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ANNUAL PROGRESS REPORT

The Science of Materials

Contract AT(11-1)-1198

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U. S. Atomic Energy Commission

by the

Materials Research Laboratory

University of Illinois

Urbana, Illinois

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April 15, 1968

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Materials Research Laboratory

University of Illinois

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INTRODUCTION

The Materials Research Laboratory building was dedicated on May 26, 1967. The principal address was given by Dr. Frederick Seitz, President of the National Academy of Sciences, whose topic was "Trends on the Academic Scene."

A review of the U. S. Atomic Energy Commission Contract AT(11-1)-1198 "Research and Training in the Science of Materials" was held on April 20-21, 1967. Approximately 20 scientists from AEC Laboratories were in attendance.

The Laboratory building became available in the late winter and spring of 1966. During the past year, all facilities planned for the building were placed in operation. These facilities were:

- Accelerator Facility (3 MEV Van de Graaff)
- Electron Microscope Facility
- Electron Microprobe Facility
- Cryogenic Facility
- Chemical Analysis Facility
- Radiochemical Facility
- Infrared Spectrophotometric Facility
- Ultraviolet Spectrophotometric Facility
- X-Ray Facility
- Metallographic Facility
- High Temperature Facility
- Toxic Materials Facility
- Crystal Growth Facility
- Machine Shop
- Glass Blowing Shop

Primary support for the operation of the Accelerator, Electron Microprobe, X-Ray, Metallographic, and Crystal Growth Facilities is supplied by Contract AT(11-1)-1198. Work performed in the Machine Shop and Glass Blowing Shop is charged to the projects on an hourly basis. The primary support for the operations of the other facilities is derived from the ARPA SD-131 contract.

Research laboratory space in the Materials Research Laboratory building is occupied by all projects of Contract AT(11-1)-1198 except those of Professors Beck, Ricketts, Wayman, and Simmons. With the exception of Professor Koehler, whose AEC-supported research is entirely housed in the Materials Research Laboratory building, and the four mentioned above, the members of Contract AT(11-1)-1198 occupy research space in both the Materials Research Laboratory and their departmental buildings.

New Projects

One new project was initiated with support from Contract AT(11-1)-1198. It is:

Physics of Refractory Materials
Principal Investigator: Wendell S. Williams, Ph.D., Associate
Professor of Ceramic Engineering and of Physics

Arrivals

Two new faculty members joined the staff of Contract AT(11-1)-1198:

Akhira Ikushima, Ph.D., Research Assistant Professor of Physics joined the group of Professor A. Granato. Dr. Ikushima is on leave from Osaka University, Osaka, Japan.

Wendell S. Williams, Ph.D., Associate Professor of Ceramic Engineering and of Physics. Professor Williams was formerly a member of the Union Carbide Corporation's research staff.

Departures

Two faculty members terminated their association with the AEC contract and left the University of Illinois:

Gary S. Snow, Ph.D., Assistant Professor of Ceramics terminated on August 31, 1967, to accept a position with the Sandia Corporation, Albuquerque, New Mexico.

Joseph F. van Landuyt, Ph.D., Research Assistant Professor of Metallurgy, on leave from the University of Antwerp, Belgium, returned to this university on September 30, 1967.

Awards and Appointments

Professor Howard K. Birnbaum, Department of Mining, Metallurgy, and Petroleum Engineering, received a Guggenheim Fellowship for research in England during his sabbatical leave.

Professor Charles P. Slichter, Department of Physics, was elected to the National Academy of Sciences during June 1967.

Leaves of Absence

Professor Howard K. Birnbaum, Department of Mining, Metallurgy, and Petroleum Engineering is on sabbatical leave at AERE, Harwell, England, II Semester, 1967-68.

Foreign Travel

Foreign travel supported by the AEC contract was limited to the participation of Professor Marvin Metzger in the International Conference on Strength of Metals and Alloys, Tokyo, Japan, September 4-8, 1967, and associated visits to Japanese laboratories.

Other foreign travel by members of the contract was:

Professor Howard Birnbaum, International Conference on Strength of Metals and Alloys, Tokyo, Japan, September 4-8, 1967. (sponsored by NSF)

Professor Jack J. Gilman, AGARD Conference, Turin, Italy, April 1967. (sponsored by the Air Force)

Professor Charles A. Wert, NATO Conference on Refractory Metals, Oslo, Norway, August 1967. (sponsored by NATO)

Visitors

Professor Yasushige Fukano of Nagoya University, Japan, was Visiting Assistant Professor of Metallurgy from December 15, 1967 to December 14, 1968, to work with Professor C. M. Wayman.

Dr. D. A. Gerlich of Tel Aviv University, Israel, was Visiting Associate Professor of Physics from July 16 to October 15, 1967, to work with Professor A. Granato.

Brief visits to the contract's research groups were made by:

Dr. Michael Bevis, Department of Metallurgy, University of Liverpool, England, who gave a seminar to the members of Professor D. Lieberman's group on March 4, 1968.

Dr. Ulrich Franck, Institut für Physikalische Chemie, Techn. Hochs., Karlsruhe, West Germany, who gave a group seminar to the members of Professor H. Drickamer's project on May 17, 1967.

Dr. H. Wollenberger of Aachen, Germany, who discussed problems of radiation damage with Professor J. Koehler (October 5, 1967).

Other visitors to the Materials Research Laboratory included:

Dr. S. Bhagavantam, Science Advisor to the Ministry of Defence, New Delhi, India (October 12-18, 1967).

Dr. S. C. Jain, Associate Director, National Physical Laboratory; Head, Department of Physics, Indian Institute of Technology, New Delhi, India (June 16-17, 1967)

Professor Heinz Pick, Director, II Physikalisches Institute, Stuttgart, Germany (June 6-8, 1967).

Significant Results

Studies of self-diffusion of Ni in Ni-Co alloys by Birnbaum showed that stacking faults provide paths of enhanced diffusion in these crystals. High purity niobium crystals were grown in a vacuum of 10^{-10} torr.

The Lieberman-Bullough theory of "composite" martensite which accounts satisfactorily for the '225' and '259' habits in steels was generalized by Lieberman to describe martensite plates with other habits.

Nuclear magnetic resonance studies of impurities in aluminum by Rowland demonstrated that the electric quadrupole contribution to the local field is a major source of the resonance relaxation which therefore depends strongly on the properties of the solute.

It was established by Granato that the contribution of the conduction electrons in aluminum to the third order elastic constants is significant. A general treatment was also given of the thermodynamic properties of crystals containing defects.

Lazarus grew single crystals of helium at pressures as high as 1000 atmospheres and obtained preliminary measurements of the thermal conductivity.

Lattice constant and thermal expansion measurements were made on ^{20}Ne and ^{22}Ne crystals by Simmons. The inadequacies of current theories of these crystals were made obvious.

A study of the spin-flip conduction electron scattering from impurities in liquid metals was made by Slichter.

Thermal conductivity measurements on titanium carbide by Williams suggest that the carbon atoms are arranged on the carbon sublattice in a manner similar to that observed in the glassy state.

Koehler's radiation damage group established that interstitial atoms in gold are mobile at 130°C . Stage II annealing was absent in very pure specimens of electron irradiated gold and all damage annealed during Stage III. The mechanism of Stage III annealing was found to be the migration of vacancies to interstitial clusters. It appears that these clusters are platelets on (111) planes which nucleate at impurities.

PROJECT TITLES AND PRINCIPAL INVESTIGATORS

05-06-01-02-0 Structure of Materials

Strain Energy in Martensitic Transformation

Carl J. Altstetter, Sc.D., Associate Professor of Physical Metallurgy

Electronic Specific Heat of Alloys of Transition Metals with
Nontransition Elements

Paul A. Beck, M.S., M.E., Professor of Physical Metallurgy

Point Defect - Dislocation Interactions

Howard K. Birnbaum, Ph.D., Professor of Physical Metallurgy

Glass and Crystalline Oxide Semiconductors

A. L. Friedberg, Ph.D., Professor of Ceramic Engineering; Head
of Department of Ceramic Engineering

W. D. Compton, Ph.D., Professor of Physics; Director of Coordinated
Science Laboratory

Mechanical and Surface Behavior of Crystals

John J. Gilman, Ph.D., Professor of Physics and Physical Metallurgy

First Order Phase Transformations in Crystalline Solids

David S. Lieberman, Ph.D., Professor of Physical Metallurgy

Dislocations and Surface Barriers--Corrosion at Lattice Defects

Marvin Metzger, Ph.D., Professor of Physical Metallurgy

Annealing of Cold-Worked Metals

Bernard G. Ricketts, Ph.D., Professor of Metallurgical Engineering

Nuclear Magnetic Resonance Studies

Theodore J. Rowland, Ph.D., Professor of Physical Metallurgy

Thin Films and Solid State Phase Transformations

C. Marvin Wayman, Ph.D., Professor of Metallurgical Engineering

The Study of the Nature of Solid Solutions of Metals

Charles A. Wert, Ph.D., Professor of Physical Metallurgy; Head,
Department of Mining, Metallurgy, and Petroleum Engineering

05-06-02-02-0 Solid State Physics, Crystal Physics

Use of Very High Pressure to Study the Structure of Matter

Harry G. Drickamer, Ph.D., Professor of Chemical Engineering and
Physical Chemistry; Member, Center for Advanced Study

Anharmonic Effects in Solids

Andrew V. Granato, Ph.D., Professor of Physics

Defect and Electronic Properties of Solids

David Lazarus, Ph.D., Professor of Physics

Properties of Noble Gas Crystals

Ralph O. Simmons, Ph.D., Professor of Physics

Magnetic Resonance in Solids

Charles P. Slichter, Ph.D., Professor of Physics

Physics of Refractory Materials

Wendell S. Williams, Ph.D., Associate Professor of Ceramic
Engineering and of Physics

05-06-02-03-0 Energetic Particle Interaction

Radiation Damage

James S. Koehler, Ph.D., Professor of Physics

AEC RESEARCH PROJECTS

05-06-01-02-0 Structure of MaterialsStrain Energy in Martensitic Transformation

Principal Investigator: Carl J. Altstetter, Sc.D., Associate Professor
of Physical Metallurgy

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Carl J. Altstetter, Associate Professor

Junior Staff: Daniel R. Hennessy, Research Assistant
Moinuddin S. Rashid, Research Assistant
Howard Savage, Research Assistant

Object: The objective of this project is to determine the mechanism by which saturated interstitial solid solutions decompose and the thermodynamic driving force for the decomposition. Attention will be focussed on oxygen and nitrogen in the bcc refractory metals of group Va (V, Nb, Ta), but with particular emphasis on vanadium. Several studies, both here and elsewhere, have outlined fairly well the similarities, but also unexplained differences between the behavior of Nb and Ta solid solutions. Surprisingly little is known about the interstitial solutions with vanadium--neither the equilibrium with the gas phase, the oxides or nitrides, nor the morphology and kinetics of formation of precipitates. It is thought desirable to learn more about vanadium in order to compare its behavior with that of its close relatives, and thus to better understand them all.

Initially, techniques will be developed to remove impurities from vanadium and to redope it with controlled quantities of solute. In this part of the work the partial pressure-temperature-composition relationships will be measured. This will allow calculation of the enthalpy of solution of the gas, the enthalpy of formation of the compounds, and the entropy of the solute. At a later stage in the program, when specimens may be reliably doped with gas, alloys with interstitial solutes will be made and precipitation of solute will be studied using electrical resistivity, metallography, and x-ray diffraction.

Vanadium has been chosen for study not only because little is known about its interactions with atmospheric contaminants, but also because it is a material having great potential for technological application. Its high melting point, good corrosion resistance and desirable nuclear properties make it promising as a nuclear reactor material. Furthermore, its low

density and cryogenic properties offer possibilities for application in space. However, just as for the other refractory metals, contamination by atmospheric gases, with concomitant degradation of properties, and oxidation remain unsolved problems. An understanding of the absorption of gases and their precipitation as oxides, nitrides or mixed compounds is basic to the further development of vanadium as a technologically important material.

Results, FY68: The thermodynamic study of the fcc-hcp transformation in pure cobalt was completed. Calorimetric calibration runs using the melting of pure tin were quite gratifying. The value for the enthalpy of melting of pure tin was reproduced, with only a few percent scatter in measured values. Calorimetric results for lanthanum were obtained and will be combined with metallographic, dilatometric and x-ray diffraction results to produce the first comprehensive study of the fcc to hexagonal transformation in lanthanum. The work on the x-ray study of defect distribution after transformation in Co-Ni crystals has been the construction of a suitable x-ray collimation and monochromatization system. Theoretical calculations were made to predict the effect of different types of stacking faults on the diffraction spot profiles in single crystals of both fcc and hcp materials. Computer programs for analysis of profiles have been written.

Results Expected, FY69: (a) The study of martensitic transformations in lanthanum will be brought to a close. (b) The problems in preparing single crystals in the form of spheres of hcp Co-Ni alloys will be solved, and the theoretical analysis of diffraction from crystals containing two kinds of stacking faults will be virtually completed. The recording and analysis of the x-ray diffraction data for martensitically transformed crystals remains to be done. (c) The study of discontinuous precipitation in Nb-N alloys will be brought very nearly to completion. A system for measurement of resistivity changes during precipitation has been tested for temperatures up to 800°C. Metallographic analysis will be straightforward and will be carried out after the general time-temperature characteristics have been determined by resistance measurements. (d) The electron microprobe techniques for analysis of precipitation in the Nb-N and Nb-Zr-N systems are just being established. The experimental problems and the correction factors for an element as light as nitrogen are many and difficult.

Results Expected, FY70: The objective of this research is to elucidate mechanisms through which solid state structural changes occur. Two types of transformation will be studied: a) martensitic, in which atoms move cooperatively, giving rise to macroscopic shears, and b) diffusional, in which the more random motion of atoms can accomplish compositional as well as structural changes.

The arrangement of dislocations which propagates the fcc-hcp martensitic transformation is not known, but the thermodynamic driving force and the energy of the debris of defects resulting from transformation has already

been measured in this program for pure cobalt. The current study of stacking fault density and distribution in single crystals of Co-Ni alloys will be completed. This work will be the analysis of the x-ray diffraction spot intensity profile data and the obtaining of supplementary electron microscopic evidence. These results will be used to refine our knowledge of this relatively simple martensitic transformation and should allow conclusions about the probable mechanism.

The work on the kinetics, morphology and composition of nitride and oxide precipitation in Nb will be completed. The analysis of results and theoretical work on the mechanism of diffusional transformation and product morphology in relation to the transformation rate will be carried on. The work on vanadium interstitial solid solutions will be begun.

Other Work: E. Gebhardt and coworkers at the Max Planck Institute in Stuttgart are doing similar work, but they are studying interstitial solute interactions in ternary alloys of Nb and Ta with carbon and nitrogen and oxygen. The principal investigator plans to spend several months in Stuttgart doing research on refractory metal-gas systems, learning new techniques and presenting some of the work done at the University of Illinois. There is no known duplication of effort in the area of proposed research.

Publications, Calendar Year 1967:

E. de Lamotte, Y. C. Huang, and C. Altstetter
Precipitation of Nitride in Niobium (Columbium) and Niobium-Zirconium Alloys
Trans. Met. Soc. of AIME 239, 1625-1630 (1967)
COO-1198-463

Also supported by the Air Force Materials Laboratory

R. Adams and C. J. Altstetter
Thermodynamics of the Cobalt Transformation
Trans. Met. Soc. of AIME (submitted to)
COO-1198-458

M.S. Theses, Calendar Year 1967:

None

Ph.D. Theses, Calendar Year 1967:

None

Electronic Specific Heat of Alloys of Transition Metals with Nontransition Elements

Principal Investigator: Paul A. Beck, M.S., M.E., Professor of Physical Metallurgy

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Paul A. Beck, Professor
Helmut Claus, Research Assistant Professor

Junior Staff: Dhruvajyoti Chakrabarti, Research Assistant
Eric James Hayes, Research Assistant

Object: Transition metals and their alloys are by far the most important structural materials and the only ones available for high temperature applications. Although the unique technological properties are largely the consequence of the high cohesive energy which is, in turn, a result of the electronic structure, a good understanding of the band structure of the transition metals is still lacking. In fact, it was only during the last decade that systematic work started in this field.

Closely connected with the understanding of the electronic structure of the transition metals are several as yet unsolved theoretical problems in the focus of current interest. Some of these problems are: What are the physical conditions for the occurrence (or absence) of magnetic moments in the alloys of 3d transition metals? What is the magnitude of the effect of the electron-phonon and of the electron-magnon (or paramagnon) interactions on the effective mass of the electrons in transition metals and the magnitude of the corresponding enhancement of the electronic specific heat?

Since measurement of the low temperature specific heat provides one of the most important experimental approaches to information on the band structure of metals and alloys, the second question mentioned above is of very great and direct concern to the experimentalist as well as the theorist. In order to evaluate the usefulness of electronic specific heat measurements in obtaining information on the band structure and in order to increase its usefulness by seeking appropriate correction for the magnon-paramagnon enhancement effect, the proposed research is aimed at a detailed experimental study of this effect in alloy systems in which one of the components is ferromagnetic, while the other component is not. In such systems, the coefficient γ of the temperature-linear term of the low temperature specific heat often has a maximum near the composition at which the alloys change from ferromagnetic to paramagnetic. It is proposed to study the various mechanisms which may possibly contribute to the measured γ values in this composition range, including various

types of magnetic contributions, as well as enhancement by electron-magnon interaction. In order to evaluate the presence or absence of magnetic contributions to the low temperature specific heat, it is proposed to extend the temperature range of the specific heat measurements up to about 30°K, to study the effect on the low temperature specific heat of cooling in a magnetic field, of cold working, of heat treatments and of alloying with aluminum. Furthermore, it is planned to make also a detailed study of the effect of the above variables on the magnetic properties of the alloys in question from 4°K to room temperature, and to make electrical resistivity measurements in the same temperature range. Equipment for the magnetic measurements, as well as for low temperature specific heat measurements with specimens cooled in a field of approximately 60 kG is in the process of being installed.

In addition to the work on paramagnon enhancement, it is proposed to study in detail the effect of atomic microenvironment on the occurrence and the magnitude of localized moments on 3d transition metal atoms in alloys with other metals. Information of this type (at present very sketchy) is of fundamental importance in understanding magnetism and the electronic structure of transition metals.

Results, FY68: Work was started on the preparation of hcp alloys in the Re-Co solid solution series and on low temperature specific heat measurements with these alloys. The very considerable metallurgical difficulties encountered in preparing homogeneous alloys in this system were overcome in the composition range up to 60 at.% Re. Initial measurements indicated an unusually large peak of γ at around 30 at.% Re. Low temperature specific heat measurements were made with three hcp solid solution alloys in the Fe-Ru system. The results showed considerable variation of γ with composition, and the possibility of a maximum at around 40 at.% Ru. Additional alloys will be necessary to clarify this point. Since in this system there is no indication of magnetism, the maximum of γ , if it does exist, must be of an origin different from that discussed above.

Apparatus was completed for the precise measurement of electrical resistivity as a function of temperature in the range from 78°K to 300°K. Resistivity measurements were made with several bcc alloys of Cr-Al solid solutions. The electronic structure of these alloys is of particular interest because of their unusually low electronic specific heat values, previously observed.

Results Expected, FY69: An investigation of the magnetic susceptibility vs. temperature for the alloy VAu_4 in the ordered and in the disordered state has been completed and a paper describing this work was published. The low temperature specific heat of disordered VAu_4 has been measured and the effect of the degree of long range order on the low temperature specific heat in this alloy will be studied. An investigation will be

started to study the effect on the magnetic properties of substituting Ti for various fractions of the Cr content in CrAu_4 and the effect of varying degrees of long range order on these properties. It is expected that the low temperature specific heat measurements in hcp Re-Co alloys as a function of composition will be largely completed. Low temperature specific heat measurements after field cooling and magnetization measurements both with and without field cooling will be started. The low temperature specific heat vs. composition data for Ni-Cu and Ni-Cu-Al alloys will be probably completed during the period in question. Low temperature specific heat measurements with such alloys after field cooling will be started.

Results Expected, FY70: It is expected that all the low temperature specific heat and magnetic measurements outlined above, aiming to establish the relative importance of the various possible contributions to the γ -peaks observed in alloys of compositions near the disappearance of ferromagnetism, will be essentially completed. During the same period the cryostatic equipment now being designed for specific heat measurements up to 30°K will be probably put into operation. It is hoped that the study of the effect of atomic micro-environment on the magnetic moment in VAu_4 and in $(\text{Ti-Cr})\text{Au}_4$ can be also completed.

Other Work: Theoretical work on the paramagnon enhancement of the low temperature specific heat is currently being done by Professor J. R. Schrieffer and his associates at the University of Pennsylvania, by Professor S. Doniach at Imperial College, London, and by Professor K. Bennemann at the University of Rochester (and probably by a number of other theoretical physicists as well). On a recent visit to the University of Pennsylvania, at the invitation of Professor Schrieffer, I gave him and his associates a detailed account of our experimental results in this field. Professor Bennemann will visit our group in March 1968 in order to inform himself of our results. Effective exchange of information has been established at least with these two theoretical groups. Experimental work in this field has been done at the Bell Telephone Laboratories at Murray Hill, New Jersey, on Rh-Ni alloys. As far as I am now aware, there is no duplication. The work at Bell Labs appears to be concerned with systems other than those investigated in our laboratory.

Publications, Calendar Year 1967:

T. M. Srinivasan, H. Claus, R. Viswanathan, P. A. Beck, and D. I. Bardos
Electronic Structure of Alloys of 3d Transition Metals with Aluminum.
Phase Stability of Metals and Alloys (McGraw-Hill Publishing Company,
New York, 1967), pp. 151-163.

COO-1198-359

Also supported by the National Science Foundation and by the Advanced Research Projects Agency, Contract SD-131

T. M. Srinivasan and H. Claus

Low Temperature Specific Heat of B.C.C. Alloys of 3d Transition Elements with 20% Al

J. Phys. Chem. Solids 28, 711-716 (1967)

Supported by the Advanced Research Projects Agency, the National Science Foundation, and by the U. S. Army Research Office (Durham)

R. W. Spor, H. Claus, and P. A. Beck

X-Ray Diffraction Study of Atomic Ordering in Two Sigma Phases

Advances in X-Ray Analysis (Plenum Press, N.Y., 1967), Vol. X, p. 213

COO-1198-385

Also supported by the U. S. Army Research Office (Durham) and by the National Science Foundation

H. Claus, A. K. Sinha, and Paul A. Beck

Atomic Environment Effect on the Magnetic Properties of VAu_4

Physics Letters 26A, 38-41 (1967)

COO-1198-493

Also supported by the National Science Foundation

D. I. Bardos, A. T. Aldred, and P. A. Beck

The Effect of Aluminum on the Saturation Moments of Fe-Ni Alloys

J. Appl. Phys. 38, 1260-1262 (1967)

Also supported by AEC at the Argonne National Laboratory

M. S. Theses, Calendar Year 1967:

None

Ph.D. Theses, Calendar Year 1967:

None

Point Defect - Dislocation Interactions

Principal Investigator: Howard K. Birnbaum, Ph.D., Professor of
Physical Metallurgy

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Howard K. Birnbaum, Professor

Junior Staff: Craig Baker, Research Assistant
Melvin F. Bluem, Research Assistant
David M. File, Research Assistant
David Franklin, Research Assistant
Bidyut Kumar Ganguly, Research Assistant

Object: The principal aim of the project is to develop an understanding of the behavior of point defects such as vacancies, interstitials and solute atoms in solids. Of particular interest is the interaction of these defects with dislocations and the role of these interactions in determining the mechanical properties of solids. In addition, the interactions between point defects are of interest. A knowledge of these interactions is necessary for an understanding of the mechanical properties of metals. These interactions have been extensively studied in fcc metals but have hardly been examined in the technologically more important bcc metals. The project will continue some of its investigations in the fcc metals but the major effort will be directed toward the bcc metals.

The use of internal friction techniques will be continued. The investigation of the stress induced motion of divacancies in quenched gold will continue with measurements of the energies of rotation, long range migration and binding of the divacancy. An attempt will be made to determine the orientation dependence of the divacancy damping peak to establish the symmetry of the divacancies; i.e., whether they are first or further nearest neighbor vacancy pairs. The internal friction technique, since it is specific toward point defects of low symmetry, enables us to separate the behavior of single and divacancies in a way which cannot be done using the previously applied resistivity measurements.

The theory of thermally activated dislocation breakaway which has been developed will be applied to various experimentally observed phenomena such as the Bordoni, Hasiguiti and cold work damping peaks in an attempt to test the range of applicability of the theory. The present extension of the damping theory points up the significance of thermal activation in experiments which have been previously interpreted using theories which apply only at 0°K.

The damping of high purity and carefully doped Nb will be determined in the frequency range of 10 Kc to 200 mc and in the temperature range of 4°K to 300°K. The role of impurity pinning points and interactions with

the lattice on the motion of dislocations will be investigated. These same problems are being investigated, using low temperature creep measurements in the same temperature range, and an attempt will be made to correlate the internal friction and creep results. The understanding of the factors which control dislocation motion in bcc metals is necessary before the range of usefulness of these metals can be extended.

As a preliminary to studying the effect of hydrogen on the mechanical properties of Nb, the diffusion behavior of hydrogen in Nb will be investigated. The jump time of hydrogen will be determined over a wide temperature range to look for evidence of clustering or quantum-mechanical tunneling. A complete investigation of the behavior of hydrogen at low temperatures has never been carried out, although it is clearly required in view of the large and deleterious effects of hydrogen on the low temperature properties of bcc metals.

The investigation of the mechanism of diffusion along dislocations will be continued with experiments designed to determine the role of solute ionic size and valence difference on the dislocation diffusivity.

Results, FY68: The theory of damping due to thermally activated dislocation breakaway has been completed. The calculated results suggest that the theory can be applied to a number phenomena such as the Bordoni peaks.

The internal friction peak due to divacancy motion has been observed to occur at about 10°C at 10 cps and to have an activation energy of about 0.7 eV. This relaxation effect which has long been sought was obtained in the absence of deformation using a new quenching and internal friction technique.

The dislocation diffusion experiments in Ni-Co were completed and showed that the stacking faults between partial dislocations are effective enhanced diffusion paths. The ratio of diffusion activation energies $Q_{\text{pipe}}/Q_{\text{lattice}}$ was about 0.45 for the extended dislocations as compared to about 0.6 for the perfect dislocations in pure Ni.

The work on bcc materials has progressed to the stage where most of the required equipment and techniques have been developed. High purity Nb single crystals have been grown and are being prepared for measurement.

Results Expected, FY69: It is expected that the dislocation diffusion experiments which are designed to determine the effects of solute size and valence difference will be completed and partially analyzed. These experiments are designed to establish the mechanism of diffusion along dislocations.

The frequency dependence of dislocation damping in Nb will have been measured in an attempt to establish whether the "vibrating string model"

applies to bcc metals. The damping changes during yielding of Nb will have been measured in the high frequency limit to establish the changes in dislocation density during yielding.

Results Expected, FY70: The attempt to understand the behavior of point defects and their interactions with dislocations will continue. The emphasis of the work will be shifted from work on the fcc metals to studies of the bcc metals such as Nb, Ta, Mo, and W. The experimental techniques used will be primarily internal friction, resistivity and low temperature deformation studies. Since the success of these experiments rests largely on the ability to prepare high purity, well-characterized single crystals, a major effort will be made in this direction. Measurements of the dislocation damping in the temperature range of 4-300°K on high purity and carefully doped Nb will be continued to determine the nature of the interactions which control the motion of dislocations. This problem will also be under continued investigation using high strain sensitivity, low temperature creep measurements on Nb.

The internal friction measurements of the diffusion behavior of hydrogen in the Nb will continue. It is expected that a careful study of D versus temperature will establish whether tunneling or clustering of H atoms will occur.

The divacancy studies in quenched gold should be completed. It is planned to extend these studies (in combination with resistivity measurements) to quenching studies in bcc metals such as Mo and W.

A series of measurements of diffusion along dislocations in bcc metals will be started. The first experiments will probably involve interstitial diffusion in Fe.

Other Work: Work designed to answer many of the same questions as those being approached in the present project is underway at Oak Ridge, Argonne, Cornell University, Oxford University and Harwell AERE. The techniques being applied at Illinois are different but complementary to those being used at these other institutions. The Illinois group is in contact with and is exchanging results with the other groups.

Publications, Calendar Year 1967:

C. R. Heiple and H. K. Birnbaum
Frequency Dependence of Dislocation Damping in Single-Crystal Copper
J. Appl. Phys. 38, 3294-3302 (1967)
COO-1198-401

H. B. Aaron, M. Wuttig, and H. K. Birnbaum
Zener Disaccommodation in Dilute FCC Co-Ti Alloys
Acta Met. (submitted to)
COO-1198-438
Also supported by Wright-Patterson Air Force Base.

C. Baker, H. Birnbaum, and M. Wuttig
 Diffusion Along Edge Dislocations in Ni-Co Alloys; The Effect of
 Stacking Fault Energy
 Journal of the Institute of Metals of Japan (submitted to)
 COO-1198-477

P. Peguin and H. K. Birnbaum
 A Theory of Internal Friction Due to Thermomechanical Unpinning of
 Dislocations
 J. Appl. Phys. (submitted to)
 COO-1198-483

D. C. Loebach, P. Bowden, and H. K. Birnbaum
 The Effect of Argon Ion Irradiation on the Internal Friction of Gold
 Acta Met. (submitted to)
 COO-1198-484

M. Wuttig, H. K. Birnbaum, and J. T. Stanley
 Interstitial Solute Trapping in Irradiated and Quenched Iron
 Acta Met. (submitted to)
 COO-1198-391

M. S. Theses, Calendar Year 1967:

C. C. Baker (H. K. Birnbaum, Adviser)
 Dislocation Pipe Diffusion in Nickel-Cobalt
 June 1967

D. C. Loebach (H. K. Birnbaum, Adviser)
 The Effect of A^+ Bombardment on the Internal Friction of Gold
 February 1967

Ph.D. Theses, Calendar Year 1967:

H. B. Aaron (H. K. Birnbaum, Adviser)
 Zener Disaccommodation in Dilute FCC Alloys
 February 1967

C. R. Heiple (H. K. Birnbaum, Adviser)
 The Frequency Dependence of Dislocation Damping in Single Crystal Copper
 February 1967

Glass and Crystalline Oxide Semiconductors

Principal Investigators: Arthur L. Friedberg, Ph.D., Professor of Ceramic Engineering, Head of Department of Ceramics;
W. Dale Compton, Ph.D., Professor of Physics,
Director of Coordinated Science Laboratory

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: W. Dale Compton, Professor of Physics
Arthur L. Friedberg, Professor of Ceramics

Junior Staff: Gordon W. Anderson, Research Assistant, Physics

Object: The objectives of this program are to examine the conduction mechanisms in semiconducting glasses and to test the applicability of existing theories for the movement of electrons in transition metal compounds.

The experimental approach has been to determine electrical conductivity and Seebeck coefficient in semiconducting glasses in the vanadium-phosphorous-oxygen system. Structural character of the glass was examined by transmission electron microscopy.

Results, FY68: The data from electrical conductivity and Seebeck coefficient measurements indicate that the mechanism of electrical conduction in these transition metal oxide glasses can be treated as a thermally activated diffusion process. Structural examinations from electron microscopy studies indicate the semiconducting glass is a phase-separated system.

Results Expected, FY69: This research program is being terminated.

Publications, Calendar Year 1967:

Gordon Wood Anderson and Fred U. Luehrs, Jr.
Structural Characterization of and Phase Separation in Vanadate Glasses
J. Appl. Phys. (submitted to)
COO-1198-471

Also supported by the Advanced Research Projects Agency, Contract SD-131

Gary S. Snow
Thermally Activated Diffusion of Electronic Carriers in Iron Phosphate
J. Electrochemical Soc. (submitted to)
COO-1198-467

M. S. Theses, Calendar Year 1967:

W. D. Long (G. S. Snow, Adviser)
Interdiffusion Between MgO and Mg_2TiO_4
August 1967

C. D. Reynolds, Jr. (G. S. Snow, Adviser)
Electrical Properties of Vanadium Phosphate Semiconducting Glass
August 1967

Ph.D. Theses, Calendar Year 1967:

None

Mechanical and Surface Behavior of Crystals

Principal Investigator: John J. Gilman, Ph.D., Professor of Physics
and Physical Metallurgy

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: John J. Gilman, Professor
Michael S. Wald, Research Associate

Junior Staff: Alvin Phillip Gerk, Research Assistant
William W. Heminghouse, Research Assistant
Hiroyuki Kaga, Research Assistant
Richard Stanton, Research Assistant
Hua-Ching Tong, Research Assistant

Object: To develop an understanding of the response of crystals to stresses of various kinds including small elastic stresses, pulses that produce plastic flow; stresses induced by electric fields, by energetic incident atomic-particles; and by adsorbed surface layers.

Growth of unusual crystals whose behavior may elucidate the relation between mechanical response and electronic structure. Field-ion microscopy for directly revealing atomic defects. Theoretical analysis. Optical property measurements. Measurements of mechanical wave propagation to obtain second- and third-order elastic constants; and to study electron-phonon interactions.

All means for transforming energy into useful work in ancient as well as modern activities depend on the abilities of solids to resist stresses without deforming excessively or breaking. The future technical results of this project cannot be predicted, but in the past work of this specific kind has led to: a) a major advance in materials for the jet engines used in supersonic aircraft; b) techniques for analyzing the response of metals to impact such as that occurring within and external to nuclear weapon systems; c) predictions for the design of nuclear reactor materials; d) design methods for high-temperature creep systems; e) improved understanding of rock mechanics by geophysicists; f) quantitative methods for specifying the properties of woods by foresters. Recent achievements have included: a) the invention of a successful phenomenological theory of plastic deformation; b) the development of successful methods for directly measuring the surface energies of solids; c) the establishment of a small but growing branch of mechanics known as dislocation dynamics which will have many future applications.

Results, FY68: Construction of laser for growth of refractory crystals. Complete measurements and interpretation of the effect of plastic deformation on the color of the purple compound, gold-aluminide (AuAl_2).

High-resolution field-ion microscope used to study metastable surface atoms. Ion-gun constructed for field-ion microscope. Third-order elastic constants of calcite measured. Continue surface studies of WC, TiC, and VC with low energy electron diffraction.

Results Expected, FY69: Measure elastic constants of tungsten carbide. Complete studies of ion-bombardment with field-ion microscope. Use laser to grow refractory crystals. Complete studies of twinning mechanism in calcite. Observe hypersonic attenuation in fluorite and the effect of electronic excitations on it. Complete comparative studies of surface structures on various carbides.

Results Expected, FY70: Perfect new crystal growth method using vapor decomposition with a laser heat source. Make direct measurements of the ranges of high-velocity incident ions in tungsten. Continue studies of electron damage and its annealing using a field-ion microscope. Make detailed optical reflectivity studies of metals and compounds and relate these to mechanical behavior. Measure adsorption induced changes in the surfaces of crystals using low-energy-electron diffraction. Improve theories of dislocation mobility and fracture at low temperatures.

Publications, Calendar Year 1967:

John J. Gilman

Microdynamical Theory of Plasticity

Advances in Materials Science II; Microplasticity (John Wiley & Sons, New York, 196), Vol. II (to be published)

Supported by the U. S. Atomic Energy Commission, Contract AT(11-1)-1198

M. S. Theses, Calendar Year 1967:

H. C. Shih (J. J. Gilman, Adviser)

Color Changes in the Compound AuAl_2 Induced by Imperfections
June 1967

Ph.D. Theses, Calendar Year 1967:

None

First Order Phase Transformations in Crystalline Solids

Principal Investigator: David S. Lieberman, Ph.D., Professor of
Physical Metallurgy

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: David S. Lieberman, Professor

Junior Staff: Bijoy Kishore Das, Research Assistant
Robert Karz, Graduate Student
Michael A. Schmerling, Research Assistant

Object: To study the morphology, kinetics, crystallography, and internal structure of martensitic and quasi martensitic phase transformations in order to better understand the mechanism operating in each case. This understanding will aid in relating structure and properties and hopefully in producing materials with specific desirable characteristics. In particular, it is intended to extend the recent application of the crystal geometric approach which permitted the probable mechanisms to be differentiated in two transformations under very different thermal conditions in the same intermetallic compound where the product internal structures were identical and the morphologies essentially the same. Another application which will be made is in the transformation in oxides and other nonmetallic systems where the energetics can be better understood using information obtainable from crystallographic analysis. The importance of the onset of some significant physical property as ferroelectricity in the titanates and the metal to nonmetal transition in the vanadium oxides in determining the mode of transformation will be investigated.

Resistivity, magnetic susceptibility, hot stage microscopy, and x-ray diffraction techniques are and will be used to determine the nature of the transformation in intermetallic compounds and nonmetallic systems and how it depends on thermal history, purity, etc. Analytical and graphical methods plus computer programs will be used to analyze the transitions. These approaches are being applied to studies of transformations in the actinides elsewhere and this work is being watched and correlated.

Results, FY68: Work completed and orally reported on the application of the Lieberman-Bullough theory of composite martensite as refined by Lieberman to habits in steels between '225' and '259' using the successfully developed computer program and CALCOMP. Connection between increase of beta prime to beta start temperature and a stabilization process in the beta prime phase has been definitely established and the activation energy determined. Latent heats also measured. Preliminary studies of transformations in the NbRu and TaRu systems completed and a sharp variation of the transition region with temperature observed; transformations seem to be without hysteresis and hence allotropic rather than polymorphic. A sequence of produce phases of decreasing symmetry observed on cooling. Some aspects of TiNi transformation work completed.

Results Expected, FY69: Precision electrical resistivity measurements made on the annealing and stabilizing process taking place in 49 at.% Cd AuCd alloys in the low temperature product phase. Results agree completely with the earlier alteration of A_s with Holding time in the product studies recently completed. Activation energy of about one-half the self-diffusion energy measured for the process which is consistent with E_m for vacancies. X-ray studies of the transformation in NbRu and TaRu begun. Wechsler-Lieberman-Read theory employed to distinguish possible transformation types in quenched and slowly cooled intermetallic compounds.

Results Expected, FY70: It is hoped that the approaches described above will permit first and second order transformations to be distinguished and their mechanisms ascertained. In particular, it is hoped to exploit the semiconducting and insulating titanates and the difference in their transformation behavior to this end. The metal to non-metal transition in oxides will also be studied. Earlier work will be reexamined to determine in more detail the nature of the dependence of morphology, structure, crystallography and kinetics on thermal history, applied fields and stresses, and doping where appropriate. The Ru alloys will be studied in detail with x-rays to determine the order of the transformation and hopefully its mechanism. Some very interesting magnetic changes will also be studied in these alloys. Application of the crystal geometric theory to other transformations will be expanded. Similarities and differences in transformations in organic solids will be determined.

Other Work: No work on the intermetallic compounds being studied here is going on elsewhere although other materials are being investigated and cognizance is maintained with this work as far as possible. Some work on KTN and other titanates being done elsewhere; complementary supplementary studies being carried out here and at Bell Labs.

Publications, Calendar Year 1967:

D. Gupta, D. Lazarus, and D. S. Lieberman
Self-Diffusion in Near Equiatomic Ordered Au-Cd Alloys
Phys. Rev. 153, 863-872 (1967)
COO-1198-369

Also supported by the U. S. Air Force Office of Scientific Research

M. S. Theses, Calendar Year 1967:

B. K. Das (D. S. Lieberman, Adviser)
Some Aspects of Phase Transformation in Near-Equiatomic Niobium-Ruthenium Alloys
August 1967

M. A. Schmerling (D. S. Lieberman, Adviser)
Studies of the Martensitic Phase Transition in Tantalum-Ruthenium Alloys
August 1967

Ph.D. Theses, Calendar Year 1967:

None

Dislocations and Surface Barriers - Corrosion at Lattice Defects

Principal Investigator: Marvin Metzger, Ph.D., Professor of
Physical Metallurgy

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Marvin Metzger, Professor

Junior Staff: Krishan Kumar Chawla, Research Assistant
José Antonio Isasi-Díaz, Research Assistant (Term. 2/10/68)
James Lankford, Jr., Research Assistant
Gregory V. Scarich, Research Assistant

Dislocations and Surface Barriers

Object: To gain an understanding of the mechanical behavior of coated and composite crystalline materials, which involves hardening mechanisms of softer substrate or matrix, mode of deformation of coating or insert and the mechanisms of interaction. The importance lies in the need to understand and control the properties of coated, clad and composite technological materials. The main lines of attack are: a) Mechanical micro-strain measurements supplemented by etch-pit observations to evaluate models of matrix strain hardening and interaction, and b) surface observations by optical and electron microscopy, etching, X-rays to investigate behavior of coating and of surface layers of substrate.

Results, FY68: (a) Completion of work on coated zinc crystals revealed some unusual strengthening and embrittling effects resulting from the formation of a hardened layer beneath the coating, the details of which have not yet been identified. Different effects produced by obstacles to primary dislocations in screw and edge orientation were attributable in part to differences in their mean free path.

(b) The cracking of anodic oxide films on aluminum as a function of thickness, external pressure and surface roughness generally followed the predictions of earlier models based on detachment and interphase slip and certain elaborations of the model were proposed.

Results Expected, FY69: (a) Completion is expected of the first phase of a microstrain study of orientation dependent work hardening in copper crystals at constant dislocation density and structure which aims to determine which secondary slip systems are influential and in what ways. This phase covers a series of crystals with axes in a [111] zone and the results indicate very little influence at small strains of the magnitudes of the resolved stresses on conjugate and critical systems, the observed differences being almost entirely in the extent of easy glide. These crystals had a dislocation density too high for effective supplementary etch-pit study.

(b) The formation during deformation of a hardened zone beneath a coating is being investigated in copper crystals which are neutron irradiated under load and sectioned for etching to determine densities and distributions of primary and forest dislocations. We expect to be able to check the hypothesis that secondary slip induced by the obstacle increases forest density at the small strains in easy glide (where this usually changes very little) and to correlate the strengthening with the extent and dislocation densities of the hardened zone. In general our work with coated specimens is directed toward supplementing and completing previous work and writing it up.

Results Expected, FY70: (a) Completion is expected of the second phase of our microstrain study of orientation dependent work hardening, which includes variation of resolved stress on the cross slip system as well as the conjugate and critical. This is being made on crystals of low dislocation density and intensive etch-pit studies are planned to identify the systems active at small strains. Our previous results on copper crystals have appeared to be in accord with the Basinski-Hirsch views of extensive secondary slip activated by primary pile-ups; however, their views are applicable in Stage II and are quite general, and the questions remain for the microregion and easy glide what specific primary-secondary interactions are involved in forming obstacles to primary dislocation motion. This project is expected to complete our direct studies of strain hardening in copper needed to provide the basis for interpreting the behavior of coated and composite specimens with a copper matrix.

(b) Although we do not have plans for work in new directions on zinc, we propose to carry our previous work to a proper conclusion by investigating the mechanisms of the coating strengthening effects, and we expect to have results in this period of an X-ray and etching study of substrate structure in zinc crystals deformed with a coating on the surface containing the slip vector.

(c) A study of fiber composites based on copper being initiated this year is expected to produce substantial results in FY70. One of the central problems in understanding the behavior of fiber composites is the micromechanism of load transfer from a softer plastic matrix to a strong elastic fiber, and we will attempt to prepare specimens with a single crystal copper matrix where dislocation distribution can be observed at small strains and combined with mechanical microstrain measurements. The situation and the experimental techniques are similar in many respects to those involved in the study of coated specimens. We propose with this project to shift our future emphasis to composites.

Corrosion at Lattice Defects

Object: Investigation of metallurgical conditions under which lattice defects influence corrosion. The results aid understanding of certain classes of corrosion and stress-corrosion cracking problems and incidentally

lead to etching techniques useful in studies of metal structure. High purity aluminum single crystals, bicrystals and polycrystalline specimens are exposed to hydrochloric acid, in which structure-sensitive corrosion is displayed. The morphological and quantitative aspects of corrosion are studied mainly by microscopic techniques. The results are likely to have relevance for metals other than aluminum which tend to form films of low conductivity.

Results, FY 68: Studies of the sites of breakdown in the acid of a pre-applied protective oxide film showed the direct effect of lattice defects such as grain boundaries to be very weak and of low level impurity segregations to be weak compared to the effects of fine cracks in the film. Such cracks may be associated with grain boundary shearing or vacancy condensation pits at dislocations, in which case the defects have strong indirect effects.

Results Expected, FY 69:

This work is not being extended except for a few check tests and finishing the preparation of the results for publication.

Similar Work Elsewhere: There is no duplication of other research in these fields.

Publications, Calendar Year 1967:

J. C. Bilello and M. Metzger
Mechanical Hysteresis in Deformed Copper Crystals
J. Appl. Phys. 38, 849-854 (1967)
COO-1198-283

M.S. Theses, Calendar Year 1967:

K. K. Chawla (M. Metzger, Adviser)
Investigation of Film Rupture Mechanisms on Strained Aluminum
February 1967

Ph.D. Theses, Calendar Year 1967:

None

Annealing of Cold-Worked Metals

Principal Investigator: Bernard G. Ricketts, Ph.D., Professor of
Metallurgical Engineering

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Bernard G. Ricketts, Professor

Junior Staff: Amit Kumar Ghosh, Research Assistant
Wei-Tong Shieh, Research Assistant

Object: A better understanding is sought of the factors which determine the structure of plastically deformed and annealed metals, particularly those which determine the texture developed by the annealing of heavily deformed metals. As a part of this study the process of nucleation of recrystallization and the factors which affect stacking fault energy are being investigated. Quantitative x-ray techniques are used to determine the textures of heavily rolled and annealed materials. The dislocation structure is determined by transmission electron microscopy.

Results, FY68: Work has been completed on the effects of fractional percentages of iron on the development of annealing textures in aluminum, and the behavior is now being compared with that of high purity aluminum with high rolling deformations but without recrystallization during rolling. A theoretical investigation has been made of the elastic modulus effect on the interaction of point defects with dislocation ribbons. An experimental check of some of these results in high purity silver is in progress.

Results Expected, FY69: An initial stage of the work on stacking faults should be completed--the interaction of vacancies with dislocation ribbons in high purity silver.

Results Expected, FY70: Texture studies on heavily-deformed high-purity aluminum should be completed. Evaluation of other factors which determine the relative rates of recovery and recrystallization will be undertaken.

Similar Work Elsewhere: No duplication of effort is known.

Publications, Calendar Year 1967:

W. T. Becker and B. G. Ricketts
The Influence of the Impurity Iron on the Annealing Texture of Aluminum
Trans. Met. Soc. AIME (submitted to)
COO-1198-400

L. C. Michels and B. G. Ricketts
Nucleation of Recrystallization in Cold-Worked Aluminum and Nickel
Trans. Met. Soc. AIME (submitted to)
COO-1198-335

W. T. Shieh
Elastic Modulus Effect in the Interaction of Point Defects with
Dislocation Ribbons
J. Appl. Phys. (submitted to)
COO-1198-451

M.S. Theses, Calendar Year 1967:

None

Ph.D. Theses, Calendar Year 1967:

None

Nuclear Magnetic Resonance Studies

Principal Investigator: Theodore J. Rowland, Ph.D., Professor of
Physical Metallurgy

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Theodore J. Rowland, Professor
Laurence D. Graham, Research Associate

Junior Staff: Kamalodin B. Danesh, Research Assistant
James A. Salsgiver, Research Assistant
S. Rajah Sundara, Research Assistant
Cherng-yuan Sun, Research Assistant

Object: Within the framework of the general objective of applying magnetic resonance techniques to the study of metallurgically important phenomena, we are presently investigating intermetallic compounds, diffusion, precipitation, and melting. The compounds V_3Si and V_3Ga have been studied intensively (elsewhere) recently with strong indications of a sharp peak in the electronic density of states at the Fermi surface. To gain information about the nature of the peak, we propose to investigate the pressure dependence of the nuclear resonance parameters in these and related compounds. Our work on the measurement of nuclear relaxation times in alloys at elevated temperatures and the interpretation of these measurements in terms of atomic and spin diffusion promises a better understanding of the diffusion process and also of the "averaging" by diffusion of internal fields. We feel that the averaging of electric field gradients through motional effects is not completely understood and requires further special attention.

In the study of precipitation we will determine through preliminary study whether this method is capable of improving our concept of the microscopic processes taking place during nucleation and growth of a second phase from a solid solution.

By following the absorption profile as a function of temperature for pure thallium in the region from room temperature to about $500^\circ C$, we hope to identify the effects of diffusion and phase changes on the indirect exchange interaction in the three phases which appear, and thus to separate exchange unambiguously from other interactions.

Results, FY68: An experimental and theoretical study of electric field gradients in alloys of copper with small additions (0.1 to 5 at.%) of Pt, Pd, Rh, and Ir was completed. Additional information on the lattice parameters of these solid solutions has since been obtained and will be used in the final calculations.

Measurement of nuclear relaxation times in alloys of aluminum with Zn, Mg, Ag and Ge were completed and preliminary interpretation in terms of the solute and solvent jump rates was begun. It was clearly established that electric quadrupole contributions to the local fields were a major source of relaxation which thus depended strongly on the properties of the solute. The activation energy for self-diffusion in pure aluminum was also determined over a wider and different temperature range than heretofore. A value significantly lower than that of previous workers using other methods was obtained.

Construction of the high pressure nuclear resonance absorption apparatus was continued. Electron paramagnetic resonance studies of alloys of chromium with 1 to 3% iron were completed.

Results Expected, FY69: All results and calculations related to the study of Cu-Pd; Pt, Rh, and Ir alloys will be completed and prepared for publication. Likewise, work on aluminum alloys mentioned above will be summarized and prepared for publication; however, in this case, investigations will continue in order to provide more accurate data on at least one system, to include other alloys, and to carefully determine the proportion of aluminum nuclei actually contributing to the observed signal. The proper local field at high temperature is not certain. An experiment using rapid (pulsed) heating will be attempted in order to separate the motional from the formation energy of vacancies in aluminum.

The copper-beryllium precipitation hardening experiments will be continued and should lead to useful results during this period. Also, the effect of pressure on the Knight shift and other parameters of V_3Si and V_3Ga will be measured. A digital data averaging system has been included in the apparatus for this experiment in order to improve our ability to increase signal-to-noise ratio while making these measurements.

Results Expected, FY70: A much improved understanding of the relation between atomic diffusion and nuclear relaxation in alloys is expected to be achieved by measurements of signal intensities at high temperatures in well characterized aluminum alloys. The intermetallic compound investigation will be extended to other alloys for which one would predict sizable changes in nuclear interaction as a result of the application of hydrostatic pressure. This promises to be a fruitful field for research, especially if the possibilities of quadrupole effects are considered.

Depending somewhat on the outcome of the present work on precipitation, this phenomenon may be examined in other systems, especially those in which the role played by a third element in the alloy is not clear. Additional topics on which we may be able to begin work are a) surface studies of finely divided platinum by means of nuclear resonance, and b) exploratory experiments on the annealing kinetics of cold worked dilute solid solutions of aluminum. The latter would require that we measure the change in resonance parameters (especially intensity and shape) as

a function of annealing temperature: such measurements should be accompanied by measurements of residual resistivity and (perhaps) hardness, for several different alloys. The rate of work hardening and subsequent dislocation annihilation and/or redistribution will depend upon the solute species and concentration.

Similar Work Elsewhere: Among the topics mentioned above, none are closely similar to work being done elsewhere. There is thus no duplication; we have, at times, cooperated with workers elsewhere to the extent of supplying data before publication. There are of course studies of resonance in metals at many institutions; we believe we are aware of their progress and areas of interest through frequent contact and correspondence.

Publications, Calendar Year 1967:

F. Y. Fradin and T. J. Rowland
NMR Measurement of the Diffusion Coefficient of Pure Aluminum
Appl. Phys. Letters 11, 207-209 (1967).
COO-1198-470

M. B. Salamon and F. J. Feigl
Paramagnetic Resonance in Cr-Fe Alloys
J. Phys. Chem. Solids (submitted to)
COO-1198-496

Also supported by the Advanced Research Projects Agency, Contract SD-131

M.S. Theses, Calendar Year 1967:

J. A. Salsgiver (T. J. Rowland, Adviser)
An Analysis of a Gated Beam Tube Nuclear Magnetic Resonance Dispersion
Spectrometer
August 1967

Ph.D. Theses, Calendar Year 1967:

F. Y. Fradin (T. J. Rowland, Adviser)
Nuclear Magnetic Relaxation and Atomic Diffusion in Aluminum Alloys
October 1967

Thin Films and Solid State Phase Transformations

Principal Investigator: C. Marvin Wayman, Ph.D., Professor of
Metallurgical Engineering

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: C. Marvin Wayman, Professor
Yasushige Fukano, Visiting Assistant Professor
Hector Clark, Research Associate

Junior Staff: Thomas P. Darby, Research Assistant
Hassell M. Ledbetter, Research Assistant
Edmund Merriman, Research Assistant
Thomas J. Patrician, Research Assistant
David D. Thornburg, Research Assistant

Object: Experimental information of a fundamental nature on the growth and properties of thin metal films and solid state phase transformations is being sought. In both of these areas technology has advanced considerably, chiefly through empiricism, while fundamentals and theory remain behind. As two cases in point, one can consider the extensiveness of thin film devices and technology, yet the phenomenon of epitaxy or oriented overgrowth is not understood; the hardening of steels is a well-known phenomenon although there is presently no consistent explanation of the relation of hardening to the solid state martensitic transformation which occurs (i.e., substructure, precipitation, etc.).

The present and proposed work consists of the following specific studies: The nucleation of Au thin films on graphite is being studied to determine the development of epitaxial films. Another program is concerned with thermoelectric power of thin films, particularly when films are discontinuous and tunneling effects become important. This is being studied for Au-Ni films, a combination showing high thermoelectric power. Another thin film program utilizing Au films is concerned with atmosphere effects on recrystallization and grain growth. Films are annealed in various atmospheres up to the range of ultra-high vacuum to determine the role of surface energies on the textures developed.

Work is continuing on the (unusual) beta-zeta solid state transformation in equiatomic Ag-Zn in hopes of more completely clarifying the nature of this transformation, which involves a structure change but can be considered to be a special type of ordering reaction. Because of the simplified crystallography of this transformation, it has been possible to carry out some preliminary quantitative work on the effects of stress in nucleation and growth. As an outgrowth of transformation induced plasticity studies, work has begun on superplastic behavior of materials. The preliminary work is concerned with a eutectic Cd-Zn alloy. Another transformation project deals with the crystallography of the martensitic

transformation which occurs in equiatomic Au-Cd. This is a transformation which should "barely happen" according to preliminary crystallographic analysis.

Results, FY68: Progress has been made in establishing conditions for reproducible growth characteristics of certain thin films, and instrumentation for thermoelectric measurements has been devised. Residual gas analysis during thin film growth and annealing is establishing some impurity effect patterns. The effect of stress on the beta-zeta transformation in Ag-Zn is now well established and related to nucleation and growth rates. A computer program has been worked out for analyses of the crystallography of transformations. Apparatus for testing of superplastic materials has been assembled and is being checked out. Preliminary work suggests the eutectic alloy in the Cd-Zn system to be well suited for transmission electron microscopy studies of superplasticity, and it has been verified that this alloy is superplastic.

Results Expected, FY69: It is anticipated that the morphology studies of the zeta phase in Ag-Zn will be completed; techniques for examining small nuclei in films about 10 Å thick in the electron microscope will be established; techniques for growing thin film Au-Ni thermocouples of varying thickness will be established and reproducible emf versus temperature results will be sought; habit planes, orientation relationships, and the macroscopic shape change for the equiatomic Au-Cd transformation will be determined; the superplastic Cd-Zn alloy will be characterized as to strain rate sensitivity; apparatus for the growth of alloy thin films will be set up; a technique for monitoring by residual gas analysis the vapor of evaporated metals (in search of impurities) will be established.

Results Expected, FY70: The experiments as indicated so far concerning the effect of stress on the beta-zeta transformation in Ag-Zn should be completed; an analysis of the experimental crystallographic information for Au-Cd will be made; the superplasticity project should move into the transmission electron microscopy stage; and mechanical properties of superplastic Cd-Zn will be studied when the alloy is in the non-superplastic state; the preparation of alloy thin films will have begun; film thickness effects on thermoelectric power will be evaluated for Au-Ni films; experiments concerned with variables affecting epitaxial growth of alloy thin films will be under way. Studies of the nucleation versus growth controlled epitaxy will continue.

Similar Work Elsewhere: No obvious overlap.

Publications, Calendar Year 1967:

S. Jana and C. M. Wayman
Martensite-to-f.c.c. Reverse Transformation in an Fe-Ni Alloy
Trans. Met. Soc. of AIME 239, 1187 (1967)
COO-1198-434

K. Otsuka and C. M. Wayman
Epitaxial Growth of Vacuum-Evaporated Co on NaCl: I. Conditions
Leading to Epitaxy
phys. stat. sol. 22, 559 (1967)
COO-1198-431

K. Otsuka and C. M. Wayman
Epitaxial Growth of Vacuum-Evaporated Co on NaCl: II. Analysis
of Diffraction Patterns
phys. stat. sol. 22, 579 (1967)
COO-1198-432

J. van Landuyt and C. M. Wayman
On the Habit Planes of the Suboxide Phases TaO_y and TaO_z
J. Less Common Metals (submitted to)
COO-1198-485

G. R. Srinivasan and C. M. Wayman
Isothermal Transformations in an Fe-7.9Cr-1.1C Alloy
Trans. Met. Soc. of AIME (submitted to)
COO-1198-460

G. R. Srinivasan and C. M. Wayman
Transmission Electron Microscope Study of the Bainite Transformation
in Iron-Chromium-Carbon Alloys
Acta Met. (submitted to)
COO-1198-461

G. R. Srinivasan and C. M. Wayman
The Crystallography of the Bainite Transformation
Acta Met. (submitted to)
COO-1198-462

J. van Landuyt and C. M. Wayman
A Study of Oxide Plate Formation in Tantalum I. Growth Characteristics
and Morphology
Acta Met. (submitted to)
COO-1198-487

J. van Landuyt and C. M. Wayman
A Study of Oxide Plate Formation in Tantalum II. Crystallographic
Analysis
Acta Met. (submitted to)
COO-1198-488

M. V. Byelous and C. M. Wayman
Temperature Changes in Thin Metal Films During Vapor Deposition
J. Appl. Phys. 38, 5119-5124 (1967)
COO-1198-494

M. S. Theses, Calendar Year 1967:

A. Chaudhuri (C. M. Wayman, Adviser)

Effect of Stress on the $\beta \rightarrow \zeta$ Transformation in an Equiatomic
Ag-Zn Alloy

February 1967

G. B. Bilow (C. M. Wayman, Adviser)

An Investigation of Thin Silver Films Vapor Deposited on Cleaved
NaCl at Low Temperatures

February 1967

Ph.D. Theses, Calendar Year 1967:

None

The Study of the Nature of Solid Solutions of Metals

Principal Investigator: Charles A. Wert, Ph.D., Professor of Physical Metallurgy; Head, Department of Mining, Metallurgy, and Petroleum Engineering

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Charles A. Wert, Professor
Charles Dollins, Research Associate

Junior Staff: Lawrence J. Gray, Research Assistant
Charles R. Rarey, Research Assistant

Object: Past work on this project has been extremely varied, encompassing (a) nuclear magnetic resonance and electron screening of impurities in alloys, (b) diffusion in transition metals using radiotracers, (c) interstitial solid solutions in refractory metals. Future work is expected to concentrate mainly on item (c) and to be specifically concerned with the following problems (in approximate order of importance).

- a) Investigation of ordered interstitial phases in refractory metals Nb, Ta, and V. Twenty years ago, Zener predicted a martensite-like ordering of interstitial solutions in all bcc metals. In the past few years such ordered structures have been observed by transition electron microscopy and electron diffraction. Apparently a whole series of such structures exist from alloys of only a few atom percent interstitial up to stoichiometric ionic compounds. Good interpretation of these observations has been hampered by lack of knowledge of exact composition. We propose to observe such structures in alloys such as Nb-C and V-C in the electron microscope and to determine local composition using the light element capability of the microprobe.
- b) Determination of phase equilibrium in binary and ternary alloys. This will involve determination of solubilities, structure and morphology of compounds, trapping of interstitials by impurities.
- c) A small study to complete the radiotracer work in vanadium. We need to know the effect of Fe additions on self-diffusion in dilute V-Fe alloys.

Impurities such as C, N and O are very important in determining properties of the transition metals. They influence many properties important in reactor technology such as strength, corrosion by liquid metals, fatigue, creep, embrittlement.

Methods of preparation of pure metals and of doping them with known quantities of impurities are already fairly well established. Structure will be determined by electron microscopy; chemistry by standard chemical methods or by microprobe where feasible; investigation of equilibrium

structures also by electron diffraction and Mossbauer effect (where feasible); internal friction in dilute alloys.

Results, FY68: a) NMR work. Many investigations were made of screening charge distribution in liquid metal alloys. Most striking effects were observed if transition metals were added in small quantity to non-transition hosts.

b) Study of Fe diffusion in V was completed. Isotope effect seems to show that two distinct modes of diffusion are present; one predominating above 1350°C, the other predominating below.

c) Interstitial alloys. Much of the effort was spent in instrumentation. A sinusoidal motion system for the Mossbauer spectrometer was constructed. The light element capability of the microprobe was developed. A correction procedure for calculating chemical composition from x-ray counts was established; a program was written for computer calculations of the analysis.

Results Expected, FY69: a) NMR work on liquid alloys will be completed.

b) Tracer diffusion in V will be completed.

c) Interstitial alloys. Nb-C alloys will be prepared and examined. One can produce solid solution, ordered structures and precipitates by control of composition and heat treatment. Techniques will be developed for transferring thinned specimens containing precipitates of size a few microns into the probe for analysis. Development of the Mossbauer procedures will be continued and research problems defined.

Results Expected, FY70: The study of carbides and ordered structures in the Nb-C alloy system should be completed. Examination will be made using combined electron microscopy, microprobe and electron diffraction procedures in oxides and nitrides of Nb and Ta. Work will begin on vanadium.

The Mossbauer study of decomposition of ordered structures during aging should be completed. Because we know how to prepare such alloys in Fe better than in any other system, we will use three types of Fe-C martensite for this study. Plain Fe-C alloys will be examined (two published papers agree that new lines appear in the spectrum in early stages of martensite decomposition, but do not agree in detail) to deduce the nature of the initial carbides formed during decomposition. Ternary alloys will also be examined, one containing a carbide-former such as Cr, the other a graphite-former such as nickel. Upon completion of this work, we will continue the work by going to ordered alloys of the refractory metals, such as V-C, using small quantities of active iron as a tracer.

Other Work: Many people have done or are doing work on ordered phases in refractory metals using electron microscopy. In general, good metallurgical control of specimen composition and knowledge of specimen chemistry is almost completely lacking. We have developed the capability to do both better.

We are in touch with Paul Shewmon's group at Argonne. We are also in touch with Gebhardt's group at Stuttgart and will be exchanging people on a short-term basis.

Publications, Calendar Year 1967:

D. A. Rigney and C. P. Flynn,
Electronic Structure in Dilute Alloys
Phil. Mag. 15, 1213-1232 (1967)

COO-1198-406

Also supported by the Advanced Research Projects Agency

C. P. Flynn, D. A. Rigney, and J. A. Gardner
Magnetic Polarization of Transitional Impurities in Aluminum
Phil. Mag. 15, 1255-1273 (1967)

COO-1198-408

Also supported by the Advanced Research Projects Agency

M. S. Theses, Calendar Year 1967:

None.

Ph.D. Theses, Calendar Year 1967:

M. C. Coleman (C. A. Wert and D. Lazarus, Advisers)
Isotope Effect for Diffusion of Iron in Vanadium
June 1967

05-06-02-02-0 Solid State Physics, Crystal Physics

Use of Very High Pressure to Investigate the Structure of Matter

Principal Investigator: Harry G. Drickamer, Ph.D., Professor of Chemical Engineering and Physical Chemistry; Member, Center for Advanced Study

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Harry G. Drickamer, Professor
Giovanni DePasquali, Research Assistant Professor
Wilfred B. Holzapfel, Research Associate

Junior Staff: Charles W. Christoe, Research Assistant
Joel A. Cohen, Research Assistant
Daniel L. Fanselow, NDEA Fellow
Donald C. Fisher, Chrysler Corporation Fellow
Curtis W. Frank, NASA Trainee
S. K. Fung, Research Assistant
Dane C. Grenoble, NDEA Fellow
George K. Lewis, Jr., Chrysler Corporation Fellow
Joseph A. Moyzis, Jr., Research Assistant (Term. 1/19/68)

Object: The objectives of our research are to study the electronic and related behavior of solids as a function of very high pressure and so relate the changes of these properties to changes of interatomic distance. One can thus hope to relate the electronic behavior of solids to the overlap of electronic wave functions. The techniques used include optical absorption, electrical resistance, X-ray scattering, and Mössbauer resonance. These techniques are applied over a pressure range of several hundred kilobars.

The importance of this work lies in the following: (a) The addition to our basic understanding of electronic interaction as a function of the overlap of wave functions. (b) The discovery of new phenomena, such as the electronic transitions in alkali metals, semiconductor-metal transitions, etc., reported earlier, or the effect of pressure on the oxidation state of iron reported below.

Results, FY68: The outstanding results in this period involve Mössbauer resonance studies. A detailed study has been made on the effect of pressure on the electronic structure of ferrocene. In α -Fe₂O₃ we have observed the effect of pressure on the magnetic field, isomer shift and quadrupole splitting. The Morin (spin flip) transition occurs at about 25 kilobars at 25°C, probably accompanied by a slight movement of the Fe³⁺ in the unit cell. FeO becomes antiferromagnetic at 25°C at about 50 kilobars. Systematic measurements of the effect of pressure on the isomer shift of Fe⁵⁷ in a series of ferric and ferrous compounds was undertaken.

Results Expected, FY69: The major accomplishment has been the establishment of the fact that ferric iron in a wide variety of compounds reduces to the ferrous state at high pressure, and that this is a reversible phenomenon. We are currently trying to characterize this phenomenon as a function of pressure, temperature, type of ligand, type of bonding, symmetry, etc. This is a lengthy investigation which will carry through next year and probably well beyond that time.

In addition, we have studied the effect of pressure on the isomer shift of Co^{57} (Fe^{57}) in metallic iron in the bcc phase and presented a theoretical analysis of the results. Studies have also been made of Co^{57} in Cu, V, and Ti resulting in both isomer shift and f number data. Again, a rather thorough analysis has been presented. The magnetic field has been measured as a function of pressure in a series of Co-Pd alloys containing 5, 8, 9, 12, 15 atomic percent cobalt. An analysis of the results is being undertaken.

Results Expected, FY70: The major projects to be undertaken are:

- (1) Mössbauer studies of the effect of pressure on the oxidation state of ionic iron.
- (2) Mössbauer studies of the effect of pressure on iron in biologically active materials such as hemin, hematin, and hemochromes.
- (3) Mössbauer studies of magnetism in Pd-Co alloys.
- (4) Mössbauer resonance studies of iron in unusual valence states in carbonyls and related compounds.
- (5) Mössbauer studies of covalent iron in phosphides, arsenides, antimonides, sulfides, and selenides.
- (6) Mössbauer studies of iron as an impurity in oxides.
- (7) The development of a clamp cell to permit experimental studies at liquid helium temperature and 250 kilobars.
- (8) The development of a cell to permit study of samples of 2 to 3 milligrams at 300 kilobars.

Other Work: There is a great deal of very good high pressure work in the hydrostatic range (10-20 kilobars). A few laboratories, (e.g., Bell Labs, Geophysics at U.C.L.A.) work in the range to 150 kilobars. Much of this work is geophysical. There is a minimal overlap with our work from these people. As far as I know, there is no other laboratory in the world making significant studies in the range to several hundred kilobars.

Publications, Calendar Year 1967:

A. R. Champion, R. W. Vaughan, and H. G. Drickamer
Effect of Pressure on the Mössbauer Resonance in Ionic Compounds of Iron
J. Chem. Phys. 47, 2583-2590 (1967)
COO-1198-447

A. R. Champion and H. G. Drickamer
Effect of Pressure on the Mössbauer Resonance in Potassium Ferrocyanide,
Potassium Ferricyanide, and Insoluble Prussian Blue
J. Chem. Phys. 47, 2591-2594 (1967)
COO-1198-448

H. G. Drickamer
Pi Electron Systems at High Pressure
Science 156, 1183-1189 (1967)
COO-1198-426

R. Ingalls
Isomer Shift of Fe^{57} in Iron
Phys. Rev. 155, 157-165 (1967)
COO-1198-388

R. Ingalls, H. G. Drickamer, and G. DePasquali
Isomer Shift of Fe^{57} in Transition Metals under Pressure
Phys. Rev. 155, 165-170 (1967)
COO-1198-389

Also supported by the U. S. Office of Naval Research

R. W. Vaughan and H. G. Drickamer
Effect of Pressure on the Mössbauer Resonance in Ferrocene and Pyrite
J. Chem. Phys. 47, 468-472 (1967)
COO-1198-435

R. W. Vaughan and H. G. Drickamer
High-Pressure Mössbauer Studies on $\alpha\text{-Fe}_2\text{O}_3$, FeTiO_3 , and FeO
J. Chem. Phys. 47, 1530-1536 (1967)
COO-1198-436

A. R. Champion and H. G. Drickamer
The Effect of Pressure on the Mössbauer Resonance in Hemin and Iron
Phthalocyanine
J. Chem. Phys. (submitted to)
COO-1198-449

H. G. Drickamer
The Effect of Interionic Distance on the Ligand Field in NiO
J. Chem. Phys. (submitted to)
COO-1198-452

K. Rama Reddy and R. W. Anderson
A Method to Reduce the Instrumental Line Widths in Mössbauer Spectroscopy
Rev. Sci. Instr. (submitted to)
COO-1198-468

M. S. Theses, Calendar Year 1967:

S. C. Fung (H. G. Drickamer, Adviser)
A High Pressure Low Temperature Mössbauer Apparatus
February 1967

Ph.D. Theses, Calendar Year 1967:

A. R. Champion (H. G. Drickamer, Adviser)
The Mössbauer Effect in Iron Compounds under High Pressure
June 1967

R. W. Vaughan (H. G. Drickamer, Adviser)
High Pressure Mössbauer Studies of Iron Compounds
June 1967

Anharmonic Effects in Solids

Principal Investigator: Andrew V. Granato, Ph.D., Professor of Physics

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Andrew V. Granato, Professor
Akira Ikushima, Research Assistant Professor
Tetsuro Suzuki, Research Assistant Professor

Junior Staff: Edwin R. Fuller, Research Assistant
Jon Holder, Research Assistant
Russell F. Marshall, Research Assistant
Edwin R. Naimon, Research Assistant
Joseph F. Thomas, Research Assistant (Term. 12/31/67)

Object: The objective of the project is the establishment of equations of state for solids, the determination of interatomic potentials, the checking of relations which should exist between various anharmonic effects, and the calculation of defect properties in crystals.

The approach used is the measurement and interpretation of second and third order elastic constants. These are obtained by measurements of sound velocities under both hydrostatic and uniaxial stress. The importance of the method lies in the fact that the data obtained can hardly be obtained by other methods with the same accuracy.

Results, FY68: The third order elastic constants of aluminum have been measured. These cannot be interpreted by neglecting the effects of the conduction electrons, as was the case with the previous materials we have studied. Calculations for the third order elastic constants of simple metals have been developed. The effect of dislocations on the measurements has been studied by taking measurements on NaCl, where the third order constants can be determined both from measurements on elastic modes which are sensitive to dislocations and modes which are not. Results found in both ways give the same results in the stress range used.

Results Expected, FY69: The effect of point defects on the second order elastic constants of LiF and Cu is under study. Measurements of the third order elastic constants of BaF₂ have been made. A calculation of the second and third order elastic constants of simple metals using a simple pseudopotential is nearly complete. A general treatment of the thermodynamic properties of imperfect crystals has been worked out.

Results Expected, FY70: Third order elastic constants already found will be used in an effort to understand better the second order elastic constants of perfect materials. Materials will be studied for which internal deformation parameters are needed to specify atom positions not given by the external strain and a theoretical framework for such materials will be

developed. This will include hexagonal crystals and also III-V and II-IV compounds, for which the degree of covalency and ionicity is important in determining the elastic properties.

A program has been initiated for the study of ultrasonic attenuation and velocity changes in materials undergoing second order phase changes.

Other Work: There is no duplication of work at other laboratories.

Publications, Calendar Year 1967:

Y. Hiki, J. F. Thomas, Jr., and A. V. Granato
Anharmonicity in Noble Metals; Some Thermal Properties
Phys. Rev. 153, 764-771 (1967)
COO-1198-356

K. D. Swartz
Anharmonicity in Sodium Chloride
Journal of the Acoustical Society of America 41, 1083-1092 (1967)
COO-1198-394

D. Gerlich
The Third Order Elastic Moduli of Barium Fluoride
Phys. Rev. (submitted to)
COO-1198-489

M.S. Theses, Calendar Year 1967:

None

Ph.D. Theses, Calendar Year 1967:

None

Defect and Electronic Properties of Solids

Principal Investigator: David Lazarus, Ph.D., Professor of Physics

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: David Lazarus, Professor
James J. Burton, Research Associate
Duk Nong Yoon, Research Associate

Junior Staff: Robert A. Beyerlein, Research Assistant
S. Fain, Research Assistant
Rondo N. Jeffrey, Research Assistant
Willis Mock, Jr., Research Assistant
William A. Spurgeon, Research Assistant
P. Vernes, Research Assistant

Object: Studies are made of the transport and electronic properties of solids, with and without defects, to ascertain the basic physical laws which bear on the strength and stability of materials under a variety of environmental conditions. Particular emphasis is placed on studies of the mechanism for diffusion in solids and on the effects of hydrostatic pressure on a number of basic physical properties. A variety of experimental techniques are used, including methods for generation of high pressure, precision electrical resistivity and thermoelectric power measurements, tracer diffusion studies, magnetic susceptibility techniques, thermal conductivity measurements, and resonance methods.

Results, FY68: Work has been completed on the determination of the effect of pressure on the thermoelectric power of aluminum, gold, nickel, and platinum, and on the electrical resistivity of aluminum and gold; evidence has been found for the existence of strongly bound divacancies in aluminum at high temperatures. Apparatus has been completed for growing single crystals of solid helium at pressures up to 1000 atmospheres, and preliminary values of thermal conductivity have been determined. Work was completed on the measurement of the isotope effect for diffusion of iron in vanadium (in collaboration with C. A. Wert's group), indicating the onset of a new diffusion mechanism at high temperatures. Work was completed (in collaboration with C. P. Flynn's group) on a study of the Knight shifts and susceptibilities of alloys of aluminum with rare-earth metals; evidence indicated the existence of magnetic bound states. Preliminary results have shown that self-diffusion in titanium shows a normal pressure dependence, contradicting previous studies which indicated anomalies. Apparatus was completed to study the effects of large thermal gradients on diffusion in gold and other metals.

Results Expected, FY69: Work will be completed on the study of thermal conductivity in pure crystals of solid helium grown at various pressures over the entire range of the hcp phase. Work will be essentially completed

on a study of the effects of large thermal gradients on self-diffusion and diffusion of selected impurities in gold and nickel. Work will be completed on a study of the effects of pressure on self-diffusion in titanium. New programs will be started on studies of the effects of stress on the Fermi surfaces of alkali metals, of X-ray studies of helium crystals, of annealing of quenched vacancies in aluminum, of ferromagnetic resonance in alloys, and of the effects of pressure on the intrinsic electrical conductivity of alkali halides.

Results Expected, FY70: Work should be nearing completion on most of the new projects currently underway: on the electronic structure of alkali metals, on X-ray studies of helium crystals, on the annealing of quenched vacancies in aluminum, on ferromagnetic resonance, and on ionic conductivity at high pressures. New programs will probably be initiated to study the effects of pressure on second-order phase transitions, generation and motion of vacancies in aluminum and its alloys, and the effects of pressure on the phonon-drag phenomenon in thermoelectricity.

Other Work: None of our work directly duplicates work done elsewhere. Largely through correspondence, meetings, and exchange of preprints, we maintain close contact with other groups studying related problems. In many cases the "other groups" are headed by former students from our group, so close personal relations are easily maintained.

Publications, Calendar Year 1967:

C. P. Flynn, G. W. Stupian, and D. Lazarus
Spin Polarization Near Localized Moments in Metals
Phys. Rev. Letters 19, 572-577 (1967)
COO-1198-454

Also supported by the Advanced Research Projects Agency

D. Gupta, D. Lazarus, and D. S. Lieberman
Self-Diffusion in Near Equiatomic Ordered Au-Cd Alloys
Phys. Rev. 153, 863-872 (1967)
COO-1198-369

Also supported by the U. S. Air Force Office of Scientific Research

R. R. Bourassa, D. Lazarus, and D. A. Blackburn
The Effect of High Pressure on the Thermoelectric Power and Electrical Resistance of Aluminum and Gold
Phys. Rev. (submitted to)
COO-1198-465

W. Seward and R. Rosenbaum
A Simple and Inexpensive Liquid Nitrogen Level Controller
Rev. Sci. Instr. (submitted to)
COO-1198-441

Also supported by the Advanced Research Projects Agency

M.S. Theses, Calendar Year 1967:

None

Ph.D. Theses, Calendar Year 1967:

R. R. Bourassa (D. Lazarus, Adviser)

The Effect of High Pressure on the Thermoelectric Power and Resistance
of Aluminum and Gold

June 1967

M. C. Coleman (C. A. Wert and D. Lazarus, Advisers)

Isotope Effect for Diffusion of Iron in Vanadium

June 1967

G. W. Stupian (D. Lazarus, Adviser)

Magnetic Susceptibility and Knight Shift Measurements in Rare Earth
Aluminum Alloys

June 1967

Properties of Noble Gas Crystals

Principal Investigator: Ralph O. Simmons, Ph.D., Professor of Physics

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Ralph O. Simmons, Professor

Junior Staff: Frederick H. Kabat, Research Assistant
David S. Kupperman, Research Assistant
William E. Schoknecht, Research Assistant
Ronald W. Wilkins, Research Assistant
Richard Windecker, Research Assistant

Object: This project supports study of the lattice dynamics and defect properties of noble gas crystals, which exhibit prominent anharmonic and quantum zero-point effects. These simple fcc crystals serve as a regular family of model substances, upon which critical and relatively independent tests may be made of theories of anharmonic dynamics and of atomic interactions in the condensed state. Because the cohesion of these solids is small and they are highly compressible, anharmonic and strain-dependent effects can be studied over a wide range of conditions, including an extensive portion of the melting line. Experimental techniques employed include the growth and manipulation of massive single crystals, high-precision x-ray lattice parameter and diffracted x-ray intensity measurements versus pressure and versus temperature, precise bulk volumetric change determinations, and elastic wave velocity measurements by ultrasonic diffraction methods.

Results, FY68: During this period our direct measurements of thermal vacancy content of krypton were carried out and analyzed. The vacancy enthalpy is only two thirds of the lattice potential energy per atom. This indicates that the lattice and electronic relaxation are much more severe around the vacancy than previously thought, or that many-body forces contribute significantly to the cohesion of krypton, or both. Next, extensive measurements (for the first time) of the bulk modulus of krypton over the whole temperature range permitted a critical analysis of equation-of-state work on condensed gases. Existing theory is adequate at 0°K, if sufficient account of anharmonicity is made, but theories are generally inadequate at finite temperature. A complete thermal properties table for solid krypton was prepared, over the entire temperature range at low pressure. Its detailed interpretation in model-independent lattice dynamical terms, including the effects of thermal defects, was carried out. Krypton is the first crystal of any kind for which an extensive and reliable analysis is possible over the entire temperature range.

Results Expected, FY69: X-ray lattice-constant and thermal expansion data taken earlier on this project on single crystals at ^{20}Ne and ^{22}Ne have been analyzed. Isotopic differences at 0°K are represented best by a Born-van Karman anharmonic model with only nearest-neighbor interactions. On the other hand, isotopic differences at finite temperatures are represented best by anharmonic cell models. Neither of these models represent other thermal property changes with better than 20 to 50% accuracy and neither employs a realistic interatomic potential, however. The large effects exhibited by neon have therefore made plain the inadequacies of current theories of these effects. Next, analysis of the thermal properties of argon, krypton, and xenon is being carried out upon best current data for the family as judged by critical tests for internal consistency. Contributions to scalar properties by anharmonicity are represented by a single volume-dependent parameter, whose values are to be compared with several specific models. Finally, velocities of sound waves in CaF_2 crystals have been determined through the diffraction of laser light. The apparatus will be further proved at low temperature by sound velocity measurements in liquid krypton and neon, for which no measurements are available, but for which they are required in the interpretation of electron and hole mobility measurements being carried out in England.

Results Expected, FY70: Direct measurement of thermal vacancy content in neon crystals should be completed. Strong lattice anharmonicity and large quantum zero-point effects are expected to affect the nature and extent of the defect structure in qualitatively few ways.

Continuation of the program to measure single-crystal elastic stiffness of noble-gas crystals by means of ultrasonic pulse-echo and He-Ne laser light scattering is proposed. No reliable data appear in the literature. Results would give evidence primarily about short-range interatomic interactions.

Measurements, by x-ray diffraction, of Debye-Waller factor and of elastic stiffnesses will be attempted in special apparatus now under construction. Information about several negative moments of the lattice-wave spectrum should also be obtained, to complement neutron scattering work elsewhere.

Design and construction of an apparatus for equation-of-state studies to relative volume changes of 20% should be completed. Accuracy is expected to be about two orders of magnitude greater than for existing methods, so that meaningful tests of recent theories can be carried out.

Eventual cooperation with the University of Illinois Coordinated Science Laboratory is envisaged, leading to far-infrared (10 to 1000μ) absorption measurements of defect-induced polarization. Fourier-transform spectroscopy of very large specimens is required. For neutral defects the results should yield direct lattice wave spectra, with some additional local mode evidence.

Other Work: Theoretical and experimental work on noble gas crystals in the national laboratories and in U. S. universities complements the present project but in no way duplicates any portion of it. Experimenters in this rather difficult area, including the scientists of this project, communicate with one another often and frankly, which promotes continued cooperation and makes maximum use of available resources.

This project's elastic stiffness measurements are in direct competition with current work being attempted in England, Belgium, and Germany. To date no group has produced satisfactory results.

Publications, Calendar Year 1967:

D. N. Batchelder, D. L. Losee, and R. O. Simmons
X-Ray Diffraction Study of Ne, Ar, and Kr Crystal Growth
Crystal Growth, Supplement to the Journal of Physics and Chemistry of Solids, edited by N. S. Peiser (Pergamon Press, New York, 1967),
pp. 843-847
COO-1198-337

D. N. Batchelder, D. L. Losee, and R. O. Simmons
Measurements of Lattice Constant, Thermal Expansion and Isothermal
Compressibility of Neon Single Crystals
Phys. Rev. 162, 767-775 (1967)
COO-1198-444

D. L. Losee and R. O. Simmons
Vacancy Concentrations Measurements and Many-Body Force Effects in Krypton
Crystals
Phys. Rev. Letters 18, 451-453 (1967)
COO-1198-428

A. O. Urvas, D. L. Losee, and R. O. Simmons
The Compressibility of Krypton, Argon, and Other Noble Gas Solids
J. Phys. Chem. Solids 28, 2269-2281 (1967)
COO-1198-433

M. S. Theses, Calendar Year 1967:

None

Ph.D. Theses, Calendar Year 1967:

D. L. Losee (R. O. Simmons, Adviser)
Equilibrium Vacancy Concentration Measurements and Thermal Properties
of Solid Krypton
June 1967

A. O. Urvas (R. O. Simmons, Adviser)
The Compressibility of Krypton, Argon, and Other Noble Gas Solids
August 1967
Also supported by the U. S. Department of State Studentship

Magnetic Resonance in Solids

Principal Investigator: Charles P. Slichter, Ph.D., Professor of Physics

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Charles P. Slichter, Professor
Howard A. Blackstead, Research Associate
Dankward Schmid, Research Associate

Junior Staff: E. Kevin Cornell, MMM Fellow
George Fredericks, Research Assistant
Edwin A. S. Lewis, Research Assistant
Paul Schwartz, Research Assistant
Paul R. Spencer, Research Assistant
Lawrence R. Whalley, Research Assistant

Object: One of the most active areas of modern theoretical physics is so-called "many body theory" in which one attempts to understand phenomena involving many strongly interacting particles. Two examples are the response of a dense electron gas to a test electric charge (leading to the phenomenon of screening) and the behavior of ordered systems near a second-order phase transition. Three of the four items listed below are of this sort. The first two items concern electric screening and the fourth is concerned with second-order phase transitions. Our third item is concerned with a further understanding of a technique of nuclear resonance which greatly enhances one's ability to detect weak nuclear resonances. We are applying it to the study of structural defects which occur in extremely low concentration.

(1) Measurement of the spin-flip scattering cross section of conduction electrons from various atoms. Dilute alloys are made of various elements dissolved in Li or Na. We study the width of the conduction electron resonance as a function of impurity concentration, deducing the desired cross section from the data. The cross section gives information about the scattering phase shifts and thus the screening.

(2) Study of electric field gradients near impurity atoms in metals. The quadrupole coupling of Al atoms near to impurities is measured by three methods--two using metal powders, the third single crystals. The quadrupole splittings measure the electric field gradients produced by the foreign atom. They can be compared with theoretical predictions.

(3) Investigate the sensitivity of nuclear double resonance. NaCl crystals are doped with various impurities. The quadrupole splitting of atoms near a Cl is observed by double resonance. The signal strength versus impurity concentration is studied.

(4) Temperature change of the order parameter in second-order phase transitions near the transition point in Gd metal and NH_4Cl . Magnetization versus temperature for Gd is measured. Length versus temperature and piezoelectric constant is studied for single crystals of NH_4Cl .

Results, FY68:

- (1) Resonances were seen in liquid metals for which impurities are soluble which were not in our previous studies below the melting point.
- (2) Quadrupole transitions were observed in Al samples containing Ag, Cu, Ge, Mg as impurities, thus extending our previous results on Al containing Zn.
- (3) Double resonance was observed of several impurities in NaCl.
- (4) Single crystal samples were prepared of Gd and NH_4Cl . Preliminary length versus temperature measurements were made on NH_4Cl .

Results Expected, FY69:

- (1) We have measured the scattering cross section of Bi in Na, a crucial test from the theoretical viewpoint. We hope to complete measurements of Sb, Pt and Pd also.
- (2) We have found quadrupole lines in single crystals on Al-Mg. By varying orientation of the magnetic field relative to the crystal, we can identify the crystallographic site of the nucleus observed. A technique for producing single crystal samples of doped metals is under development. It is being applied to Al containing Mg.
- (3) Substantial improvements in apparatus stability and signal-to-noise have improved double-resonance detection sensitivity. We are searching for new lines in the NaCl-K and NaCl-Ag systems.
- (4) Experiments on length versus temperature and piezoelectric constant versus temperature are in progress for NH_4Cl . Experiments on magnetization versus temperature for Gd continue.

Results Expected, FY70: Conduction Electrons: Our previous experiments gave anomalous results for the valence dependence of the spin-flip scattering cross section of conduction electrons from impurities in Na and Li metals. Some elements which do not form solid solutions with the alkali metals do form liquid alloys. We will extend our measurements to molten alkali to study these impurities.

Structure of nonmagnetic point imperfections: Our work in Al with Zn, Ag, and Mg impurities has led us to the observation of quadrupole couplings of Al nuclei near to the impurities. Very recently we have

observed the splitting in single crystals of Al containing Mg. This experiment, never previously done, gives us finally a means of identifying the crystallographic location of the nucleus relative to the impurity, much as is done in the INDOR experiments in insulating crystals. We shall actively push this work in Al and other host metals. We shall continue double nuclear resonance in insulating crystals.

Phase Transitions: We expect to continue work on the scaling laws of second-order transitions, including the ferromagnetic transition in Gd and the order-disorder transition in NH_4Cl and ND_4Cl .

Other Work: Although there is work being done in this general area, we are not duplicating work being done elsewhere.

Publications, Calendar Year 1967:

M. Satoh, P. R. Spencer, and C. P. Slichter
Nuclear Quadrupole Spectra in NaCl Crystals Containing Br^- Impurities by Nuclear Double Resonance
J. Phys. Soc. Japan 22, 666 (1967)
COO-1198-405

J. H. Pifer
Magnetic Resonance of Mn^{++} in PbS , PbSe and PbTe
Phys. Rev. 157, 272-276 (1967)
COO-1198-407

J. H. Pifer
Modulation and Cross Relaxation Effects on the Lineshape of Strongly Saturated Nuclear Magnetic Resonance in Solids
Phys. Rev. (submitted to)
COO-1198-464

N. Fernelius and C. P. Slichter
A Magnetic Resonance Study of the Screening of Electrical Fields in Metals Using Zinc Impurities in Aluminum
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M.S. Theses, Calendar Year 1967:

None

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None

Physics of Refractory Materials

Principal Investigator: Wendell S. Williams, Ph.D., Associate Professor
of Ceramic Engineering and of Physics

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Wendell S. Williams, Associate Professor
Kurt Bachmann, Research Associate
Lee George Radosevich, Research Associate

Junior Staff: David L. Kohlstedt, Research Assistant

Object: The object of this program is to improve our physical understanding of the behavior of certain refractory materials through the generation of experimental data on well characterized samples and the application of current ideas of solid state physics. The materials selected for this investigation are the cubic carbides of the Column IV and V transition metals. These materials have high melting points and great hardness and are hence of interest as structural materials for service under extreme conditions. Some progress has been made in understanding bonding and dislocation behavior in the carbides, and we plan to continue this line of investigation.

Results, FY68: The influence of point defects in high concentrations (10-20% carbon vacancies) on heat transport by phonons and electrons at low temperatures was studied in single crystals of TiC_x . The usual maximum in thermal conductivity found for crystalline solids between liquid helium temperature and liquid nitrogen temperature was not present. Instead the thermal conductivity was extremely low and increased monotonically with increasing temperature. A theoretical treatment of phonon scattering by point defects was not adequate to rationalize the observations. Rather, the observed behavior is similar to that exhibited by glasses. We suggest that the fcc sublattice of carbon in rocksalt-structure TiC_x can more profitably be regarded as a glassy network rather than a crystalline array with many point defects. However, the fcc sublattice of titanium is relatively defect-free and leads to sharp low-energy electron diffraction patterns also obtained in this investigation.

Single crystals of TiC , ZrC , and NbC were studied with a LEED apparatus. A square net of sharp diffraction spots was obtained from {100} cleavage surfaces in all samples. The crystals were then lightly oxidized at high temperatures so that the initial stages of surface modification could be studied. New diffraction spots appeared and we have proposed models to explain them.

The influence of electric currents and fields on the diffusion of carbon atoms in the cubic carbides at high temperatures is also under investigation.

The result of this study will be a value for the effective charge of the diffusing particle. We hope then to obtain a value for the charge of the carbon atom at a lattice site. Various experimental and theoretical studies suggest that this charge is not zero; if charge separation exists in the compound, some of the binding is ionic. A better understanding of the unusually strong binding in the carbides is part of our purpose.

Results Expected, FY69 and FY70: The low temperature thermal conductivity experiment will be concluded. Many samples of carbides with various concentrations of carbon vacancies will be measured. The new "glassy" model will be examined critically and appropriate theories of heat transport in glassy solids will be applied.

Apparatus for the electromigration experiment will be assembled and measurements made. The redistribution of carbon due to the applied field and current will be studied with the electron probe microanalyzer. A model for the diffusion of carbon in the carbides will be developed. The relation between the effective charge during a diffusion jump and at the lattice site will be analyzed. The theory of electromigration will be improved to include more details of the band structure. Conclusions will be reached about the amount of ionicity in the bonding of TiC.

A new project on the influence of electric fields and currents on bulk dislocation mobility will be launched.

Other Work: A modest amount of fundamental research on the transition metal carbides is in progress elsewhere. We are in touch with these groups: (1) University of Minnesota (Toth): Low temperature specific heat; superconductivity. (2) RIAS (Lye, Venables): Band structure; vacancy ordering. Additional efforts exist in England, France, and Russia and at some of the National laboratories in the U. S. We do not believe that our work duplicates any of the above.

Publications, Calendar Year 1967:

Wendell S. Williams

Field Ion Microscopy of Graphite

J. Appl. Phys. (submitted to)

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None

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None

05-06-02-03-0 Energetic Particle InteractionRadiation Damage

Principal Investigator: James S. Koehler, Ph.D., Professor of Physics

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: James S. Koehler, Professor
James C. North, Research Assistant Professor
James G. Ring, Research Assistant Professor
Choochun Lee, Research Associate (Beginning 2/1/68)
Yoshiharu Shimomura, Research Associate

Junior Staff: Ronald R. Berliner, Research Assistant
L. S. Edelheit, NASA Fellow
Alan Gordon, Research Assistant
William D. Hyatt, Research Assistant
Ronald E. McKeighen, Research Assistant
Charles C. Meek, NSF Trainee

Object: The objectives of our study are to understand the production of point defects during irradiation of metals, alloys, and semiconductors and to find out what atomic processes occur in the various stages of thermal annealing. In addition, we would like information about the nature and properties of the various defects present.

In germanium and silicon the defect migration energies of interstitials and vacancies are not well known and their dependence on the position of the Fermi level is not well known. The location of the defect levels in the band gap is also not well known. The following experiments should clarify these properties.

Results, FY68:

1. The major findings on the stage III annealing of pure electron irradiated gold were:

- (a) The interstitial is mobile at 130°K in gold.
- (b) The damage retained on electron irradiation at 130°K results from the trapping of mobile interstitials at impurities.
- (c) No stage II annealing is observed in highly pure specimens.
- (d) Stage III annealing occurs by vacancy migration to interstitial clusters.

- (e) The activation energy observed $E_{III} = 0.85 \pm .03$ eV is the same as that found in fast quenched specimens.
 - (f) In highly pure specimens essentially all of the damage anneals in stage III. (There is less than 2% of $\Delta\rho$ (130°K) left.)
 - (g) The sinks in an irradiated gold specimen differ from those in a quenched specimen.
2. Valuable electron microscope observations on electron irradiated and quenched gold were completed. The major findings were:
- (a) Small defect clusters exist in electron irradiated gold if it is examined before stage III annealing.
 - (b) In 5 nines pure gold after 6×10^{18} e/cm² (3 MEV), one observes about 10^{16} clusters/cm³. In 6 nines pure gold one observes about 10^{15} clusters/cm³. The clusters are about $30 \text{ \AA} \pm 20 \text{ \AA}$ in size.
 - (c) The strain field observed suggests that they are platelets on the (111) planes.
 - (d) If the irradiated sample is thinned after warming to 100°C for 30 minutes, no clusters are seen.
 - (e) A gold specimen quenched from 900°C to -20°C and then examined below stage III shows no clusters. If, however, such a specimen is annealed at 30°C for 15 days, vacancy tetrahedra are observed.
 - (f) Since the defect clusters are not vacancy tetrahedra and since they are present in an irradiated specimen below the stage III annealing temperature, we believe that they are interstitial clusters produced during irradiation and nucleated at impurities.

Results Expected, FY69:

1. Stage III in Silver. Proton and deuteron irradiation data and quenching plus equilibrium data suggest that in copper and silver $E_{III} < E_M^V$ (i.e., the activation energy associated with stage III is smaller than the vacancy migration energy). To date no data of high quality has been obtained following electron irradiation.

It is possible that oxygen may alter the activation energy observed in stage III. We wish to check this by examining high purity silver which has been carefully annealed in vacuum in an effort to avoid oxygen.

2. Geometry of Interstitials by Anomalous X-ray Transmission. Quantitative measurements of the changes in the anomalous x-ray transmission which occur upon irradiation and annealing of single crystals are being done. At present the counting apparatus can detect changes

greater than 0.6%. Unfortunately we estimate that six days of 3.5 MEV electron irradiation at 1 microampere per cm^2 will be required to give observable changes in copper. Efforts are therefore being made to improve the accuracy to which counting measurements can be made.

If changes of 0.3% can be detected, then the following experiments will be done: Single crystals of copper will be irradiated with 3.5 MEV electrons to a total dose of 5×10^{18} e per cm^2 . This will introduce a fractional defect concentration of 3×10^{-4} . The changes in x-ray transmission resulting in various anomalously transmitted beams will be examined. The (220), (440), (200), and (400) reflections will be measured in crystals 0.1 cm thick. In addition, changes which occur during annealing will be measured.

We hope to determine from this experiment whether the interstitial in copper occupies a cube center position or whether it consists of two atoms with axis along a (100) direction and with the center of the dumbbell at a lattice site.

If this venture succeeds, a similar experiment will be done on gold where the stage I annealing structure differs considerably from that seen in copper and silver.

Present theoretical calculations suggest that the atomic displacements near the interstitial which is a member of a close interstitial vacancy pair do not differ much from those near an isolated interstitial. This prediction will be checked by measuring transmission changes which occur on annealing one of the close pair peaks.

Another experiment is as follows: Dyson, Anthony, and Turnbull [J. Appl. Phys. 37, 2370, 2925 (1966)] have evidence that copper, silver, and gold exist as interstitials in lead. The solubilities of copper and gold in lead are 0.025 at. % (326°C) and 0.08 at. % (200°C). Anomalous transmission experiments made on these dilute alloys would be of value to check this suggestion.

3. Ion Injection. An ion source capable of producing well-collimated beams of ions of any desired kind has been constructed. Focussed beams of 50 KEV copper, gold, and silicon have been produced. The beam intensities are .5 milliamperes for copper and gold and 5 microamperes for silicon and germanium.

These beams will be used as channeling projectiles into single crystals. In the first experiments a given type projectile will be used on a crystal made of the same kind of atom (for example silicon on a silicon crystal). The first experiments will be done on silicon and germanium. The ions will be implanted at as low a temperature as possible (20°K or less), and the subsequent ion distribution will be determined using capacitance measurements. Since the carriers freeze out at low temperature,

it is not certain that measurements of dc/dv versus V (i.e., rate of change of capacitance with applied voltage) can be made much below 40°K . Channeling theory and room temperature experiments suggest that the channeled particles will have a range of some thousands of Angstroms. If the resulting interstitials are mobile at the irradiation temperature, then the penetration should be an order of magnitude larger. We wish to determine whether the interstitials are mobile or not at the lowest temperature that we can measure charge distribution. This will be done in n , p , and intrinsic germanium and silicon. The cryostat, goniometer for aligning the specimen, and the ion source are complete. More sensitive capacitance measurements will probably be required. The activation energies of interstitial migration in semiconductors have not been determined.

We also intend to inject gold ions into gold in an attempt to determine the migration energy associated with the interstitial in gold. Single crystal gold films can be made by epitaxial growth on NaCl . Such crystals can be prepared flat to 0.3° over an area 0.2 cm in diameter. The film has the (100) direction normal to the film and can be $300\text{ \AA}$ to 1 micron thick. Electrical resistance measurements will be made on the 1 micron thick crystal before and after implantation at 10°K . If the interstitials are mainly in the 10^{-5} cm near the surface, then the increase in resistivity after implantation will be small--about 10% of the initial value. If the interstitials migrate at 10°K , then the situation will be more complicated. Some will go to the bombarded surface. Some will produce interstitial clusters and some will be trapped at impurities. We should be able to decide whether the interstitials are mobile at the irradiation temperature from the production curve, i.e., from measurements of $\Delta\rho$ versus integrated incident particle flux. If the production reaches saturation at small integrated flux (i.e., 10^{14} injected ions per cm^2), the interstitials are probably not mobile at 10°K . If, on the other hand, $\Delta\rho$ increases as the square root of the integrated flux for integrated fluxes as large as 10^{16} ions per cm^2 , the interstitials are probably mobile at 10°K . In the latter case annealing at higher temperatures will not alter $\Delta\rho$.

There is another gold implantation experiment which would be valuable. We would like to measure the volume change of the specimen associated with the implantation of a gold interstitial. This can be done by measuring the bending of a gold strip which occurs when gold ions are injected into one face of the strip. If the average penetration depth is t , the thickness of the foil d , its length ℓ_0 , the volume increase for interstitial ΔV_I (in atomic volumes), then the angle θ through which the outer end of the foil rotates is:

$$\theta = \text{radians} \approx \frac{2\ell_0 c_I (\Delta V_I)^{1/3}}{d - t}$$

Thus if $c_I = 10^{-4}$, $\Delta V_I = 1.5$ atomic volumes, $\ell_0 = 1\text{ cm}$, $d = 10^{-2}\text{ cm}$, and $t = 10^{-4}\text{ cm}$, we find $\theta = 2.3 \times 10^{-2}$ radians or about 1.3 degrees. This

can be measured using a Tuckerman strain gauge or using a capacitance measurement.

Results Expected, FY70:

1. Location of Interstitials by Channeling. It has been discovered that well-collimated ion beams will penetrate deep into single crystals if they enter parallel to an open "hole" or channel through the crystal. The energy loss rate dE/dx for the channeled ion can be one-half or one-third that of an ion incident in a random direction.

We would like to use 3 MEV protons or α particles to determine by channeling the nature of the interstitial in irradiated copper and in irradiated gold. One expects that the $\langle 110 \rangle$ channeling should be diminished slightly by the presence of interstitials; the transmission along $\langle 100 \rangle$ will probably not be much altered. However $\langle 112 \rangle$ channeling will be markedly reduced by cube centered interstitials, but will be uninfluenced by the presence of split $\langle 100 \rangle$ interstitials. For a one mil thick copper crystal having $C_I = 10^{-4}$ there are 2.16×10^{16} interstitials per cm^2 of specimen surface. Since there are 3.77×10^{15} $\langle 112 \rangle$ channels per cm^2 of surface for a specimen cut with a $\langle 112 \rangle$ direction normal to the surface, one should expect a measurable change in transmission upon irradiation. A detailed calculation predicts a 0.29% change. This is small because the dechanneling cross section is very small.

2. Interstitial Migration Energies in Germanium and Silicon. Germanium would be irradiated with 3 MEV electrons at 10°K . The density of charged defects introduced into n type, p type and intrinsic germanium would be measured by using the metal semiconductor junction capacitance and Schottky's equation, i.e.,

$$N(x) = \frac{2}{q\epsilon A^2} \frac{dv}{d(1/C^2)}$$

In this case, if no defects migrate at the irradiation temperature, we expect $N(x)$ to be constant. If the interstitial is mobile at the irradiation temperature or at the lowest temperature that one can make measurements, then $N(x)$ should be a function of x since interstitials can disappear at the bombarded surface. We believe that capacitance measurements can be made down to 20°K . Moreover, if we measure the shape of $N(x)$ versus X we should be able to determine the interstitial diffusion constant at the irradiation temperature if the interstitials migrate at that temperature. If the irradiation temperature is below the temperature at which the defects migrate, but the temperature of measurement is above the migration temperature, then interesting transients should be seen.

3. The Electron Microscopy of Point Defects. The present resolving power of the electron microscope is better than $5\text{\AA}$. If one is exceedingly careful, one can resolve the germanium or the gold lattice. However, one would

prefer to see point defects but without the crystal lattice. We believe that we can avoid observing the lattice. Such observations will be tried when we receive our high resolution pole piece for the Hitachi electron microscope.

Other Work: We do not know of any other group working on the same experiments. There are some groups attempting to investigate the same problems, but the experiments proposed here differ from theirs. For example: Watkins at the General Electric Laboratory uses electron spin resonance to determine the migration energies of defects in silicon. MacKay and Klontz at Purdue University measure conductivity and Hall coefficient in silicon and germanium. We believe that several independent measurements, all of which fit one interpretation, are required to prove an interpretation.

Publications, Calendar Year 1967:

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Phys. Rev. 155, 553-562 (1967)
COO-1198-393

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The Diffusion of the Solvent and the Solute in Dilute Alloys of Face-Centered-Cubic Metals
J. Phys. Chem. Solids 28, 317-323 (1967)
COO-1198-381

R. K. Sharma, C. Lee, and J. S. Koehler
Vacancy Annealing in Three Different Experiments in Gold
Phys. Rev. Letters 19, 1379-1381 (1967)
COO-1198-472

Also supported by the U. S. Army Research Office (Durham)

Y. N. Lwin, M. Doyama, and J. S. Koehler
Stage III Annealing Study of Electron-Irradiated Pure Aluminum
Proceedings of the International Conference on Solid State Physics Research with Accelerators, Allen N. Goland, editor (Physics Department, Brookhaven National Laboratory, Upton, New York 11973, to be published); also
Phys. Rev. (submitted to)
COO-1198-499

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M. S. Theses, Calendar Year 1967:

None.

Ph.D. Theses, Calendar Year 1967:

None

AEC PERSONNEL

Senior Staff

Carl J. Altstetter, Associate Professor, Department of Mining, Metallurgy and Petroleum Engineering
Kurt Bachmann, Research Associate, Department of Physics
Paul A. Beck, Professor, Department of Mining, Metallurgy and Petroleum Engineering
Howard K. Birnbaum, Professor, Department of Mining, Metallurgy and Petroleum Engineering
Howard A. Blackstead, Research Associate, Department of Physics
James J. Burton, Research Associate, Department of Physics
Hector Clark, Research Associate, Department of Mining, Metallurgy and Petroleum Engineering
Helmut Claus, Research Assistant Professor, Department of Mining, Metallurgy and Petroleum Engineering
W. Dale Compton, Professor, Department of Physics; Director of Coordinate Science Laboratory
Giovanni DePasquali, Research Assistant Professor, Department of Physics
Charles Dollins, Research Associate, Department of Mining, Metallurgy and Petroleum Engineering
Harry G. Drickamer, Professor, Department of Chemistry and Chemical Engineering; Member, Center for Advanced Study
Arthur L. Friedberg, Professor, Department of Ceramics; Head of Department
Yasushige Fukano, Visiting Assistant Professor, Department of Mining, Metallurgy and Petroleum Engineering
John J. Gilman, Professor, Department of Mining, Metallurgy and Petroleum Engineering and Department of Physics
Laurence D. Graham, Research Associate, Department of Mining, Metallurgy and Petroleum Engineering
Andrew V. Granato, Professor, Department of Physics
Wilfred B. Holzapfel, Research Associate, Department of Physics
Akira Ikushima, Research Assistant Professor, Department of Physics
James S. Koehler, Professor, Department of Physics
David Lazarus, Professor, Department of Physics
Choochon Lee, Research Associate, Department of Physics
David S. Lieberman, Professor, Department of Mining, Metallurgy and Petroleum Engineering
Robert J. Maurer, Professor, Department of Physics; Director of Materials Research Laboratory
Marvin Metzger, Professor, Department of Mining, Metallurgy and Petroleum Engineering
James C. North, Research Assistant Professor, Department of Physics
Lee George Radosevich, Research Associate, Department of Physics
Bernard G. Ricketts, Professor, Department of Mining, Metallurgy and Petroleum Engineering
James G. Ring, Research Assistant Professor, Department of Physics
Theodore J. Rowland, Professor, Department of Mining, Metallurgy and Petroleum Engineering

George A. Russell, Professor, Department of Physics; Associate Director of Materials Research Laboratory
 Dankward Schmid, Research Associate, Department of Physics
 Yoshiharu Shimomura, Research Associate, Department of Physics
 Ralph O. Simmons, Professor, Department of Physics
 Charles P. Slichter, Professor, Department of Physics
 Tetsuro Suzuki, Research Assistant Professor, Department of Physics
 Michael S. Wald, Research Associate, Department of Mining, Metallurgy and Petroleum Engineering
 C. Marvin Wayman, Professor, Department of Mining, Metallurgy and Petroleum Engineering
 Charles A. Wert, Professor, Department of Mining, Metallurgy and Petroleum Engineering; Head of Department
 Wendell S. Williams, Associate Professor, Department of Physics and Ceramic Engineering
 Duk Nong Yoon, Research Associate, Department of Physics

Junior Staff

Gordon W. Anderson, Research Assistant, Department of Physics
 Craig Baker, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
 Ronald R. Berliner, Research Assistant, Department of Physics
 Robert A. Beyerlein, Research Assistant, Department of Physics
 Melvin F. Blum, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
 Dhruvajyoti Chakrabarti, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
 Krishan Kumar Chawla, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
 Charles W. Christoe, Research Assistant, Department of Physics
 Joel A. Cohen, Research Assistant, Department of Physics
 E. Kevin Cornell, MMM Fellow, Department of Physics
 Kamalodin B. Danesh, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
 Thomas P. Darby, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
 Bijoy K. Das, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
 Lewis S. Edelheit, National Aeronautics and Space Administration Fellow, Department of Physics
 Samuel C. Fain, Research Assistant, Department of Physics
 Daniel L. Fanselow, National Defense Education Act Fellow, Department of Chemical Engineering
 David M. File, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering

- Donald C. Fisher, Chrysler Corporation Fellow, Department of Chemical Engineering
- Curtis W. Frank, National Aeronautics and Space Administration Trainee, Department of Chemical Engineering
- David Franklin, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Edwin R. Fuller, Research Assistant, Department of Physics
- Shun Chong Fung, Research Assistant, Department of Chemistry and Chemical Engineering
- George Fredericks, Research Assistant, Department of Physics
- Bidyut Kumar Ganguly, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Alvin P. Gerk, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Amit K. Ghosh, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Alan Gordon, Research Assistant, Department of Physics
- Lawrence J. Gray, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Dane C. Grenoble, National Defense Education Act Fellow, Department of Chemical Engineering
- Eric J. Hayes, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- William W. Heminghaus, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Daniel R. Hennessy, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Jon Holder, Research Assistant, Department of Physics
- William D. Hyatt, Research Assistant, Department of Physics
- Rondo N. Jeffrey, Research Assistant, Department of Physics
- Frederick H. Kabat, Research Assistant, Department of Physics
- Hiroyuki Kaga, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Robert Karz, Graduate Student, Department of Mining, Metallurgy and Petroleum Engineering
- David L. Kohlstedt, Research Assistant, Department of Physics
- David S. Kupperman, Research Assistant, Department of Physics
- James Lankford, Jr. Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Hassell M. Ledbetter, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering
- Edwin A. S. Lewis, Research Assistant, Department of Physics
- George K. Lewis, Jr., Chrysler Corporation Fellow, Department of Chemical Engineering
- Russell F. Marshall, Research Assistant, Department of Physics
- Ronald E. McKeighen, Research Assistant, Department of Physics
- Charles C. Meek, National Science Foundation Trainee, Department of Nuclear Engineering
- Edmund Merriman, Research Assistant, Department of Mining, Metallurgy and Petroleum Engineering

Willis Mock, Jr., Research Assistant, Department of Physics
 Edwin R. Naimon, Research Assistant, Department of Physics
 Thomas J. Patrician, Research Assistant, Department of Mining, Metallurgy
 and Petroleum Engineering
 Charles R. Rarey, Research Assistant, Department of Mining, Metallurgy
 and Petroleum Engineering
 Moinuddin S. Rashid, Research Assistant, Department of Mining, Metallurgy
 and Petroleum Engineering
 James A. Salsgiver, Research Assistant, Department of Mining, Metallurgy
 and Petroleum Engineering
 Howard Savage, Research Assistant, Department of Mining, Metallurgy and
 Petroleum Engineering
 Gregory V. Scarich, Research Assistant, Department of Mining, Metallurgy
 and Petroleum Engineering
 Michael A. Schmerling, Research Assistant, Department of Mining, Metallurgy
 and Petroleum Engineering
 William E. Schoknecht, Research Assistant, Department of Physics
 Paul Schwartz, Research Assistant, Department of Physics
 Wei-Tong Shieh, Research Assistant, Department of Mining, Metallurgy and
 Petroleum Engineering
 Paul R. Spencer, Research Assistant, Department of Physics
 William A. Spurgeon, Research Assistant, Department of Physics
 Richard Stanton, Research Assistant, Department of Mining, Metallurgy and
 Petroleum Engineering
 Cherng-yuan Sun, Research Assistant, Department of Mining, Metallurgy and
 Petroleum Engineering
 S. Rajah Sundara, Research Assistant, Department of Mining, Metallurgy and
 Petroleum Engineering
 David D. Thornburg, Research Assistant, Department of Mining, Metallurgy
 and Petroleum Engineering
 Hua-Ching Tong, Research Assistant, Department of Mining, Metallurgy and
 Petroleum Engineering
 Patrice Vernes, Research Assistant, Department of Physics
 Lawrence R. Whalley, Research Assistant, Department of Physics
 Ronald W. Wilkins, Research Assistant, Department of Physics
 Richard Windecker, Research Assistant, Department of Physics

Professional Staff

Donald L. Connelly, M.S., Research Physicist
 Marjorie Duggan, M.S., Research Chemist
 Judith A. Eakin, B.A., Assistant Research Chemist
 Danold W. Golightly, Ph.D., Research Chemist
 Fred U. Luehrs, Jr., Assistant Research Electron Microscopist
 Victor G. Mossotti, Ph.D., Senior Research Chemist
 William C. Phillips, Mass Spectroscopist
 Willard A. Schooley, Research Engineer
 Amy J. Unertl, B.S., Assistant Research Chemist

PUBLICATIONS

(Calendar Year 1967)

05-06-01-02-0 Structure of Materials

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