the dislocation pipes in bicrystals have confirmed the previous findings.

When one atomic percent of lead is added to a solid solution of silver, the self-diffusion coefficient of silver is roughly doubled, and the self-diffusion coefficient of lead is approximately 12 times the silver self-diffusion coefficient of dilute solution. This phenomenon is being investigated by determining the self-diffusion coefficients and activation energies for diffusion in binary solid solutions of silver containing lead, silver, copper, thallium, and others.

Contractor: Tennessee, University of, Knoxville, Tennessee

Contract: AT(40-1)-1068

Brief Title: ENERGY CHANGES FROM PLASTIC DEFORMATION

Investigator: E. E. Stansbury

Scope of Work

Experimental phases of this project have been directed toward development of adiabatic type calorimeters for operation over the temperature range 0 to 1000°C. Problems of thermocouple stability in vacuum, automatic adiabatic shield control, and conductivity of refractories have been under investigation. Calorimetric measurements of specific heats, heats of transformation and solution in solid alloy systems, and related phenomena are possible. Cylindrical specimens weighing 30 to 200 grams may be used. Measurements are usually made on continuous heating although some isothermal studies of phase transformations appear possible.

Present research is being directed toward (1) measurements of stored energy in plastically deformed metals and alloys and (2) thermodynamic measurements on iron, zirconium, and binary alloys based on these systems. Specific heats and heats of transformation of pure iron have been determined, which are consistent with data on iron-carbon alloys. Contractor: Illinois, University of, Urbana, Illinois Contract: At (11-1) - 67, Project No. 9 Brief Title: ANNEALING OF COLD WORKED METALS

Investigator: Paul A. Beck

Scope of Work

It is well known that annealing produces a variety of property changes in cold worked metals. These changes are associated with alterations in structure, some of which do not involve boundary migration (recovery), whereas others are connected with sub-boundary migration (sub-grain growth) or with the migration of grain boundaries (recrystallization or grain growth).

The purpose of the project is to investigate the fundamental nature of the structural changes mentioned and to elucidate the main physical variables affecting them. Accordingly, the scope of the project includes the study of recovery in cold worked metals, of sub-grain growth and of recrystallization.

An important part of the work is directed to the study of reorientation phenomena, resulting from grain boundary migration; a major aim of the project is the development of a generally applicable theory of annealing textures. In order to bring about a definite decision between the two major contending hypotheses, namely "oriented growth" and "oriented nucleation," further experiments will have to be carried out, involving the quantitative determination of deformation and annealing textures.

The sub-grain formation and other crystal imperfections resulting from plastic deformation and the changes on annealing are to be studied by refined X-ray methods, such as the quantitative measurement of line broadening, microbeam techniques, a convergent monochromatic beam X-ray technique, and by electron diffraction and transmission electron microscopy. Contractor:New York University, New York, New YorkContract:To be assigned.Brief Title:THE ORIGIN OF RECRYSTALLIZATION NUCLEIInvestigator:John P. Nielsen

Scope of Work

This work is based on a theory of the origin of recrystallization nuclei, particularly for secondary recrystallization, involving only grain boundary interactions. When two grains in a polycrystalline specimen meet at a point in the course of grain-boundary movements, and the new boundary created at the point is one of relatively low specific free energy, a non-equilibrium boundary condition occurs. The non-equilibrium is enhanced if the other grain boundaries involved at the point of meeting are relatively high in specific free energy. The non-equilibrium results in a correction by growth of the complex grain (two subgrains) to a large size, sufficient in many cases for it to become a self-propagating unit, i.e., a recrystallization grain. This mechanism, called "geometrical coalescence," is considered a logical origin for recrystallization nuclei, particularly for secondary recrystallization.

If this theory for the origin of secondary recrystallization nuclei is found to be correct, a basis will be established for a theory of the origin of primary recrystallization. The experimental program will be confined entirely to two-dimensional grain boundary networks which are obtained by cutting thin slabs from columnar grain specimens such that the grain boundary networks which aries of the samples are perpendicular to the surface of the slab. Grain boundary movement data are to be obtained by interrupting the annealing treatment at suitable intervals to obtain a complete grain boundary configuration by metallography. Some boundary migration velocities may also be obtained which may be helpful in determining activation energies for boundary movement.

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Contractor: Massachusetts Institute of Technology, Cambridge, Mass.

Contract: AT(30-1)-1310

Brief Title: A STUDY OF SOME OF THE MECHANICAL PROPERTIES OF METALS AT LOW TEMPERATURES

Investigator: Z. S. Basinski

Scope of Work

The purpose of the work is to obtain information that might lead to (1) a better understanding of lattice defects in metals, such as dislocations, vacant lattice sites, and interstitial atoms, (2) the generation of such defects, especially during deformation, (3) their interaction with one another and their annealing, and (4) their general role in plastic deformation. It might be expected that the imperfections would affect the specific heat at low temperatures, and thus might change significantly the deformation characteristics at low temperatures.

The materials used will probably be Al, Cu, Ag, and Fe of high purity and their dilute alloys. The study will be conducted on single crystal and possibly polycrystalline specimens. The specimens will be extended at various temperatures down to 4°K, and measurements will be made of flow stresses, strain hardening, resistivity, and stored energy; metallographic examination will also be used. Such measurements will also be made on specimens prestrained at different temperatures to show the effect of the history of the specimens on their deformation characteristics, and possibly to emphasize the differences in the deformation mechanisms that are involved. The internal friction of annealed and variously cold worked metals of very high purity, and of annealed dilute alloys might also be measured to throw light on the interactions between dislocations, vacancies, interstitials, and solute atoms. Contractor: California, University of, Berkeley, California

Contract: AT(11-1)-GEN-10, Project No. 1

Brief Title: CREEP OF ALLOYS

Investigator: E. R. Parker

Scope of Work

The object of this investigation is to study the effect of mechanical, thermal, and metallurgical factors on the creep resistance of alloys. Work to date in this field has demonstrated that a fundamental understanding of the creep of alloys depends upon (1) clarification of the role played by substructure on the properties of metals, (2) an understanding of the influence of solid solution alloy additions on the substructure, (3) a better understanding of the mechanisms of solution hardening, and (4) the effect of the nature, size, shape, and degree of dispersion of a second or higher order phase in multiphase alloys. Work has been concentrated during the past year on the first item, and results reported to date have aided considerably in clarifying the effect of substructure on mechanical properties. This program is being continued.

Quantitative data concerning the effects on mechanical properties of solid solution alloy additions to nickel have been obtained for a number of alloys. Several additional systems are currently being studied. The influence of short-range ordering on solid solution hardening is currently being investigated, and results of considerable interest have been obtained.

A program has also been initiated to study the nickel-titanium two-phase alloy region. This system has been thoroughly examined in the solid solution range.

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Contractor: Illinois, University of, Urbana, Illinois
Contract: AT(11-1)-67, Project No. 15
Brief Title: STUDY OF DIFFUSIONLESS PHASE CHANGES IN SOLID METALS AND ALLOYS
Investigator: T. A. Read

Scope of Work

The research is concerned with the dynamics of diffusionless phase changes in nonferrous metals. Much of the work is to be done on the transformations in the beta phase of the gold-cadmium system, which are characterized by the fact that they can be observed under the simplest conditions of transformation geometry, with a single plane interface between the two phases after partial transformation.

Striking features of these transformations are their failure to proceed isothermally and the stabilization against further transformation which occurs if cooling through the transformation range is interrupted. The mechanism of the relaxation process responsible for this behavior will be studied; initially, emphasis will be placed on the investigation of the effects of varying impurity contents. A related study will also be carried out on the relaxation effects which occur in the low-temperature phase after transformation is complete, and which are responsible for the unusual mechanical properties of this phase.

The transformation dynamics of these alloys may also be profoundly affected by the thermal history of the specimen before it is cooled to the transformation range. It is planned to study this phenomenon by a variety of techniques, including x-ray diffraction and measurements of electrical resistivity and density.

Whereas much of the work is to be carried out on the gold-cadmium alloys, it is not planned to confine the work to them. It is felt that studies of several alloy systems should be carried out in order that comparisons of their similarities and differences in behavior can be made, thus leading to conclusions of the widest range of validity. Contractor: Virginia, University of, Charlottesville, Virginia

Contract: AT(40-1)-1768

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

Investigator: Allan T. Gwathmey

Scope of Work

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

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 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. It is believed that screw dislocations can best be controlled in metals by growing small crystals from the vapor. Copper will probably be the first metal investigated.

The chemical properties of these small crystals which approach perfection will then be studied by methods which have been developed at this laboratory largely for investigating the properties of large single crystals in the neighborhood of $\frac{5}{8}$ in diameter.

Section III

RADIATION EFFECTS ON MATERIALS

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Contractor:Illinois, University of, Urbana, IllinoisContract:AT(11-1)-182Brief Title:RADIATION DAMAGE IN SOLIDSInvestigator:F. Seitz and J. Koehler

Scope of Work

Initial experiments, in which the resistivity changes produced in copper, silver, and gold by 12 MEV deuteron irradiation were measured, show that the simple theory of the density of displaced atoms developed by Seitz gives results which agree with experiment within a factor of two or three. The measurements also show that some annealing is produced by the irradiation itself, and that important thermal annealing occurs in these metals at 30° to 40° K.

Plans have been made to measure experimentally the concentrations of lattice vacancies and interstitials produced by irradiation at helium temperature. In addition, the changes in concentration which occur during annealing will be determined. This can be accomplished by observing the changes produced in the volume and the lattice parameter of a specimen by irradiation. The present plans call for an X-ray measurement of the lattice parameter on single crystals. The resulting data should show whether there are differences in the annealing of single and polycrystals.

It is also planned to undertake careful isothermal annealing studies to determine accurately the activation energy and the order of the annealing reaction associated with the annealing at 40° K.

Quenching experiments on pure gold wires suggest that the energy required to form a lattice vacancy is 1.28 ± 0.03 ev. Measurements of the annealing of the quenched-in vacancies are in progress. They presumably should give the activation energy required for the thermal motion of vacancies if the present interpretation of the experiment is correct. Contractor: Carnegie Institute of Technology, Pittsburgh, Pa.

Contract: AT(30-1)-1193

Brief Title: RADIATION EFFECTS IN SOLIDS

Investigator: R. Smoluchowski

Scope of Work

The main accomplishment of this contract within the last year is a continuation of an extensive investigation of changes of various properties of potassium chloride and sodium chloride upon irradiation by 440 Mev protons and by hard gamma rays. Large changes of electrical resistivity by a factor of 200 or more have been found and studied as a function of subsequent annealing. The results point to formation of, presumably neutral, clusters of defects. A qualitative support is obtained from optical absorption measurement. Preliminary results suggest that the rate of clustering is controlled by the migration of the slower negative ion vacancies. Changes of density of the order of 10^{-4} have been observed by means of a suspension method. A study of surface condition of x-ray and proton-irradiated crystals by means of multiple beam interferometry indicates that the mechanism of density change is an internal one rather than a diffusion of vacancies from the surface. The influence of proton irradiation and of quenching on x-ray coloration has been also studied. A cooperative research program with the Brookhaven National Laboratory on electrical conductivity changes in neutron-irradiated brasses has been continued. It appears quite likely that the observed drop of resistivity is connected with the formation of short range order.

The future plan is to complete a comprehensive study of conductivity, optical absorption, density, thermal conductivity, and specific heat of irradiated ionic crystals at various stages of clustering of defects. This may be supplemented by a small-angle x-ray scattering search for evidence of clustering. It is planned also to study the dependence of resistivity effects in metals upon energy of the incident protons. This may clarify the significance of recently proposed "star"-mechanism of lattice defect formation. Also, anisotropy of resistivity effects in hexagonal metals will be studied. A cooperative program with the Case Institute of Technology on influence of irradiation upon the elastic constants of alkali halides is being arranged. The joint program with the Brookhaven National Laboratory will be continued in search for an evidence of lowering of the thermal scattering of conduction electrons in irradiated metals. Contractor:Purdue University, Lafayette, IndianaContract:AT(11-1)-125Brief Title:RADIATION DAMAGE STUDIESInvestigator:Karl Lark-Horovitz

Scope of Work

The purpose of this work is the study of radiation effects on semi-conductors. These studies include measurement of (1) threshold energies for atom displacement into an interstitial position or removal from the lattice, (2) kinds of levels introduced by irradiation and the energy distribution of these levels as a function of the irradiating flux, (3) concentration of defects as influenced by the irradiating flux, (4) the scattering mechanism of the bombardmentintroduced defects, and (5) the stability of the bombardment-introduced defects. Measured properties of solids after irradiation include (1) mechanical properties, (2) elastic constants, (3) creep, (4) thermal properties, and (5) electrical properties. The work on the observed changes in properties also includes a theoretical treatment to explain the significance of these changes.

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Contractor: Brown University, Providence, Rhode Island

Contract: AT(30-1)-1772

Brief Title: RADIATION DAMAGE STUDIES

Investigator: R. Truell

Scope of Work:

The effects produced by neutron bombardment of single crystals of silicon are being studied by the use of both ultrasonic attenuation measurements and velocity or elastic moduli measurements over the frequency range from 5 to 500 mc/sec. The attenuation measurements are more sensitive to physical changes in the crystals, whereas the velocity or elastic moduli measurements are very useful in examining the anisotropy effects, especially when the changes in the solid are very large.

Preliminary work has indicated that the ultrasonic attenuation is increased about 50% over the frequency range of 50 to 300 mc/sec after neutron irradiation. This effect is being investigated both experimentally and theoretically.

The damage effects in a single crystal might well be expected to differ in the various crystallographic directions, and this anisotropy is being studied by measurements of both attenuation and elastic constants. With regard to the latter, preliminary work has shown an 8% decrease in c_{11} , a 28% decrease in c_{12} and a 17% increase in c_{44} . These results differ, not only in magnitude but in sign, with results for the changes in the elastic constants of silicon, as determined by other investigators who used the thermal diffuse x-ray scattering method. These changes as well as the discrepancy are being studied further.

In addition to silicon, other materials such as quartz, synthetic sapphire, and diamond will be investigated in this program.

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 these methods to typical metals, alloys, and inorganic compounds. The developments have included spectrometer techniques, experimental corrections, Fourier methods for analyzing the data, and methods for separating the x-ray effects resulting from different processes, such as fragmentation, random strains, stacking faults, interstitials, and vacancies. Pile-damaged materials such as Al, Cu-2 percent Si, LiF, and black P have been studied. Considerable emphasis has been placed on a simultaneous study of cold worked metal, and, in this connection, thorough studies of materials such as cold worked tungsten and cold worked alpha brass have been made.

In addition to the interest in the nature of the cold worked state, a knowledge of the similarities and differences between radiation damage and cold work is essential to a complete understanding of radiation damage. It is planned to continue to conduct these two studies in parallel. Contractor: Rutgers University, New Brunswick, N. J.

Contract: AT(30-1)-1730

Brief Title: FUNDAMENTAL STUDY OF RADIATION DAMAGE TO METALS AND ALLOYS

Investigator: Sigmund Weissmann

Scope of Work

It is the aim of this project to study:

1. Evidence of radiation damage of neutron-bombarded metal wires, metal powders, and metal single crystals by means of a special X-ray technique of high resolution which was developed at Rutgers University. This technique is based on the X-ray double crystal diffractometer principle.

2. Effects of annealing of radiation-damaged metals and alloys by means of the abovementioned technique. This study will entail low-temperature investigation as well as controlled experimentation at higher temperatures.

3. Anomalous X-ray scattering of radiation-damaged single metal crystals.

Contract: NBS Project No. 3271

Brief Title: THERMOCHEMICAL STUDY OF IRRADIATION EFFECTS

Investigator: E. J. Prosen

Scope of Work

The purpose of this study is to develop methods for measuring, and to make calorimetric determinations of, the increase in energy content of quartz, silicon carbide, and other insulators resulting from irradiation in nuclear reactors. Determinations will be made of total energy increase and of the relief afforded by annealing the insulators at various temperatures, after irradiation.

This is a cooperative program with Argonne National Laboratory and is an outgrowth and extension of the work performed at Argonne on radiation damage to insulators. Samples will be prepared and supplied by ANL.

Contractor: Bausch and Lomb Optical Co., Rochester, N. Y.

Contract: AT(30-1)-1312

Brief Title: IRRADIATION DAMAGE TO GLASS

Investigator: N. Kreidl

Scope of Work

Previous work on the coloration of glass by high energy radiation has shown the similarity between the optical effects produced in glass and those in the simple crystals. These have been interpreted with some success by analogy with theories proposed for crystals. Absorption bands interpreted as arising from F-centers and V-centers have been observed. The complex nature of glass compositions, however, and the poor ultraviolet transmission have limited the interpretations.

The success of the approach in investigating crystals and the need for such data in interpreting glass leads to the investigation of the simplest type of glass, the network formers: silicon dioxide, germanium dioxide, and boron oxide.

By controlled additions of alkaline oxides to the three network formers, it is proposed to investigate the influence of these most common network modifiers on the effects produced by radiation.

Work will also cover the investigation of some of the glasses selected from these groups to which transition elements have been added. This extended investigation will include low temperature irradiation and exposure as well as measurements of paramagnetic resonance. The program includes a study to determine if the density variations in glasses produced by thermal treatment and by radiation can be associated with the presence of vacancies made evident by differences in the degree of radiation coloration.

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