MASTER

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The Science of Materials
Contract AT(11-1)-1198

Submitted to the
U.S. ATOMIC ENERGY COMMISSION

by the
MATERIALS RESEARCH LABORATORY
UNIVERSITY OF ILLINOIS
URBANA, ILLINOIS

April 15, 1971

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Materials Research Laboratory

University of Illinois

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INTRODUCTION

The Materials Research Laboratory of the University of Illinois was established in June 1962 as an interdepartmental and interdisciplinary laboratory of the College of Engineering. The following departments of the University participate in its operation:

- Department of Ceramic Engineering
- Department of Chemical Engineering
- Department of Chemistry
- Department of Electrical Engineering
- Department of Geology
- Department of Metallurgy and Mining Engineering
- Department of Physics

The administration of the Laboratory is the responsibility of an Administrative Staff and a Steering Committee that is composed of one member of each of the participating departments. The Administrative Staff of the Laboratory is:

Robert J. Maurer, Professor of Physics, Director
John T. Stanley, Business Manager

The Steering Committee of the Laboratory is:

- J. Bardeen, Professor of Electrical Engineering and of Physics
- H. G. Drickamer, Professor of Chemical Engineering and of Physical Chemistry
- A. L. Friedberg, Professor of Ceramic Engineering; Head, Department of Ceramic Engineering
- R. O. Simmons, Professor of Physics; Head, Department of Physics
- C. A. Wert, Professor of Metallurgical Engineering; Head, Department of Metallurgy and Mining Engineering
- R. J. Maurer, Professor of Physics (ex-officio)

The operation of the Laboratory is assisted by the committees and facilities listed below:

Health and Safety

F. Wise
C. Evans
S. Brown

Machine Shop (Rm. 144, 146, 148)

D. Mapother
F. Wise

Analytical Laboratory (Rm. 320, 331, 355, 384, 387, 388)

C. Evans

Key Punch, Calculator, Typewriter (Rm. 212)

D. Ginsberg
J. Stanley

Reading Room (Rm. 208)

R. Maurer

Seminar (Rm. 280)

R. Maurer

Electron Microscope (Rm. 62, 66)

H. Birnbaum
M. Metzger

Autotrol (Rm. 212)

P. Handler

Crystal Growing Laboratory (Rm. 414)

H. Birnbaum

Materials Test Laboratory (Rm. 413)

C. Altstetter

X-Ray Laboratory (Rm. 369)

C. Altstetter
T. Rowland
G. Stucky

Metallographic Laboratory (Rm. 362)

C. Altstetter
C. Wert

Microprobe Laboratory (Rm. 58, 66)

C. Wert
J. Woodhouse

High Temperature Laboratory (Rm. 448, 452)

S. Brown

Toxic Materials Laboratory (Rm. 454)

S. Brown

Radiochemistry Laboratory (Rm. 222, 28A)

C. Evans
G. De Pasquali

Accelerator Laboratory (Rm. 12)

J. Koehler

Infrared Laboratory (Rm. 231)

M. Klein
F. Brown

Spectrophotometric Laboratory (Rm. 317)

F. Brown

Cryogenic Laboratory (Rm. 9, 29)

D. Mapother

Sigma 5 Computer (Rm. 241)

R. Marshall
V. Metze
R. Maurer
C. Duke
H. Stapleton
G. Metze
W. Hardy
F. Stahl

Scanning Electron Microscope Laboratory
(Rm. 56)

C. Wert
J. Woodhouse

Raman Laboratory (Rm. 226)

M. Klein

Four new facilities were obtained with ARPA funds during the past year. These were a Scanning Electron Microscope (JSM-U3), a Materials Test Apparatus (810 Material Test System), a Raman apparatus (Spex 1401 double monochromator, high power lasers), and a Xerox Data Systems Sigma 5 computer with 53,000 words of memory, 50 megabytes of disk storage, magnetic and paper tape drives and a Calcomp graphical recorder. Twelve teletype terminals provide time sharing service. Mr. Russell Marshall is manager of the computer operation and is assisted by Mrs. V. Metze, Research Assistant, Coordinated Science Laboratory. An Advisory Committee consists of C. Duke, H. Stapleton, G. Metze, W. Hardy, and F. Stahl.

Also, a 125 KG superconducting magnet and a 70 KG uniform field (10 ppm) superconducting magnet were added to the Laboratory facilities.

Certain of the facilities have supporting staffs of which the largest is that of the Analytical Laboratory and Radiochemistry Laboratory. These laboratories, which are under the supervision of Dr. C. A. Evans, provide chemical analytical services to the projects of the Materials Research Laboratory. The staff of the Analytical and Radiochemistry Laboratories is as follows:

- C. A. Evans, Ph.D., Senior Research Chemist
- F. N. Abercrombie, Ph.D., Research Chemist
- J. F. Wolcott, M.S., Research Chemist
- J. Eakin, B.A., Assistant Research Chemist
- A. Unertl, B.S., Assistant Research Chemist
- W. Phillips, Mass Spectroscopist

These laboratories are supported by the ARPA Contract HC 15-67-C-0221.

The Electron Microscope Laboratory, which is also supported by the ARPA contract, is staffed by Mr. R. Anderson, Electron Microscopist.

The Electron Microprobe Laboratory and the Scanning Electron Microscope Laboratory which are supported by the AEC contract, is staffed by Mr. John Woodhouse, B.A., Research Microprobe Analyst.

The Accelerator Laboratory, which is supported by the AEC contract and provides primary support for the radiation damage research, is staffed by Mr. B. Clymer and Mr. A. Delio, Accelerator Technicians.

Work performed in the Machine Shop (employs 12 men) is charged to the projects on an hourly basis.

New Projects

E. Neville Pugh, Department of Metallurgy and Mining Engineering
Study of Acoustic Emission During the Propagation of Stress-Corrosion
Cracks

Supported by Contract AT(11-1)-1198

Projects Terminated

D. S. Lieberman, Department of Metallurgy and Mining Engineering
Phase Transformations in Crystalline Solids

Supported by Contract AT(11-1)-1198

Personnel Changes

Professor E. Neville Pugh of the Department of Metallurgy and Mining Engineering has started an AEC project in MRL. Professor Pugh, who received his Ph.D. at the University of Wales (Cardiff) in 1959, joined the Metallurgy Department of the University of Illinois in 1970.

Dr. Victor Mossotti, Senior Research Chemist, accepted an Associate Professorship of Chemistry at the University of Minnesota. His position has been filled by Dr. Charles A. Evans, formerly at the Kennecott Copper Company. Dr. Evans received his Ph.D. in analytical chemistry in 1968 at Cornell University.

Professor George Russell, who has been Associate Head of the Materials Research Laboratory, has resigned this position to join the Graduate College of the University of Illinois.

Mr. James Pence has resigned the position of Departmental Business Manager of the Materials Research Laboratory to occupy a similar position in the Department of Physics. Mr. John Stanley, formerly Laboratory Manager for the School of Life Sciences, has replaced Mr. Pence.

Professor Ralph Simmons, whose research is supported by the AEC contract of MRL, was appointed Head, Department of Physics, on September 1, 1970. Professor Simmons has taken the place of Professor G. M. Almy on the MRL Steering Committee.

Awards

Professor Carl J. Altstetter, Department of Metallurgy and Mining, received the W. L. Everitt Undergraduate Teaching Excellence Award, University of Illinois, 1970.

Professor Paul A. Beck, Department of Metallurgy and Mining, was named Fellow, American Society for Metals, 1970.

Professor Marvin Wayman, Department of Metallurgy and Mining, received the Stanley H. Pierce Award, University of Illinois, 1970.

Professor Harry G. Drickamer, Department of Chemical Engineering, participated in the NATO International Advanced Study Institute on High Pressure Physics held at the Delft University of Technology in The Netherlands. Professor Drickamer is a member of the Advisory Committee of the Institute.

Professor Drickamer was the recipient of the W. N. Lacey Lectureship in Chemical Engineering at Cal Tech in March 1971.

Professor R. O. Simmons, Department of Physics, was appointed Head of the Department, September 1970.

Leaves of Absence

Professor C. P. Slichter, Department of Physics, is on leave for 1970-71 at the Bell Telephone Laboratories, Murray Hill, New Jersey.

Professor D. S. Lieberman, Department of Metallurgy and Mining Engineering, is on leave II Semester 1970-71 at the Department of Metallurgical Engineering, Technion-Israel Institute of Technology, Haifa, Israel.

Foreign Travel

No foreign travel expenses have been charged to the AEC contract during the past year.

Visitors

E. Fromm, Research Scientist at Max Planck Institut, Germany, is a Visiting Associate Professor in the Department of Metallurgy, working with Professor Charles Wert from February through June of 1971.

The following visitors are reported here, although the primary association was with ARPA supported members of the Laboratory:

Zhorez Ivanovich, Deputy Section Head, A. F. Ioffe Physics-Technical Institute, USSR Academy of Sciences, will work with Professor Nick Holonyak, Jr., Department of Electrical Engineering, for six months beginning November of 1970. This visit is being made under the provisions of the Agreement on Scientific Exchanges with the National Academy of Sciences of the USA and the Soviet Academy.

Harold F. Symmons, CSIRO, National Standards Laboratory, Division of Physics, Sydney, Australia, was a Visiting Research Associate Professor of Physics from September 1 - November 15, 1970.

Philippe Choquard, Professor of Physics, Ecole Polytechnique, Lausanne, Switzerland, was a Visiting Professor of Physics during the period February 1 - March 15, 1971, working with Professors John Bardeen and David Pines.

James R. Schneider is a Visiting Scientist in the Department of Physics for the II Semester of 1970-71 and is working with Professor Miles V. Klein's group. Dr. Schneider is on sabbatical leave from the University of Dayton, Dayton, Ohio.

William E. Hayes, Oxford University, England, is visiting in the Department of Physics for the period March 1 - August 31, 1971, working with Professor F. C. Brown.

C. W. Woo has an appointment as a Visiting Associate Professor in the Department of Physics for 1970-71. Professor Woo is working with Professor Bardeen's group.

Special Events

The Summer Undergraduate Research Program which supported about a dozen undergraduate students who participated in research projects during the summer months has been terminated. The University funds which supported the program will not be available for the summer of 1971.

Since the Advanced Research Projects Agency plans to terminate its support of the Materials Research Laboratory on June 30, 1972 and the National Science Foundation plans to offer support at that time, an Advisory Committee of NSF visited the Laboratory on February 16th and 17th for purposes of evaluation. The Committee members were Dr. Robert Sproull, Dr. Bruce Hannay, Dr. William Harris, Dr. William Wright, Dr. Howard Etzel, Dr. Angelo Bardasis. Dr. Donald Stevens of the AEC accompanied the Committee.

1970 Symposium on Thermal Expansion of Solids, Sante Fe, New Mexico, June 10-12, 1970. The 1968 Symposium of this title was held in September of that year at the Gaithersburg Laboratories at the National Bureau of Standards, co-sponsored by the Bureau and by Westinghouse Astronuclear Corporation. Its success in bringing together a broad variety of materials scientists and engineers from many government, industrial, and academic laboratories prompted the organization of a second symposium. For the second, the Materials Research Laboratory joined with Sandia Laboratories, Albuquerque, New Mexico, to organize and arrange an extensive program of meetings held in the State Land Office Building, Santa Fe, June 10-12, 1970.

Preparation and printing of announcements and of the Summary Program were carried out in the Materials Research Laboratory. Professor R. O. Simmons, Department of Physics, was co-chairman of the Symposium. Forty-three papers were presented and discussed by about 200 materials scientists and engineers. Extensive critical discussion and rather unusual contacts between working ceramists, metallurgists, chemists, physicists, and mechanical engineers were again the rule. The Proceedings of the Symposium are presently being edited for publication later this year in a special section of the JOURNAL OF APPLIED PHYSICS.

Significant Results

Important advances were made in understanding the properties of body-centered-cubic metals. Among these advances were the discovery of $V_{16}N$ in aged specimens of vanadium-nitrogen with less than 6% nitrogen in the form of disc shaped precipitates with a {100} habit plane (Altstetter). Diffusion measurements in Nb have shown the existence of H-H pairs, and H-O and H-N pairs have been found in Nb-O and Nb-N alloys, respectively. Binding energies and diffusion energies of the pairs have been measured and quantum mechanical tunneling of H demonstrated at low temperatures (Birnbaum). The solubility limit of hydrogen in Nb has been determined from 90°K to 300°K and the rate of hydride formation measured in this same temperature range (Wert). In a study of solutions of carbon in vanadium, a sequence of carbides have been found. The stable phase V_2C is incoherent with the metal (Wert). The activation volume for self-diffusion in beta titanium was found to be characteristic of the bcc metals despite anomalous diffusional behavior (Lazarus).

The study of transition metal alloys by magnetic Mössbauer and specific heat measurements has indicated that Co-Re solid solutions in the range from 65° to 70% Co are not ferromagnetic but superparamagnetic systems (Beck). Also, a study of the magnetic properties of Fe-Al alloys and the effect of heat treatment on the atomic order of Fe_3Al has been explained (Beck). Diffusion of Fe and Co in FeCo was measured in the fcc, bcc and CsCl-type ordered phase and significant information concerning the atom jump mechanisms was obtained (Lieberman).

The understanding of the properties of transition metal carbides was advanced by a determination of the activation energy for motion of carbon vacancies in titanium carbide at high temperatures by measurement of the chemical diffusion coefficient of carbon (Williams). Also, the electrical resistance of vanadium carbide was measured from 2°K to 1673°K. A precise determination was made of the temperature of the transition from disordered carbon vacancies to a superlattice. The transition was found to be first order (Williams).

Examination of tungsten wire-copper composites has shown that, contrary to the usual assumptions, the initial density of dislocations due to differential contraction strains is a strong function of the fiber fraction! The variation in initial density dominates the subsequent behavior of the copper matrix--an unexpected result (Metzger).

Thin film studies using cobalt have suggested that recrystallization, rather than oriented nuclei, are responsible for epitaxy (Wayman). Thermocouples have been developed (for the measurement of film temperatures during evaporative deposition) which have a response time of 10^{-8} seconds and are suitable for study of laser beam characteristics (Wayman).

Magnetic spin resonance experiments have determined the energy of migration of vacancies in aluminum (Rowland). A novel double resonance technique (Electric-Magnetic Double Resonance) has been tested with encouraging results. The importance of the technique is that it will permit the observation of small quadrupole couplings which cannot be observed by previous methods (Slichter).

Advances were made in the study of the fundamental properties of crystals. A novel technique using inertial stresses permitted the measurement of long range atomic forces in NaCl (Granato). A rigorous test was made of the renormalized harmonic approximation theory of crystal dynamics by determining the temperature dependence of the ultrasonic wave velocities in solid neon (Simmons). Debye-Waller factors were measured in crystalline Kr and Ne over a wide temperature range (Simmons). A comprehensive investigation of the magnetic and electronic properties of liquid alloys was completed and, jointly with Professor C. Flynn, a general theory of the electronic impurity bands was developed (Wert).

Experiments on crystalline defects resulted in a determination of the vacancy concentration in solid argon (Simmons).

In the very high pressure studies it has been possible to isolate specific products from solid state reactions of organic compounds (Drickamer). The surprising low spin to high spin transition with increasing pressure of a number of iron compounds has been explained (Drickamer).

PROJECT TITLES AND PRINCIPAL INVESTIGATORS

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

Mechanisms of Solid State Transformations

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

Electronic Structure of Transition Metal Alloys

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

Point Defect - Dislocation Interactions

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

Phase Transformations in Crystalline Solids

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

Dislocations and Surface Barriers

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

Decomposition of Unstable Solid Solutions

John E. Morral, Ph.D., Assistant Professor of Metallurgical Engineering

Study of Acoustic Emission During the Propagation of Stress-Corrosion Cracks

E. Neville Pugh, Ph.D., Associate Professor of 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

Solid State Phase Transformations and Thin Films

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 Metallurgy and Mining Engineering

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

Use of Very High Pressure to Investigate the Structure of Matter

Harry G. Drickamer, Ph.D., Professor of Chemical Engineering and
Physical Chemistry

Anharmonic Effect 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

Nuclear Magnetic Resonance in Solids
Charles P. Slichter, Ph.D., Professor of Physics

Physics of Refractory Materials
Wendell S. Williams, Ph.D., 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 MaterialsMechanisms of Solid State Transformations

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

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Carl J. Altstetter, Professor

Junior Staff: William J. Nickerson, Research Assistant
Dilip M. Shah, Research Assistant

Object: There are remarkable changes in the properties of body-centered-cubic refractory metals when they absorb the atmospheric contaminants oxygen and nitrogen. The phase relationships in such interstitial alloys are being studied as a key to the understanding and control of mechanical properties of vanadium and niobium. These two metals hold great promise for nuclear power reactor structural applications provided the embrittlement and oxidation problems can be ameliorated sufficiently. In this research program vanadium and niobium and their alloys are doped by heating wire or strip specimens in an atmosphere containing a fixed, known quantity of oxygen or nitrogen.

Mechanical property changes in V-N alloys will be studied as a function of the amount and form of $V_{16}N$ or other precipitated nitride. Solid solution behavior of oxygen in niobium and other bcc metals is being determined by emf measurements. The alloy under study is made one electrode in a solid electrolytic cell and its oxygen activity is determined in reference to a standard electrode by measuring the reversible cell potential.

Results, FY71: We have completed the initial phase of the V-N work in which the dilute nitride, $V_{16}N$, has been identified as the one which forms from supersaturated solid solutions up to 6 at.% N. Its structure and growth morphology has been determined by electron and x-ray diffraction. This work has implications with regard to interstitial site occupancy and solute-solute interactions in bcc metals.

Results Expected, FY72: The apparatus for emf measurements will yield results for model systems of electrodes and for different temperatures. Apparatus for doping the relatively thick specimens of alloys of interest will be constructed. Vanadium and niobium and their alloys will be doped with oxygen for determination of their solution thermodynamic properties

and oxide phase boundaries. Mechanical property results will be obtained for V-N alloys as a function of composition and aging treatment.

Results Expected, FY73 and Beyond: The electrolytic method of studying refractory metal-oxygen systems will be applied to other metals. Possibilities for use of the technique in nitrogen-containing systems will be explored. Electrolytic oxidation control is a possibility well worth investigation. Understanding of the phase relationships in binary V-N alloys will be applied to more complicated metal systems.

Publications, Calendar Year 1970:

D. I. Potter

Prediction of the Operative Slip System in CsCl Type Compounds Using Anisotropic Elasticity Theory

J. of Materials Science and Engineering 5, 201-209 (1970)
COO-1198-630

M. S. Rashid and C. J. Altstetter

A Computer Program for X-Ray Line Broadening Analysis

J. of Applied Crystallography 3, 120 (1970)
COO-1198-656

H. Savage and C. Altstetter

Composition of Phases in Nb-N and Nb-Zr-N Alloys

J. of Less Common Metals 22, 399-407 (1970)
COO-1198-657

M. S. Rashid and C. J. Altstetter

X-Ray Study of Deformed and Transformed Co-Ni Single Crystals

J. Appl. Phys. 41, 5180-5187 (1970)
COO-1198-684

D. Hennessy and C. Altstetter

Precipitation Kinetics in Niobium(Columbium)-Nitrogen Alloys

Metallurgical Transactions 1, 1185-1188 (1970)
COO-1198-700

Donald Potter and C. J. Altstetter

Ordered Orthorhombic Subnitride of Vanadium

Scripta Metallurgica 4, 849-852 (1970)
COO-1198-757

M. Rashid and C. Altstetter

Defects and Strengthening in Transformed Crystals

Proceedings of the Conference on the Strength of Metals and Alloys,
(American Society for Metals, Metals Park, Ohio, 1970), pp. 843-847

M. S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

D. I. Potter (C. J. Altstetter, Adviser)

Phase Transformation Involving Interstitial Ordering in the Vanadium-Nitrogen System

October 1970

Also supported by the National Aeronautics and Space Administration

Electronic Structure and Magnetism in Transition Metal Alloys

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
Benjamin de Mayo, Research Associate

Junior Staff: B. C. Cheng, Research Assistant
R. W. Tustison, Research Assistant

Object: The most important structural materials in engineering applications at both ambient and high temperatures are the alloys of transition metals. The alloying behavior of metals is strongly dependent on their electronic structure. The electronic structure of the transition metals is, therefore, of practical importance in the continuing search for stronger alloys, particularly in high temperature applications. In addition, considerable scientific interest is attached to the magnetic behavior of alloys of the 3d transition metals. Experimental studies in recent years established the fact that the magnetic moments associated with the atoms of 3d metals in their alloys depend strongly on the local atomic environment. Specific information on this effect for various 3d metals is basic to a deeper understanding of magnetism. The project aims at the experimental study of the electronic structure of transition metal alloys, with particular emphasis on magnetic properties.

Results, FY71: (1) A detailed study of the bulk magnetic properties of Fe-Al alloys with 25 to 50% Al has been completed. The effect of heat treatments affecting the atomic order in Fe₃Al can now be accounted for in detail on the basis of the local atomic environment model. The magnetic properties of FeAl were found to be consistent with this model. Mössbauer spectroscopic study of the Fe-Al alloys is continuing.

(2) Work on the magnetic properties of fcc Au-Fe alloys with 5 to 15% Fe has been almost completed. At low temperatures the moments associated with the Fe atoms are frozen without long range magnetic order. Short range magnetic order appears to be associated with clusters of Fe atoms, whose size can be varied at will by heat treatment.

(3) A study of the magnetic properties of Co-rich hcp Co-Re solid solutions is continuing. The results obtained so far indicate that the 65 and 70% Co alloys are not ferromagnetic (as previously believed) but superparamagnetic with very large magnetic cluster moments.

X too short

X too short

Results Expected, FY72: The work on Co-Re alloys will be finished. Cr-Fe-Al bcc alloys will be studied by means of magnetic measurements and Mössbauer spectroscopy. Low temperature specific heat work previously done at this laboratory indicated the presence of magnetic clusters over a wide composition range. The effect of metallurgical treatments (such as deformation and low temperature anneal) on the magnetic properties will reveal any tendency for short range atomic order or clustering. In addition to throwing light on the effect of local atomic environment on magnetism, our studies are thus expected to give also information on the metallurgical structure of these alloys, important in view of the use of Cr-Fe-Al alloys in high temperature structural applications.

Results Expected, FY73 and Beyond: Detailed study of the magnetic properties of several other binary and ternary alloys in relation to the metallurgical state (e.g., short range order, clustering) is expected to result in specific information on the local atomic environment effect and, thus, to help in laying the ground for a better understanding of magnetism.

Publications, Calendar Year 1970:

E. J. Hayes and P. A. Beck
Low Temperature Specific Heat and Magnetic Study of Precipitation in a
Cu-Co Alloy
Metallurgical Transactions 1, 3267-3271 (1970)
COO-1198-724

M. S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

D. Chakrabarti (P. A. Beck, Adviser)
Transport Properties of Cr-Al Solid Solution Alloys
June 1970

E. J. Hayes (P. A. Beck, Adviser)
Low Temperature Specific Heat and Magnetic Study of Precipitation in
Cu-Co Alloys
June 1970

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
Rudolf E. Booker, Research Associate

Junior Staff: Joseph J. Au, Research Assistant
Melvin F. Bluem, Research Assistant
David File, Graduate Student

Object: One of the major limitations on the use of high strength materials is their performance in adverse environmental conditions. In the case of bcc alloys this includes their behavior in environments where they can incorporate interstitial hydrogen in the lattice and their behavior at cryogenic temperatures when they contain interstitial solutes (H, O, N, C). The diffusive behavior of hydrogen in Nb and Fe is being examined using internal friction and magnetic disaccommodation techniques. The interaction of hydrogen with other interstitials (O, N) are being studied as is the isotope effect on the diffusion (using deuterium). It is expected that these results will be of importance in the problem of hydrogen embrittlement of Nb and Fe (which is being studied under an ONR contract). The mechanical properties of Nb and Nb-N alloys at cryogenic temperatures is being studied using microstrain techniques. The factors which determine the strength and ductility of bcc metals are being investigated.

Results, FY71: The diffusion of H in Nb and Nb-O, Nb-N alloys has been studied in the temperature range 4°K to 300°K. It was shown that hydrogen interacts to form H-H pairs (with a binding energy, B, of 1500 cal/mole and a diffusion energy, Q, of 1200 cal/mole) in pure Nb and H-O, H-N pairs (B = 2000 cal/mole and Q = 4000 cal/mole) in Nb-O and Nb-N alloys. Quantum mechanical tunneling of H was established at low temperatures in these alloys. These results suggest ways of controlling the diffusion of H and influencing the low temperature hydrogen embrittlement.

The parameters which affect dislocation motion in the microstrain region ($\epsilon < 10^{-5}$) were established in pure Nb and shown to be significantly different than at higher strains. These results provide a critical test of the theories of dislocation behavior in bcc metals and appear to be consistent with strengthening mechanisms based on dislocation-interstitial interactions.

Results Expected, FY72: The studies of H in Nb alloys will be extended to include long range diffusion of H using the "Gorsky" effect and to include the behavior of deuterium in Nb. The use of the Gorsky effect will allow the measurements to be extended to much higher temperatures. Initial studies of H and D in Fe will be completed and the studies of the effects of C and N solutes on H diffusion in Fe will be undertaken. The studies of the behavior of dislocations in Nb and Nb-N alloys will be completed. These results should lead to a fairly complete description of the early stages of deformation in Nb. The information obtained from the diffusion and deformation experiments will be used as a basis for beginning a study of the effect of hydrogen on the low temperature mechanical properties of Nb. These experiments will again employ the microstrain techniques for studying low temperature deformation.

Results Expected, FY73 and Beyond: The internal friction and magnetic techniques will be applied to studies of the diffusive behavior of other light elements in bcc metals. Of particular interest is the diffusion of He in Nb and in Ni. The diffusion studies will be extended to examine the nucleation and growth of small gas bubbles containing He or H₂. The effect of these bubbles on the mechanical properties of bcc metals will be studied.

Publications, Calendar Year 1970:

B. D. Trott and H. K. Birnbaum
Extension of a Theory of Damping Due to Dislocations
J. Appl. Phys. 41, 4418-4433 (1970)
COO-1198-372

B. K. Ganguly, C. Baker, and H. K. Birnbaum
The Frequency Dependence of Dislocation Damping in Niobium
J. Phys. Chem. Solids 31, 1877-1881 (1970)
COO-1198-664

H. K. Birnbaum
Dislocation Pipe Diffusion in Silver Single Crystals
Metallurgical Transactions 1, 2025 (1970)
COO-1198-690

B. D. Trott and H. K. Birnbaum
Time Dependence of Dislocation Damping
J. Appl. Phys. 41, 4434-4438 (1970)
COO-1198-705

H. K. Birnbaum, B. L. Eyre, and W. Drotning
The Effect of Diffusivity Gradients on Diffusion to Dislocations
Phil. Mag. (submitted to)
COO-1198-761

D. G. Franklin and H. K. Birnbaum
An Anelastic Study of Quenched Gold
Acta Met. (submitted to)
COO-1198-769

M. S. Theses, Calendar Year 1970

None

Ph.D. Theses, Calendar Year 1970

D. G. Franklin, III (H. K. Birnbaum, Adviser)
Anelastic Study of Divacancy Damping in Gold
June 1970

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 K. Das, Research Assistant
Michael A. Schmerling, Research Assistant

Object: An understanding and application of phase transformations in intermetallic compounds.

Results, FY71: Diffusion of Fe and Co in an equiatomic FeCo alloy which exhibits fcc, bcc, and CsCl-type ordered phases was studied. Activation energies and frequency factors were obtained for the fcc and bcc phases. Isotope parameters were also obtained and correlation factors were calculated. The observed decrease in the isotope-effect parameter with increasing order during the bcc to CsCl-type transformation is due to an increase in the atom jump correlation with the onset of long range order.

Reversible phase transformations in equiatomic Nb-Ru alloys have been studied by electrical, optical, x ray and magnetic measurements. On cooling, the cubic beta phase transforms to a face centered tetragonal phase and the latter transforms to a face centered orthorhombic phase. A mechanism for the transformations has been proposed. The phase transformations of the equiatomic Ta-Ru alloys have also been investigated. Compositions in the vicinity of 50-50 undergo a two step cubic to tetragonal to orthorhombic martensitic transformation with decreasing temperature while compositions which deviate substantially from 50-50 show a single cubic to tetragonal transformation on cooling.

Results Expected, FY72: This project has been terminated.

Publications, Calendar Year 1970:

S. G. Fishman, D. Gupta, and D. S. Lieberman
Diffusivity and Isotope Effect Measurements in Equiatomic Fe-Co
Phys. Rev. B 2, 1451-1460 (1970)
COO-1198-688

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

B. K. Das, M. A. Schmerling, and D. S. Lieberman
Some Aspects of Phase Transformation in Near-Equiatomic Niobium-Ruthenium
Alloys
Materials Science and Engineering 6, 248-254 (1970)
COO-1198-711

M. A. Schmerling, B. K. Das, and D. S. Lieberman
Phase Transformations in Near Equiatomic Ta-Ru Alloys
Metallurgical Transactions 1, 3273-3278 (1970)
COO-1198-715

M.S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

None

Dislocations and Surface Barriers

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 K. Chawla, Research Assistant
Prabir Ranjan Bhowal, Research Assistant

Object: Dislocation distributions and chemical behavior of composite and coated crystalline materials are examined with particular attention to matrix hardening at small strains. Current strain hardening theories are based on highly idealized configurations at substantial strains, and the aim is to develop realistic models of matrix flow stress at small strains and provide information applicable to the design of technological materials containing hard phases. Microstrain mechanical techniques and dislocation etch pitting are used.

Results, FY71: In tungsten wire reinforced copper composites, techniques were developed for etch pitting sections and dislocation density gradients determined up to fairly high densities in undeformed and deformed matrix. The initial densities were greatly increased by differential contraction strains and this was a strong function of fiber fraction, contrary to the usual assumption made in treating composites. It was this variation in initial state which dominated subsequent matrix behavior and not pile-up or plastic constraint as have been proposed previously as responsible for fiber-matrix interactions.

too short

Results Expected, FY72: Our previous observations on the dislocation density-flow stress relations in copper crystals at small strains have shown that the often used empirical correlation developed at larger strains for a limited range of conditions is quite different at small strains. The relations between flow stress and dislocation density and distribution will be determined at small strains in multiple glide situations for several initial densities and applied to the analysis of flow stress gradients in plastically inhomogeneous materials.

Results Expected, FY73 and Beyond: Attention will continue to be focussed on characterization of matrix dislocation structure and systems will be sought where the composite can also be prepared for examination by transmission electron microscopy. Studies will be extended to systems where the matrix has a basically different mode of microyielding, such as a copper alloy matrix, and its response determined as a function of the ratio of dislocation mean free path to fiber spacing.

Publications, Calendar Year 1970:

O. P. Arora and M. Metzger
Corrosion Anisotropy in Aluminum
Proceedings of the Fourth International Congress on Metallic Corrosion,
(N.A.C.E., Houston, Texas) (submitted to)
COO-1198-750

M. S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

None

Decomposition of Unstable Solid Solutions

Principal Investigator: John Morral, Ph.D., Assistant Professor of
Metallurgical Engineering

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: John E. Morral, Assistant Professor

Junior Staff: Durgam G. Chakrapani, Research Assistant
Phillippe L. Lecocq, Research Assistant

Object: The object of this work was to develop an understanding of the kinetic processes which lead to age hardening in binary and ternary alloys, especially with regard to the influence of composition and thermal cycles.

Results, FY71: (1) A computer program was written which predicts the kinetic behavior of ternary alloys from that of binary alloys.
(2) The kinetic behavior of various ternary systems was surveyed.
(3) The field of defect annealing was reviewed and applied to the problem of altering heat treatment time cycles.

Results Expected, FY72: This project will be terminated at the end of August 1971.

Publications, Calendar Year 1970:

None

M.S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

None

Study of Acoustic Emission During the Propagation of Stress-Corrosion Cracks

Principal Investigator: E. Neville Pugh, Ph.D., Associate Professor of Metallurgy

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: E. Neville Pugh, Associate Professor

Object: At the present time, the performance of numerous structural materials is limited by their susceptibility to stress-corrosion cracking. Service failures are frequent and costly, recent examples including the cracking of Ti oxidizer tanks (N_2O_4) in the Apollo program and of 304 stainless steels in the Nine-Mile Point Reactor (high-purity water). Because of their complexity and multi-disciplinary nature, the mechanisms of these failures remain uncertain, but it is evident that real progress in this area requires much basic knowledge.

The present program forms part of a broad study of the mechanisms of stress-corrosion phenomena. The initial phase is directed towards establishing an important fact--is crack propagation a continuous or discontinuous process? This question, which is the subject of much controversy, will be investigated in several systems by means of acoustic-emission and detailed fractographic studies.

Results, FY 71: Not applicable--new project.

Expected Results, FY72: Previous studies have indicated that stress-corrosion fracture surfaces in several systems exhibit a series of parallel markings, which, according to some workers, correspond to successive positions of the crack tip during discontinuous brittle fracture. If this view is correct, then the stress waves generated during crack propagation should be emitted as a series of discrete events. Acoustic-emission studies will be undertaken to resolve this issue. From the time intervals between discrete emissions, if they are found to occur, and from the macroscopic crack velocity, it will be possible to calculate the spacing between the surface markings and this can be compared with observed values.

The studies will be initiated on the non-metal AgCl because this system offers several advantages experimentally. The technique will then be applied to alpha-brass bi-crystals in ammoniacal solutions.

Results Expected, FY73 and Beyond: It is envisaged that the acoustic-emission studies will be extended to several commercially important alloys, including Al alloys, stainless steels and Ti alloys. Once the nature of crack propagation is established, further effort will be focussed on a more detailed understanding of the cracking process. For example, the failure of alpha-brass in aqueous ammonia is thought to result from the repeated formation and rupture of a brittle cuprous-oxide layer, so that attention would be given to the mechanism of oxide growth in this system.

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: Sudhansu Chakravorty, Research Assistant

Object: The strength of metals for use at very high temperature can be improved by the addition of dispersions of inert particles. However, marked directional properties at high temperature may be developed by fabrication processes such as rolling. The objective, therefore, is to determine the mechanism by which annealing textures are developed in such alloys and the extent to which these textures may affect mechanical properties.

Results, FY71: The effectiveness of dispersions of CuAl_2 as they modify annealing textures in rolled Al has been compared with the solute form, and a similar investigation of nickel containing dispersions of thoria has been undertaken.

Expected Results, FY72: This project will terminate on August 1971.

Publications, Calendar Year 1970:

W. T. Shieh and B. G. Ricketts
Experimental Study of the Elastic-Modulus Effect in the Interaction of Vacancies with Dislocations and Dislocation Ribbons in Pure Silver
J. Appl. Phys. 41, 522-531 (1970)
COO-1198-538

M. S. Theses, Calendar Year 1970:

A. K. Mukhopadhyay (B. G. Ricketts, Adviser)
The Effect of a Dispersed Second Phase on the Annealing Texture of Rolled Aluminum-Copper Alloys
October 1970

Ph.D. Theses, Calendar Year 1970:

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
Ernst D. von Meerwall, Research Associate

Junior Staff: Lance Confer Labun, Research Assistant
Robert R. Rahn, Inland Steel Fellow
Andrew C. Yen, Research Assistant

Object: We are examining atomic diffusion in aluminum alloys by means of magnetic relaxation in order to critically compare the information so gained with that obtained by chemical and tracer techniques. Earlier work has shown that the various measurements do not determine the same physical quantities but taken together permit more detailed analysis of the solute and solvent jump processes than has previously been possible. The aluminum base alloys being studied are of considerable importance, also the problems being attacked are fundamental in nature. The precipitation process in copper-beryllium binary alloys is being investigated by resonance and microscopic techniques. The resonance data indicates an effect of lattice strain, as might be expected around very small coherent precipitate particles. Our work is an attempt to introduce a quantitative treatment of this effect, and thus to further the understanding of precipitation hardening processes.

Results, FY71: A successful measurement of the energy of migration of vacancies in aluminum was completed during the past year, also the activation energy for self-diffusion was redetermined. The values obtained (0.55 and 1.32 eV respectively) are believed to be accurate within the temperature range from 260-500°C. The significance of the work lies in the fact that the new measurements derive from experimental methods completely independent of those used heretofore, and thus confirm the migration energy obtained from certain quench-anneal experiments.

Several vanadium base transition metal solid solutions have been investigated by comparing their observed nuclear resonance absorptions with synthesized curves based upon assumed electric field gradient distributions. The work is finished, but is not considered to be complete without more detailed understanding of vanadium-oxygen and nitrogen alloys. The latter are being studied.

Results Expected, FY72: We plan to complete work on several aluminum base alloy systems in this period. In particular data on Al-Ge, Si, In, Cu, and one or more transition metal solutes will be obtained, as well as more extensive data on Al-Ag. This will make possible improvements in the theory,

and should enable us to treat correlated motion and impurity vacancy binding energies more realistically.

An exploratory investigation of the microwave resonance response of molten organic salts will be undertaken in this period.

Results Expected, FY73 and Beyond: Since work actually in progress at any one time depends somewhat on student preference and support, several studies which are dormant at present are to be resumed as soon as possible. These include concentrated copper alloys, finely divided platinum (surface effects and catalytic action), order-disorder transformations, and nuclear resonance at high pressure. In addition one student claims an interest in polymers and we have been discussing quantitative studies based on the unique surface characteristics of certain polymer crystals.

Publications, Calendar Year 1970:

F. Y. Fradin and T. J. Rowland
 ^{63}Cu Nuclear Magnetic Relaxation in Cu-Ni Alloys Near the Critical Concentration
 Solid State Communications 8, 1047-1050 (1970)
 COO-1198-805

R. H. Geils
 Electronic Balancing Circuit for a Crossed-Coil NMR Spectrometer
 Rev. Sci. Instr. (submitted to)
 COO-1198-739

E. von Meerwall and T. J. Rowland
 Quadrupolar Effects in the ^{51}V NMR of V-Based Transition Metal Alloys
 Solid State Communications (submitted to)
 COO-1198-771

F. Y. Fradin and T. J. Rowland
 Comments on Effects of Electron-Electron Interactions on Nuclear Spin-Lattice Relaxation Times in Aluminum
 Phys. Rev. (submitted to)

M. S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

None

Solid State Phase Transformations and Thin Films

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

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: C. Marvin Wayman, Professor
Hau-Ching Tong, Research Associate

Junior Staff: Gilles P. Asselin, Research Assistant
Yue-Kong Au, Research Assistant
Ivan Cornelis, Research Assistant
Thomas P. Darby, Research Assistant

Object: Comparatively new techniques are used to study transformation phenomena in solids, and the growth and behavior of thin solid films. It is of future intent to employ well-characterized thin films for more extended studies of phase transformations in the solid state. Major features of interest are crystallographic relationships between phases and identification of nucleation sites. The former provides information on how transformed regions grow, and the latter indicates how and why transformations begin. These characteristics are of both academic and technological importance: the crystallographically related substructure in many transformations imparts a further structure sensitivity (mechanical behavior, character of diffusion reactions such as tempering, etc.); if nucleating sites are understood there should be considerable advantage to transformations following "controlled" nucleation.

Present investigations: comparison of the crystallography of martensitic and "bainitic" transformations in beta Cu-Zn alloys; mechanistic implications of surface relief topography in diffusion controlled precipitation reactions in Al-Cu alloys; feasibility of preparing certain alloy films by sputtering; effect of substrate irregularities and gaseous contamination on thin film growth.

Results, FY71: Certain metallic thin film combinations have been well characterized and their thermoelectric properties have been used to determine the temperature rise in thin films per se during growth and the rise in temperature (due to electron irradiation) during specimen observation in an electron microscope. There are several indicated applications of such thin film thermocouples (for example, determining the rise and fall characteristics of a laser beam, and any other application where a sensing response time of 10^{-8} seconds is required). An analysis of the growth of evaporated cobalt films in controlled conditions indicates the importance of gaseous contaminants (particularly nitrogen) in inhibiting epitaxial growth; further, the results obtained suggest that recrystallization in both continuous and discontinuous

films gives rise to epitaxy, in contrast to the "oriented nuclei" hypothesis. Investigations of the martensitic and "bainitic" transformations in beta brass are establishing definite characteristics peculiar to each, and present indications are that previous crystal structure interpretations for the martensitic phase may be in error. Attention is presently given to the superelastic behavior in beta brass martensite (which we have recently discovered) and its consequences. A general purpose DC-RF sputtering apparatus has been assembled, and special targets for the sputtering of small amounts of exploratory materials have been designed and tested. Preliminary films of copper, Cu₃Au, 18-8 stainless steels, and Fe-Ni alloys have been made; the films and sputtering variables are under study. Initial observations (by interferometry) of surface relief effects attendant to diffusion controlled precipitation reactions indicate the importance of surface nucleation and diffusion; it appears that interferometry may be used in such transformations to determine quantitatively the role of solute surface diffusion.

Results Expected, FY72: The newly designed sputtering apparatus is expected to be reliably functioning, and accordingly thin films of numerous materials of interest can hopefully be prepared at will. It will be important to establish proper annealing treatments to develop typical polycrystalline specimens (sputtered thin films are often "amorphous") for further studies as outlined in "Object". The initial results obtained by interferometry studies on the CuAl₂ precipitate (indicating the importance of surface diffusion and nucleation) will be extended to the Cu-Be and other systems deemed appropriate for comparison and analysis. Thin film studies will attempt to reinforce tentative conclusions on the effect of gaseous impurities, in-situ recrystallization, and the nature of the substrate surface; of further concern will be the origin of dendritic growth which we have observed under certain conditions (chromium vapor deposited on NaCl and graphite flakes). Although the martensitic and "bainitic" transformations in beta brass have been considered as analogous, the present work suggests that the substructures (and crystallographic mechanisms) are fundamentally different. This will be investigated in further detail.

Results Expected, FY73 and Beyond: The use of thin films per se for investigation of various phenomena in solids (particularly alloys) is promising. It is considered that the present work will establish guidelines to the extent that films of arbitrary material and composition can be manufactured for any intended purpose--with particular regard to reproducible polycrystalline films. It is hoped that the initial work on the nucleation and growth of transformations in such films will have been carried out and that both advantages and shortcomings will be realized. As a result of the present work, certain variables affecting the growth and final character of evaporated metallic films seem to be established. It will be necessary to ascertain that the various ideas are of universal validity. The important question of distinguishing

between, and recognizing common features of transformations will be probed to greater depths. The salient feature of martensitic transformations is the invariant plane strain nature of the relief observed at a free surface. But a definite, and sometimes similar relief, is also observed for diffusion controlled transformations, obviously of a different character. It is the present feeling that future, and detailed work on the relief effects attendant to solid state transformations will clarify differences, and will provide important implications of a mechanistic nature. A clear separation of the various types of transformations (martensitic, massive, bainitic, spinodal, ordering, etc.) cannot but help exploit the advantages of each, once the fundamental characteristics are clearly in hand.

Publications, Calendar Year 1970:

K. Shimizu, M. Oka, and C. M. Wayman

The Association of Martensite Platelets with Austenite Stacking
Faults in an Fe-8Cr-1C Alloy

Acta Met. 10, 1005 (1970)

COO-1198-686

T. P. Darby and C. M. Wayman

The Growth of Au Films on Graphite in Ultra-High Vacuum
phys. stat. sol. 1, 729 (1970)

COO-1198-693

Y. Fukano and C. M. Wayman

Dendritic Growth of Evaporated Chromium

J. of Crystal Growth 7, 163-176 (1970)

COO-1198-695

H. M. Ledbetter and C. M. Wayman

A Computer Program for Martensite Crystallography

J. Mat. Sci. and Eng. 5, 872 (1970)

L. Delacy and I. Cornelis

The Variation of Stacking Order and Structure Symmetry in Copper-
Base Martensites

Acta Met. 18, 1061 (1970)

C. M. Wayman and H. M. Clark

Surface Relief Effects in Solid State Phase Transformations

Phase Transformations (American Society for Metals, 1970), Ch. 2, pp 59-114

C. M. Wayman

Martensitic Transformations

Modern Diffraction Techniques Applied to Problems in Materials Science,

edited by J. Van Landuyt, R. Gevens, and S. Smelinckx, (North Holland
Publishing Company, Amsterdam, 1970), Ch. 7, pp. 187-232

K. Shimizu, M. Oka, and C. M. Wayman
Transmission Electron Microscopy Studies of 225 Martensite in an Fe-8%
Cr-1% C Alloy
Acta Met. (submitted to)
COO-1198-770

M. S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

H. Ledbetter (C. M. Wayman, Adviser)
The Crystal Structure and Martensitic Transformation in Near
Equiatomic AuCd
February 1970

E. A. Merriman (C. M. Wayman, Adviser)
Superplastic Deformation in a Eutectic Alloy of Cadmium and Zinc
February 1970

T. J. Patrician (C. M. Wayman, Adviser)
Nucleation and Growth of Evaporated Cobalt Films
February 1970

C. R. Rarey (C. M. Wayman and C. A. Wert, Advisers)
Mössbauer Study of the Tempering of Martensite
February 1970

The Study of the Nature of Solid Solutions of Metals

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

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Charles A. Wert, Professor
Eckehard Froom, Visiting Associate Professor

Junior Staff: Dwight Diercks, Jones and Laughlin Fellow
Bidyut K. Ganguly, Research Assistant
Ramamurthy K. Viswanadham, Research Assistant

Object: Alloys of the important body-centered-cubic metals with interstitial carbon, nitrogen, oxygen, and hydrogen are being investigated. (1) Characteristics of the interstitial compounds are under study--crystal structure, composition, crystallographic relationship with the metal lattice. (2) The effect of compounds on the overall mechanical properties of the alloys is being determined. (3) The kinetics of reactions which occur as the compounds form are being related to diffusion rates of constituents and nucleation of phases. The overall purpose is to determine as much as possible about the sequence of compounds which form during metallurgical treatments, the nature of the phases and their effect on the mechanical properties of the alloys.

Results, FY71: (1) The solubility limit of hydrogen in Nb has been determined from 90°K to 300°K. The rate of hydride formation in the metal has been determined over this range.

(2) The formation of a sequence of carbides from solutions of carbon in vanadium has been deduced to be the following: Solid solution → Carbide I → Carbide II → V_2C . The stable phase V_2C has been investigated by electron microscopy and electron diffraction. It is incoherent in the metal; the misfit strain of 14% is taken up by generation of dislocations. Carbide II is a fine-scale, coherent phase barely visible by electron microscopy. Carbide I is deduced only from property changes; it is too fine to be seen by electron microscopy.

(3) An investigation of the magnetic and electronic properties of liquid metal alloys has been completed. Solvents were Cu, Zn, Ga, Ge and their mixtures; solutes were Mn, V, Fe, and Co in dilute concentration. A comprehensive theory of the electronic nature of the impurity bands in these metals has been determined. (This is a joint study with Professor Flynn.)

(4) An investigation of the kinetics of liquid-phase sintering of iron powder has been carried out using electron-microprobe examination. The sintering--which is often carried out commercially using a liquid Cu-Co alloy--involves a micro-chemical interaction at the solid-liquid interface of great complexity. The first phase of the study will be completed this year.

Results Expected, FY72: (1) Investigation of the lower carbides in V-C alloys will be completed. Mechanical effects will be examined during formation of the carbides.

(2) Study will begin on the carbides of niobium. An attempt will be made to examine a commercial Nb-W-Hf-C alloy.

(3) Examination of the dislocation characteristics of the Nb-NbH two phase mixture will be made.

Results Expected, FY73 and Beyond: (1) Examination of variety of carbides, nitrides, oxides, and hydrides of transition metals. One needs to determine the long range interactions which produce the variety of compounds observed.

(2) Extension of techniques being successfully used in simple systems to more complicated alloys. First steps should be the simultaneous formation of carbides and nitrides or carbides and oxides in Nb or V-based alloys.

(3) Growing knowledge of nature of hydrogen bearing alloys should gradually lead to better understanding of mechanical effects of hydrogen in bcc metals.

Publications, Calendar Year 1970:

Yuh Fukai and Kenji Watanabe
Nuclear Magnetic Resonance in Aluminum Alloys
Phys. Rev. B 2, 2353-2360 (1970)
COO-1198-713

C. Wert, D. O. Thompson, and Otto Buck
Internal Friction of Nb-H Alloys
J. Phys. Chem. Solids 31, 1793-1798 (1970)
COO-1198-717

C. A. Wert
Damping of Interstitial Atoms in bcc Metals
J. Phys. Chem. Solids 31, 1771-1783 (1970)
COO-1198-718

N. Dahlstrom, C. C. Dollins, and C. A. Wert
The Cold-Work Peak in Undeformed Niobium
Acta Met. (submitted to)
COO-1198-759

C. P. Flynn, J. J. Peters, and C. A. Wert
Excited Configurations of Local Moments
Phys. Rev. Letters (submitted to)
COO-1198-773

Also supported by the Advanced Research Projects Agency,
Contract HC 15-67-C-0221

M. S. Theses, Calendar Year 1970:

R. K. Viswanadham (C. A. Wert, Adviser)
The Internal Friction of Nb-H Alloys
February 1970

Ph.D. Theses, Calendar Year 1970:

C. R. Rarey (C. A. Wert and C. M. Wayman, Advisers)
Mössbauer Study of the Tempering of Martensite
February 1970

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 of Physical Chemistry; Member, Center for Advanced Study

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Harry G. Drickamer, Professor
Giovanni De Pasquali, Research Assistant Professor

Junior Staff: Cecil B. Barger, Research Assistant
Vincent C. K. Chiu, Research Assistant
Curtis W. Frank, Teaching Assistant
Myron Kuhlman, NSFG Fellow
Nicholas A. Halasa, Research Assistant and NSF-T
Sun H. Moon, University of Illinois Fellow
Byron Okamoto, Teaching Assistant
Priestly J. Wang, Research Assistant

Object: The purpose of this project is the investigation of the electronic behavior of solids, using very high pressure as a primary tool. Present experimental techniques permit optical absorption and luminescence measurements to 160 kilobars, electrical resistance studies to 500 kilobars, x-ray diffraction measurements to 400 kilobars, and Mössbauer resonance studies to 250 kilobars.

Basically, our approach is to study the relative displacement of one set of energy levels with respect to another as the pressure increases, concentrating on those systems where these displacements lead to a new ground state. We then study and try to understand the physical and chemical characteristics of these new ground states.

Projects currently active include: (1) Mössbauer resonance and optical absorption studies of the chemistry of iron including changes of oxidation state and spin state. There is considerable emphasis on solid biological prototype systems.

(2) Optical absorption studies of electronic transitions in transition metal, rare earth, and actinide compounds.

(3) Studies of electronic properties of organic crystals and complexes with emphasis on the chemical properties of the new ground states, and the types of possible solid state reactions.

(4) High pressure photochemistry in the solid state.

Results, FY71: (1) We have improved our capabilities for infrared absorption studies at high pressure and extended the wavelength range to 13.0 microns.

(2) We have significantly improved our understanding of the effect of pressure on the oxidation state and especially of the spin state of iron. We have explained the rather surprising low spin to high spin transition in a number of materials. We have made considerable progress in explaining the behavior of iron in solid biological prototype material.

(3) We isolated a number of specific products from solid state reactions in organic compounds and complexes and made progress in elucidating their structure.

Results Expected, FY72: (1) We expect to extend our studies of the high pressure chemistry of iron, especially in biological prototype molecules. By the end of FY73 much of this should be completed.

(2) We are initiating some work on the behavior of iron in ferrites.

(3) We are initiating some work on the electronic transitions in rare earth and actinide compounds.

(4) We hope to initiate work on the high pressure chemistry of copper.

(5) We hope to intensify considerably our efforts on understanding the behavior of solid electron donor acceptor complexes at high pressure, and their role in solid state reactivity.

(6) We hope to initiate studies of hydrogen bonding in the solid state at high pressure.

(7) We hope to initiate photochemical studies at high pressure.

Results Expected, FY73 and Beyond: Our major mission in the next several years is to obtain a thorough understanding of electron transfer and related processes in solids at high pressure; to deepen our understanding of the electronic behavior of solids; and to generalize the chemistry of the solid state. The emphasis will be (1) on the chemistry of transition metal, rare earth, and actinide elements, and (2) on the behavior of organic molecules and complexes in the solid state. In particular, we feel that photochemistry will be an important tool.

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 100 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 1970:

H. G. Drickamer, D. C. Fisher, and D. C. Grenoble
High Pressure Mössbauer Studies of the Oxidation State and Spin State
of Iron Compounds

Les Propriétés Physiques Des Solids Sous Pression, (Editions Du Centre
National De La Recherche Scientifique-15, quai Anatole-France-Paris-
VII^e, 1970), pp. 55-63

COO-1198-643

C. W. Christoe and H. G. Drickamer

Effect of Pressure on the Quadrupole Interaction in Iron-Fluorine
Compounds

Phys. Rev. B 1, 1813-1822 (1970)

COO-1198-646

H. G. Drickamer, V. G. Bastron, D. C. Fisher, and D. C. Grenoble
The High-Pressure Chemistry of Iron

Journal of Solid State Chemistry 2, 94-104 (1970)

COO-1198-681

H. S. Möller and H. G. Drickamer

Application of High Pressure to Magnetism in Metals and Alloys
Comments on Solid State Physics 2, 199-204 (1970)

COO-1198-691

M. Avinor and G. De Pasquali

Synthesis of Manganese Disulfide

Journal of Inorganic and Nuclear Chemistry 32, 3403-3404 (1970)

COO-1198-706

M. Avinor and G. De Pasquali

Synthesis of Manganese Diselenide

Journal of Inorganic and Nuclear Chemistry 32, 3595 (1970)

COO-1198-727

H. G. Drickamer

Electronic Structure and Electronic Transitions at High Pressure
Comments on Solid State Physics 3, 53-59 (1970)

COO-1198-738

H. G. Drickamer

Revised Calibration for High Pressure Electrical Resistance Cell
Rev. Sci. Instr. 41, 1667-1688 (1970)

COO-1198-745

G. A. Samara and H. G. Drickamer
Pressure Studies of Ferroelectric Properties
Comments on Solid State Physics 3, 1-7 (1970)

H. S. Müller and H. G. Drickamer
Pressure Dependence of the Curie Temperature and Magnetization in
Ferromagnetic PdFe Alloys
J. Phys. Chem. Solids (submitted to)
COO-1198-726

M. Avinor and G. De Pasquali
Sodium, Potassium, Rubidium, and Cesium Dithioferrates (III)
Inorganic Synthesis (submitted to)
COO-1198-743

C. B. Barger, M. Avinor, and H. G. Drickamer
The Effect of Pressure on the Mössbauer Resonance for Fe^{57} in MnS_2 , MnSe_2
and MnTe_2
Inorganic Chemistry (submitted to)
COO-1198-747

C. B. Barger and H. G. Drickamer
The Effect of Pressure on the Electronic Structure of Binuclear and
Mononuclear Fe(III)-Phenanthroline Complexes
J. Chem. Phys. (submitted to)
COO-1198-751

D. C. Fisher and H. G. Drickamer
The Effect of Pressure on the Spin State of Iron in Ferrous Phenanthroline
Compounds
J. Chem. Phys. (submitted to)
COO-1198-774

V. C. Bastron and H. G. Drickamer
Solid State Reactions in Organic Crystals at Very High Pressure
Journal of Polymer Science (submitted to)
COO-1198-776

D. C. Grenoble and H. G. Drickamer
The Effect of Pressure on the Electronic Structure of Ferric Hydroxamates
and Ferrichrome A
Proc. Natl. Acad. Sci. U. S. (submitted to)
COO-1198-777

D. C. Grenoble and H. G. Drickamer
The Effect of Pressure on the Electronic Structure of Protoporphyrin IX,
Hemiporphyrins, and Related Compounds
J. Chem. Phys. (submitted to)
COO-1198-780

D. C. Grenoble and H. G. Drickamer

The Effect of Pressure on the Electronic Structure of Phthalocyanine and
Iron-Phthalocyanine Derivative

J. Chem. Phys. (submitted to)

COO-1198-784

M. S. Theses, Calendar Year 1970

M. Kuhlman (H. G. Drickamer, Adviser)

High Pressure Reactions of Several Solid Organic Charge Transfer Complexes
June 1970

Ph.D. Theses, Calendar Year 1970

None

Anharmonic Effect 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
Edward R. Naimon, Research Associate

Junior Staff: Edwin R. Fuller, Jr., Research Assistant
David T. Read, Research Assistant
William F. Weston, Research Assistant

Object: The objective of the project is the establishment of the equations of state of solids, the determination of interatomic potentials, the determination of anharmonic effects, and the determination of defect properties of crystals. The technique used is the measurement of the second and third order elastic constants by determination of sound velocities under hydrostatic and uniaxial stress. The results are of fundamental significance for the understanding of the thermal and mechanical properties of solids.

Results, FY71: (1) Measurements of long range (beyond first two neighbors) forces in NaCl by the use of inertial stresses.

(2) Review article on the use of third order elastic constants in determining thermal equilibrium properties of solids.

(3) Measurement of third order elastic constants in materials (CdS) with partly ionic - partly covalent bonding.

(4) Calculation of the temperature dependence of elastic constants of cubic crystals.

Results Expected, FY72: (1) Studies of phase transitions in crystals.

(2) Relations between thermal equilibrium and mechanical properties of crystals.

Results Expected, FY73 and Beyond: (1) Applications of finite elasticity theory to properties of imperfect crystals.

(2) Mechanical studies of ferroelastic crystals.

Publications, Calendar Year 1970:

A. Ikushima

Ultrasonic Attenuation in MnF_2 Near the Néel Temperature

J. Phys. Chem. Solids 31, 283-289 (1970)

C00-1198-627

A. Ikushima

Anisotropy of the Ultrasonic Attenuation in MnF_2 Near the Néel Temperature

J. Phys. Chem. Solids 31, 939-946 (1970)

COO-1198-647

K. Kawasaki and A. Ikushima

Velocity of Sound in MnF_2 Near the Néel Temperature

Phys. Rev. B 1, 3143-3151 (1970)

COO-1198-651

J. Holder

Improvements on Pulse Superposition Velocity Measurements

Rev. Sci. Instr. 41, 1355-1356 (1970)

COO-1198-710

Also supported by Advanced Research Projects Agency,

Contract HC 15-67-C-0221

J. Holder and A. V. Granato

Thermal Expansion of Solids Regarding Phonons as Defects

J. Appl. Phys. 41, 5152-5154 (1970)

COO-1198-748

Also supported by Advanced Research Projects Agency,

Contract HC 15-67-C-0221

J. Holder and A. V. Granato

Thermodynamic Properties of Solids Containing Dislocations

Fundamental Aspects of Dislocation Theory, edited by Symmons, de Wit, and Bullough, (National Bureau of Standards Publication 317, Washington, D. C., 1970), Vol. II, p. 1223-1226

Also supported by Advanced Research Projects Agency,

Contract HC 15-67-C-0221

T. Suzuki

Second and Third-Order Elastic Constants of Aluminum and Lead

Phys. Rev. (submitted to)

COO-1198-704

A. Ikushima and R. Feigelson

Acoustic Study of the Critical Phenomena in FeF_2 Near the Néel Temperature

J. Phys. Chem. Solids (submitted to)

COO-1198-720

J. Holder and A. V. Granato

Third Order Elastic Constants and the Thermal Equilibrium Properties of Solids

Physical Acoustics (submitted to)

COO-1198-764

Also supported by Advanced Research Projects Agency,

Contract HC 15-67-C-0221

M. S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

E. R. Naimon (A. V. Granato, Adviser)
The Second and Third Order Elastic Constants of Magnesium
October 1970

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
Michael D. Feit, Research Associate
Georges Martin, Research Associate

Junior Staff: Robert Beyerlein, Research Assistant
Mark S. Jackson, Research Assistant
William A. Spurgeon, Research Assistant
Jonathan Weiss, Research Assistant

Object: Experimental and theoretical studies are undertaken to investigate the defect and electronic properties of solids as a function of pressure, over a range to 10 kbar, and of temperature over a range of 1°K to the melting point. Particular emphasis is placed on studies of diffusion mechanisms, Fermi surfaces, magnetic structures, and thermoelectric properties. These properties are basic to the application of a wide variety of solids under a large range of environmental conditions.

Results, FY71: The activation volume for self-diffusion in titanium, despite the anomalous diffusional behavior, was shown to be characteristic of other bcc metals. Studies were completed of the high pressure characteristics of some thermocouple materials. The effect of uniaxial compressions on the Fermi surface of aluminum was studied. Work was completed on the internal hyperfine interactions on a series of MnSb-Cr alloys from the ferromagnetic to the antiferromagnetic phases. Work continues on studies of pressure effects on defects in alkali halides and on magnetic properties of superparamagnetic and ferromagnetic alloys. A theoretical study of the dynamics of atom motion during the diffusion process is underway. Work has been started on a study of the effect of pressure on the low-temperature thermopower of metals.

Results Expected, FY72: Work should be completed on studies of the effects of uniaxial strain on the Fermi surface of aluminum, and of the effects of pressure on the superparamagnetic-ferromagnetic transition in Pd-Ni alloys. The dynamical analysis of the diffusion process should be completed. Studies should be completed of the effects of pressure on sodium tracer diffusion in NaCl. Apparatus should be completed for the low-temperature, high-pressure thermopower studies. A new program of study of the effects of pressure on vacancy-impurity interactions in alkali halides may be undertaken, as well as others, as yet unspecified.

Results Expected, FY73 and Beyond: The current program will be extended in directions dictated by the results obtained during the next year. It is hoped to develop new programs for studying defect and electronic properties of solids, including, in particular, the effects of nonequilibrium conditions and defect-defect interactions.

Publications, Calendar Year 1970:

J. J. Burton

Configuration, Energy, and Heat Capacity of Small Spherical Clusters of Atoms

J. Chem. Phys. 52, 345-352 (1970)

COO-1198-649

J. J. Burton and D. Lazarus

Divacancy Motion Energy in Gold

Appl. Phys. Letters 16, 131-132 (1970)

COO-1198-667

S. C. Fain, Jr., and D. Lazarus

X-Ray Investigation of Solid Helium

J. Appl. Phys. 41, 1451-1454 (1970)

COO-1198-677

S. C. Fain, Jr., and D. Lazarus

Combined Thermal Conductivity and X-Ray Study of Hexagonal-Close-Packed Helium-4

Phys. Rev. A 1, 1460-1467 (1970)

COO-1198-682

R. N. Jeffery and D. Lazarus

Calculating Activation Volumes and Activation Energies from Diffusion Measurements

J. Appl. Phys. 41, 3186-3187 (1970)

COO-1198-702

J. J. Burton and D. Lazarus

Annealing of Quenched Defects in Gold

Phys. Rev. B 2, 787-798 (1970)

COO-1198-712

D. N. Yoon and R. N. Jeffery

Pressure and Composition Dependence of the Order-Disorder Critical Temperature of CoFe

Solid State Communications (submitted to)

COO-1198-679

P. Vernes, M. Pasternak, J. Groves, and D. Lazarus
Mössbauer-Effect Investigation of the Magnetic Phases in $\text{Mn}_{1-x}\text{Cr}_x\text{Sb}$ System
Phys. Letters (submitted to)
COO-1198-755

Also supported by the National Science Foundation

M. D. Feit
Some Formal Aspects of a Dynamical Theory of Diffusion
Phys. Rev. (submitted to)
COO-1198-758

R. N. Jeffery
The Effect of High Pressure on Self-Diffusion in Beta-Titanium
Phys. Rev. (submitted to)
COO-1198-763

M. S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

R. N. Jeffery (D. Lazarus, Adviser)
The Effect of High Pressure on Self-Diffusion in Beta-Titanium
October 1970

Properties of Noble Gas Crystals

Principal Investigator: Ralph O. Simmons, Professor of Physics;
Head, Department of Physics

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Ralph O. Simmons, Professor
H. Reinhard Balzer, Research Associate

Junior Staff: William H. Hardy, University Fellow
Frederick N. Kabat, Research Assistant
David S. Kupperman, Research Assistant
William E. Schoknecht, Research Assistant
Larry Schwalbe, Research Assistant and U. S. Steel Fellow
Ronald W. Wilkins, Research Assistant and
Humble Oil Fellow

Object: This project supports the study of anharmonic and quantum zero-point effects upon the lattice dynamics of crystals. Such effects are exceptionally large in noble gas crystals, and can be quantitatively investigated in the laboratory over a broad range of temperatures and densities. Noble gas crystals are ideal model substances upon which critical and relatively independent tests may be made of theories of lattice dynamics and of the nature of atomic interactions in the condensed state. Techniques used include the preparation and handling of massive single crystals, analysis of their purity and crystal perfections, x-ray lattice parameter and Bragg and diffuse intensity measurements, optical comparator measurements, ultrasonic and laser light-scattering methods.

Results, FY71: (a) During this period the temperature dependence of ultrasonic wave velocities in polycrystalline Ne was determined up to the triple-point temperature. The results are a stringent test for current RHA ("renormalized harmonic approximation") theories of crystal dynamics; in a two-body potential model, one must go to second order of self-consistency to achieve agreement with experiment. (b) Our normal-beam equatorial x-ray diffractometer was used to measure Debye-Waller factors in crystalline Kr and Ne from 2.5K up to their respective triple points. Kr results are consistent with measurements of heat capacity, entropy, equilibrium vapor pressure, and phonon dispersion by means of slow neutrons, but disagree sharply with two previous experiments based upon the Mössbauer effect. Ne results in the range 15 - 25K show a large temperature variation as yet unexplained by theory. (c) A new high-precision x-ray diffractometer was designed and constructed for use on solid He. (d) Our P-V-T apparatus for precise x-ray studies up to $\Delta V/V \approx 20\%$ continued work on Ne and Ar. Measurements of thermal vacancy content of solid Ar were completed.

Results Expected, FY72: (a) Our unique high-precision x-ray diffractometer will be applied to studies on possible structural excitations in bcc ^3He at various densities and for similar studies on other solid phases of ^3He and ^4He . Useful precise P-V-T data should also be produced. There is high current activity by theoreticians in studying possible "crystal superfluidity," non-localized defects, and also unorthodox phonon interactions in such quantum crystals. (b) The same apparatus can also be adapted to study the controversial x-ray scattering by liquid solutions of ^3He and ^4He , if some other group does not produce satisfactory $S(q)$ data at small q soon. (c) X-ray Debye-Waller factors for crystal Ar and Xe will be determined, and the results compared to RHA theory for the entire family Ne, Ar, Kr, Xe. A considerable amount of computer calculation is required, including new work on thermal-diffuse-scattering corrections at very low temperatures. (d) The difficult direct measurement of thermal vacancy content in solid Xe should be completed. At issue is the question of non-central interactions in the solid state of such polarizable neutral atoms. (e) Some additional high-precision x-ray P-V-T studies on single crystals, in the range up to 1 kbar ($\approx \Delta V/V \approx 20\%$) will be undertaken. These solids provide a well-defined phonon system whose interaction can be varied systematically by changing V and by changing substance.

Results Expected, FY73 and Beyond: We expect to use our computer-controlled x-ray diffractometer to measure isodiffusion contours from the heavy crystals, to obtain second-order elastic constants. No reliable second-order constants are available at low temperature, where theories are most reliable, because of the practical problems of bonding crystal specimens to transducers (for pulse-echo ultrasonic work) or of windows (for Brillouin spectroscopy). Emphasis will be maintained upon search for an adequate model for real interatomic interactions in the condensed state. We expect the ^3He and ^4He crystal cryostat to be extended to studies over a wider variety of solid phases of these quantum crystals.

The physical characteristics of greatest interest are those whose explanation is obscured in ordinary solids by the presence of other interactions (such as Coulomb fields) or whose explanation is apparently involved with modern many-body theoretical techniques. Various such techniques have been developed in principle and appear promising; they have so far seen relatively little numerical application.

Publications, Calendar Year 1970:

R. O. Simmons

The Measurement of Thermal Expansion by Diffraction Methods

Proceedings of the 1968 Symposium on Thermal Expansion of Solids, edited

by R. F. Kirby, (U. S. National Bureau of Standards, Miscellaneous Publications, 1970)

COO-1198-600

R. O. Simmons

Use of fcc Metals as Internal Temperature Standards in X-Ray Diffraction

J. Appl. Phys. 41, 2235-2240 (1970)

COO-1198-607

D. S. Kupperman and R. O. Simmons

Sound Velocities in Polycrystalline Krypton

Journal of Physics C: Solid State Physics (submitted to)

COO-1198-752

M. S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

R. C. Windecker (R. O. Simmons, Adviser)

The X-Ray Debye Temperature of Solid Krypton

October 1970

D. S. Kupperman (R. O. Simmons, Adviser)

The Velocity of Sound in Solid Krypton

October 1970

Nuclear Magnetic Resonance in Solids

Principal Investigator: Charles P. Slichter, Ph.D., Professor of Physics;
Member, Center for Advanced Study

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Charles P. Slichter, Professor
David V. Lang, Research Assistant Professor

Junior Staff: James B. Boyce, Research Assistant
David Chan-wai Lo, Research Assistant
William J. Meyer, Research Assistant
Thomas Stakelon, Teaching Assistant
Lawrence R. Whalley, Research Assistant

Object: (1) Study of Dilute Magnetic Systems. One of the most basic problems of solid state physics is the circumstances under which magnetism occurs. For this reason there is great theoretical and experimental interest in systems of dilute amounts of transition metal atoms (such as Fe) which one conventionally thinks of as magnetic in nonmagnetic host metals (such as Cu, Al, or Ag). The interesting feature of such systems is that under some circumstances (e.g., Mn in Cu at room temperature) the "magnetic atom" acts magnetic, but in other cases (e.g., Mn in Al at room temperature, or Mn in Cu at very low temperatures) it is non-magnetic. Theoretical treatments attempt to calculate the electronic wave function of the impurity-conduction electron system. They lead to predictions of the magnetic and electric couplings of nuclei near to the impurity. We are attempting to measure these couplings by nuclear magnetic resonance studies of Cu-based alloys.

(2) Electric-Magnetic Double Resonance. Our double resonance experiments on the sodium resonance in NaCl containing small amounts of Ag detected the Na at the 100 position relative to the Ag, but could not find the 110 sodiums. The hypothesis (supported by theory) is that the quadrupole coupling is too small to split the signal from the main line (of the Na nuclei far from the Ag) so that the resonance is obscured by the much stronger main line absorption.

It appears theoretically possible to use a double resonance method in which one frequency goes with an applied electric field tuned nearly at twice the nuclear resonance frequency (a forbidden magnetic transition) to observe such small quadrupole couplings obscured by normal techniques. Preliminary results suggest the method works, though we cannot yet be sure. If it does it will provide an important way to resolve otherwise unresolvable resonance lines.

Results, FY71: (1) Dilute Magnetic Systems. Apparatus for making alloys of Cu containing Fe, Cr, Co, Ni, Mn has been completed and alloys of 0.5% (or smaller) concentrations have been prepared. Revamping of three NMR rigs to do: (i) steady-state strong field resonance, (ii) pulsed strong-field resonance, (iii) field-cycled double resonance has been completed (or nearly so) in order to achieve the high sensitivity necessary for useful work on magnetic alloy systems. The sensitivity has been calibrated using dilute alloys of Al with Mg and Zn.

(2) Electric-Magnetic Double Resonance. Apparatus development has continued to improve ease of operation of this very difficult experiment and to eliminate spurious instrumental effects. We believe, from crystal orientation studies, that we have demonstrated the existence of the effect.

Results Expected, FY72: (1) Dilute Magnetic Systems. We hope to determine the electric and magnetic couplings at Cu nuclei near impurities for one or more of the systems Cu: Mn, Fe, Co, Cr, Ni.

(2) Electric-Magnetic Double Resonance. We hope to demonstrate conclusively the validity of the scheme by measuring the quadrupole coupling of the Na atom nearest to the Ag impurity in the NaCl: Ag system. This coupling has not previously been capable of being measured since with conventional means the resonance line is obscured by a much stronger resonance.

Results Expected, FY73 and Beyond: Dilute Magnetic Systems. We expect to continue the experimental study of selected host-impurity systems, with emphasis on as broad a variation of the significant parameters as possible (temperature, magnetic field, impurity species, impurity concentration, host species). Utilization of the MRL superconducting magnet facility should give important increases in sensitivity, important for going to samples of low concentration to verify that the coupling between impurities can be neglected.

Publications, Calendar Year 1970:

P. R. Spencer, H. D. Schmid, and C. P. Slichter
Sensitivity of Nuclear Magnetic Double Resonance
Phys. Rev. B 1, 2989-3001 (1970)
COO-1198-654

E. A. S. Lewis
Heat Capacity of Gadolinium Near the Curie Point
Phys. Rev. B 1, 4368-4377 (1970)
COO-1198-678

J. R. Asik, M. A. Ball, E. K. Cornell, and C. P. Slichter
Spin-Flip Scattering of Electrons in Metals
Proceedings of International Symposium on Electron and Nuclear
Magnetic Resonance, (Plenum Press, 1970) pp. 187-195
COO-1198-741

C. P. Slichter, H. Seiden, P. Schwartz, and G. Fredericks
The Order-Disorder Transition in NH_4Cl : I. Phenomenological Theory
Phys. Rev. (submitted to)
COO-1198-734

G. E. Fredericks
The Order-Disorder Transition in NH_4Cl : II. Thermal Expansion
Phys. Rev. (submitted to)
COO-1198-740

P. Schwartz
The Order-Disorder Transition in NH_4Cl : III. Specific Heat
Phys. Rev. (submitted to)
COO-1198-742

M.S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

None

Physics of Refractory Materials

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

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: Wendell S. Williams, Professor

Junior Staff: Joseph G. Baldoni, Research Assistant
Lance Breger, Research Assistant
George H. Emmons, Research Assistant
Alvin P. Gerk, Research Assistant
Subhash Kulkarni, University Fellow
Lawrence W. Shacklette, Research Assistant

Object: The transition-metal carbides are among the most refractory materials known. However, their great hardness decreases rapidly with increasing temperature. For successful structural application in reactors, jet engines or other extreme environments, the softening must be understood and controlled. A study of dislocation motion at high temperatures at stresses below the yield stress is in progress to help in elucidating the details of plastic deformation in the carbides. The large deviations from stoichiometry in these compounds influence all their physical properties. In one system, vanadium-carbon, the usual random arrangement of carbon vacancies becomes ordered below 1250°C to form a superlattice. The influence of ordering on transport properties, thermal properties and mechanical properties is being studied and may lead to new design choices for this class of refractory materials.

Results, FY71: Apparatus for measuring the electrical resistivity of a metallic solid at temperatures up to 1400°C was constructed. Resistivity versus temperature data were taken for vanadium carbide single crystals over this temperature range and, with other apparatus, down to 2°K. The phase change from disordered carbon vacancies to a superlattice arrangement reported in the literature was verified, the first precise determination of the transition temperature was made, the order of the transition was determined and found to agree with our prediction (1st order), and the importance of random vacancies as scattering centers for conduction electrons in these non-stoichiometric solids was demonstrated. Other apparatus for determining the change in heat capacity through the order-disorder transition by an AC current technique was constructed. Apparatus for studying internal friction in carbides at temperatures up to 1100°C by ultrasonic techniques was completed. Apparatus for pulling thin tensile specimens for dislocation velocity measurements at high temperatures was completed. Preliminary studies of dislocations with the Lang x-ray camera were conducted.

Results, FY72: A theoretical analysis of the role of vacancies in controlling the electrical resistivity and electronic component of the thermal conductivity of non-stoichiometric metallic compounds will be conducted. The data presently being collected on ordered and disordered vanadium carbide and previous measurements of residual resistivity and dependence of resistivity on metal/non-metal ratio will then be interpreted. The heat capacity change associated with the V_6C_5 $VC_{0.83}$ transition will be measured and compared to our predicted curve. High temperature internal friction data on TiC, ZrC, NbC and TaC will be obtained and interpreted in terms of dislocation kink motion. Lang x-ray photographs of dislocations in the carbides and in Ge will be taken and studied. High quality single crystals of WC will be produced for studies of electrical and mechanical properties.

Results Expected, FY73 and Beyond: The relation between electronic and mechanical properties of covalently bonded solids will continue to be the theme of the work. The eventual goal is the design of super-hard materials.

Publications, Calendar Year 1970:

L. G. Radosevich and W. S. Williams
Thermal Conductivity of Transition Metal Carbides
J. Am. Ceram. Soc. 53, 30-33 (1970)
COO-1198-628

D. L. Kohlstedt, W. S. Williams, and J. B. Woodhouse
Chemical Diffusion in Titanium Carbide Crystals
J. Appl. Phys. 41, 4476-4484 (1970)
COO-1198-709

D. L. Kohlstedt and W. S. Williams
Investigation of Charge Distribution in Titanium Carbide Using Electromigration
Phys. Rev. (submitted to)
COO-1198-708

W. S. Williams
Transition-Metal Carbides
Progress in Solid State Chemistry (submitted to)
COO-1198-772

M.S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

D. L. Kohlstedt (W. S. Williams, Adviser)
Electromigration and Chemical Diffusion in Titanium Carbide
February 1970

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

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

Supporting Agency: U. S. Atomic Energy Commission

Senior Staff: James S. Koehler, Professor
Edward J. Gutman, Research Associate
Kurt W. Schroeder, Research Associate

Junior Staff: Ronald Berliner, Research Assistant
Bruce S. Brown, Research Assistant
Ronald E. McKeighen, Research Assistant

Object: Defects in metals and in semiconductors, particularly those introduced by irradiation will be studied. Irradiation of semiconductors at temperatures down to 0.3°K will be carried out. Possible non-thermal defect migration will be investigated. The symmetry, thermal migration energy, charge states, energy levels and other properties of the defects will be examined both experimentally and theoretically. The electron diffraction of defects will be measured. Channeling and high temperature damage will be studied.

Results, FY71: The measurements of the anomalous X ray transmission of copper before and after electron irradiation at 15°K is being repeated. It is anticipated that enough additional data to determine the geometrical configuration of the interstitial in copper will be obtained.

A theoretical calculation of the structure and properties of the neutral interstitial in diamond is being made. Thus far calculations have assumed the tetrahedral structure. Jahn-Teller distortion will be calculated.

Channeling studies using 1 MeV protons on silver single crystals have been undertaken. At present the transmission of unirradiated crystals is affected by some defect. Either a surface oxide or a contaminant could be responsible. Gold crystals will be used to decide.

The channeling apparatus can be used to determine the crystal potential which steers the protons. Theoretical studies are being made to determine the most accurate way to measure the crystal potential.

Apparatus to detect the electron scattering by defects in solids is being constructed. The apparatus will use 100 KeV electrons. Initially quenched gold single crystals will be studied. The evolution of vacancy clusters will be followed.

Results Expected, FY72: We intend to produce damage in silicon and in germanium using either thermal neutrons or 1 KeV neutrons. The irradiation will be done at 4.5°K or if necessary at 1.6°K. These experiments are aimed at finding out whether the ionization associated with electron irradiation produces non-thermal migration.

We also intend to determine the geometrical structure of interstitials in gold and in silicon and germanium. Such measurements will either be made by electron diffraction or by anomalous x-ray transmission.

A theoretical treatment of channeling including both planar and axial channeling will be given and measurements of the crystal potential will be made.

Results Expected, FY73 and Beyond: A heavy ion source for the Van de Graaff accelerator will be constructed. Interstitials will be introduced by channeling at 10°K into single crystals of gold, silicon, and germanium. The properties of the interstitials will be studied by ac hopping conductivity by thermal and radiation annealing and by other methods.

High temperature electron damage in gold will be studied to determine whether impurities nucleate the damage and to decide whether the clusters are arranged at random or not.

Publications, Calendar Year 1970:

Y. Shimomura

Defects Observed in Electron Irradiated Pure Silver by Electron Microscopy
J. Appl. Phys. 41, 749-759 (1970)
COO-1198-652

L. S. Edelheit, J. C. North, J. G. Ring, J. S. Koehler, and F. W. Young, Jr.
Effect of Point Defects in Copper on the Anomalous Transmission of X-Rays
Phys. Rev. B 2, 2913-2923 (1970)
COO-1198-675

J. S. Koehler

An Attempt to Design a Strong Solid
Phys. Rev. B 2, 547-551 (1970)
COO-1198-703

K. Schroeder

Influence of Trapping at Impurity Atoms on Diffusion Annealing of Metals
After Electron Irradiation
Radiation Effects 5, 255-263 (1970)
COO-1198-728

K. Izui, J. Elston, and J. S. Koehler
Effect of High Temperature Electron Irradiation in Gold
Radiation Effects (submitted to)
COO-1198-729

M. Doyama, J. S. Koehler, Y. N. Lwin, E. A. Ryan, and D. G. Shaw
Annealing Study of Dilute Aluminum Alloys Electron Irradiated at Liquid
Nitrogen Temperature
Phys. Rev. (submitted to)
COO-1198-767

W. D. Hyatt and J. S. Koehler
Helium Temperature Annealing of Electron-Irradiated N Type Germanium
Phys. Rev. (submitted to)
COO-1198-768

M.S. Theses, Calendar Year 1970:

None

Ph.D. Theses, Calendar Year 1970:

L. S. Edelheit (J. S. Koehler, Adviser)
The Effect of Point Defects in Copper on the Anomalous Transmission of
X-rays
February 1970

W. D. Hyatt (J. S. Koehler, Adviser)
Low Temperature Annealing of Electron Irradiated Germanium
June 1970

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- L. S. Edelheit (J. S. Koehler, Professor of Physics, Adviser), "The Effect of Point Defects in Copper on the Anomalous Transmission of X-rays," February 1970.
- D. G. Franklin, III (H. K. Birnbaum, Professor of Physical Metallurgy, Adviser), "Anelastic Study of Divacancy Damping in Gold," June 1970.
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- D. S. Kupperman (R. O. Simmons, Professor of Physics, Adviser), "The Velocity of Sound in Solid Krypton," October 1970.
- H. Ledbetter (C. M. Wayman, Professor of Metallurgical Engineering, Adviser), "The Crystal Structure and Martensitic Transformation in Near Equiatomic AuCd," February 1970.
- E. A. Merriman (C. M. Wayman, Professor of Metallurgical Engineering, Adviser), "Superplastic Deformation in a Eutectic Alloy of Cadmium and Zinc," February 1970.
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