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METALS and CERAMICS DIVISION

Annual Progress Report

Period Ending June 30, 1974

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METALS AND CERAMICS DIVISION ANNUAL PROGRESS REPORT FOR PERIOD ENDING JUNE 30, 1974

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OCTOBER 1974

OAK RIDGE NATIONAL LABORATORY
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FOREWORD

The technical content of this Metals and Ceramics Division annual progress report is limited to those activities sponsored by the AEC Division of Physical Research. Reports dealing with progress of various activities sponsored by the AEC Division of Reactor Research and Development are distributed under the following TID-4500 categories: UC-77 — Gas-Cooled Reactor Technology, UC-78 — Light-Water Reactor Technology, UC-79 — Liquid Metal Fast Breeder Reactor, and UC-80 — General Reactor Technology.

The following progress reports have been issued covering work done during the reporting period: *Fuels and Materials Development Program Quart. Progr. Rep. Sept. 30, 1973,* ORNL-TM-4405; ibid., *Dec. 31, 1973,* ORNL-TM-4524; ibid., *Mar. 31, 1974,* ORNL-TM-4620; and ibid., *June 30, 1974,* ORNL-TM-4688. *Mechanical Properties Test Data for Structural Materials Quart. Progr. Rep. Oct. 31, 1973,* ORNL-4936; ibid., *Jan. 31, 1974,* ORNL-4948; ibid., *April 30, 1973,* ORNL-4963; and ibid, *July 31, 1974,* ORNL-4998. *Gas-Cooled Reactor Programs Annu. Progr. Rep. Dec. 31, 1973,* ORNL-4975. *Semiannual Progress Report Irradiation Effect on Reactor Structural Materials Jan. 31, 1974,* HEDL-TME-74-9.

While the narrative contents of this report cover only the activities sponsored by the AEC Division of Physical Research, the publications, patents, and papers presented at scientific meetings listed at the end of the report pertain to the total activity under way in the Metals and Ceramics Division. A divisional organization chart is also included.

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SUMMARY

PART 1. STRUCTURE OF MATERIALS

The research described in this Section emphasizes the relationships among the structure of materials and their various properties. Included are theoretical studies, x-ray diffraction work, fundamental ceramics research, and crystal physics investigations.

To understand radiation damage effects in hexagonal crystals, we analyzed the elastic interactions between small defects and defect clusters, including an examination of void formation in zirconium in the presence of thermal fluctuations, hydrostatic pressure, and changes in other metallurgical variables. The effect of the discreteness of a solid lattice on diffusion is studied by solving difference equations for the hopping transport.

We continue to study electronic states in disordered solids by developing theories that go beyond the single-site approximation. We have applied the coherent potential approximation to the calculation of the electronic states in substoichiometric PdH and have shown that the technique can be extended to treat substoichiometric compounds in general. We are comparing theories of liquid metals in order to select the best approach to calculating their electronic properties.

We have applied our band theory technique in which wave functions calculated for a muffin-tin potential by the Korringa-Kohn-Rostoker method are used as trial functions for discrete variational method calculations on the related problem using a non-muffin-tin potential. We calculated the non-muffin-tin effects in the band structure of Rb, Nb, and Pd.

We have applied multiple scattering methods to clusters of atoms for treating electronic states of disordered systems and bonding.

X-ray diffraction work has focused on three major areas: (1) development of mathematical procedures for analysis of diffraction data from short-range structures, (2) optimization of experimental techniques and analyses of small-angle scattering data, and (3) structural studies of non-stoichiometric boron carbides. In the first area we have found closed-form solutions for intensity distributions from certain models of the omega structure formed in bcc alloys. In the second we have provided a useful calibration of polyethylene as an absolute intensity standard, correlated positron-annihilation and small-angle scattering data from neutron irradiated aluminum, and accurately measured the zero-angle scattering of water. In the third area we have determined the nature of the structural defect produced by carbon depletion of boron carbide and developed a general structural interpretation of the boron-carbon equilibrium diagram.

Transmission electron microscopy was used to study the microstructure of a series of deformed polycrystalline UO_2 specimens and fuel-sphere pyrolytic carbon coatings. Absorption spectroscopy has been used to study the optical spectra of transition metal ions present in crystalline compounds. A new program has been initiated for the study of thermodynamic, chemical, and structural properties of transition metal tellurides.

Crystals of a number of high-melting, refractory materials were grown. We have induction-melted ThO_2-tungsten mixtures and directionally solidified the eutectic that consisted of tungsten fibers in the ThO_2 matrix. For both binary and ternary metal oxide-metal systems we are investigating

the requirements for steady-state growth and the morphological dependence on composition and temperature gradient. Studies on the origin of the 3400-cm^{-1}-region infrared bands of synthetic and natural quartz led to the conclusion that the impurity perturbation of the OH vibration was strongly influenced by the impurity site and that probably the tunnel sites were more important than interstitial or lattice sites. EPR studies of transuranic ions in single-crystal hosts are reported. Czochralski growth of single-crystal akermanite ($Ca_2MgSi_2O_7$) and gehlenite ($Ca_2Al_2SiO_7$) is described. The optical absorption spectra of nominally pure and manganese-doped Czochralski-grown single crystals of Mg_2SiO_4 were measured from 1 to 7.5 eV. Radioluminescence of $SrCl_2$ doped with Cm^{3+} and Es^{2+} was measured.

PART 2. PHYSICAL PROPERTIES AND TRANSPORT PHENOMENA

Research areas included in this section are alloy oxidation, diffusion in solids, physical property studies, and the evaluation of metallurgical variables for superconducting materials.

Studies of the mechanisms of oxidation of alloys were continued. The defect concentrations and diffusion coefficients for oxide films on uranium-base alloys were investigated. The oxidation behavior of tantalum-base alloys was characterized in terms of internal oxidation, stress generation, and oxidation kinetics; in a related study the diffusion rates of hafnium and oxygen in tantalum were also measured.

A computer code was written and used in the examination of interdiffusion and intrinsic diffusion information for binary alloy systems. Atomic mobilities and vacancy wind effects were theoretically analyzed for previously obtained data on silver-zinc-cadmium solid solutions. Similar data were obtained and analyzed for vanadium-titanium solid solutions. Hafnium tracer diffusion in monocrystalline tantalum was examined in the range 1200 to 2000°C.

Work continued on techniques to obtain more accurate physical property results on solids. The capacity of the Computer Operated Data Acquisition System (CODAS) was expanded. The accuracy of thermal expansion coefficient data produced by a dilatometer operated by CODAS is now better than $\pm 1 \times 10^{-7}$/K from 300 to 1000 K. The Lorenz number and lattice conductivity values for pure molybdenum were derived from data on molybdenum-niobium alloys and compared favorably with our previous work. Measurements on pure chromium were extended to 1200 K. Dense polycrystalline samples of cesium and rubidium halides were prepared for transport studies to high reduced temperatures (T/Θ_D). Specific heat studies were completed on Ni_3Fe, continued on aluminum, and initiated on technetium. Thermal expansion data were obtained on monoclinic and cubic Eu_2O_3 and single-crystal and natural NaCl.

Fluxoid pinning in niobium containing small amounts of either yttrium or gadolinium was studied by dc magnetization measurements. Superconducting transition temperatures of chemically vapor deposited tungsten-rhenium alloys were measured, and the increases over arc-melted material were correlated with the formation of an A-15 phase or the presence of impurities. High critical current densities were found in thick $Nb_{12}Al_3Ge$ superconducting deposits made by high-rate sputtering. New equipment was developed for ac loss measurements and for critical current measurements in superconductors under mechanical stress.

PART 3. DEFORMATION AND MECHANICAL PROPERTIES

The relationship between the structure of materials and their physical and mechanical properties was traced in several projects. The effect of oxygen absorption on the structure of a Ta-W-Hf alloy was investigated by transmission electron microscopy, and the changes observed were related to the corresponding changes in the fracture properties of the alloy. The thermodynamic properties of solid solution alloys were considered in terms of the possible temperature dependence of the entropy or heat of mixing of the alloys. The short- and long-range ordered structures of Ni_4Mo were studied by x-ray techniques. Low-temperature phase transformation mechanisms in a U-Nb-Zr alloy were studied by x-ray diffraction and dilatometry. Crystal reorientation by mechanical twinning was shown to be an operative deformation mechanism in the early stages of rolling of this alloy. The cause of texture gradients through the thickness in cold-rolled niobium plates was correlated with the geometry of the zone of deformation in rolling.

PART 4. RADIATION EFFECTS

The Radiation Effects Program is focused on the development of improved materials for reactor applications. The effort includes both studies of simple model materials aimed at uncovering mechanisms and basic processes occurring during particle irradiation as well as work on complex alloys directed at defining properties of materials in potential reactor systems.

Effects of specimen purity on the formation of dislocation loops in aluminum by electron irradiation are described, and factors influencing the long-range alignment or ordering of loops in neutron-irradiated nickel and aluminum are presented. Both swelling and postirradiation mechanical properties of the 6061-T6 aluminum alloy used in the High Flux Isotope Reactor (HFIR) hydraulic tube are shown to be largely determined by silicon formed by a thermal neutron reaction. Effects of high helium concentrations and displacement levels approximating those expected in fusion reactors have been investigated in stainless steel irradiated in HFIR, where the helium is generated by a two-step reaction of nickel with thermal neutrons. Swelling is significantly higher and strengthening and ductility lower than for irradiations in fast reactors, where much lower levels of helium are formed. Compositional changes and combinations of deformation and annealing treatments have been identified that both decrease swelling and increase postirradiation ductility in stainless steel irradiated in a fast reactor. Segregation of helium and minor impurities to grain boundaries of irradiated stainless steel has been demonstrated by fracturing specimens in an Auger electron spectroscopy facility equipped with a residual gas analyzer. Analysis of intergranular corrosion of stainless steel fuel claddings indicates a common rate-limiting mechanism for a range of irradiation conditions. Careful examination of electron diffraction patterns from "TiC" precipitate particles formed during neutron irradiation has revealed an orthorhombic rather than cubic symmetry, suggesting a structure similar to V_2C. A statistical analysis of the size distribution of voids in irradiated stainless steel implies the possibility that the mechanism controlling void growth changes as the voids grow

larger. The absence of voids in the vicinity of pre-existing TiC precipitate particles in titanium-stabilized type 316 stainless steel suggests the possibility of controlling swelling by providing a uniform distribution of the precipitate particles.

PART 1. STRUCTURE OF MATERIALS

1. THEORETICAL RESEARCH

J. S. Faulkner

We have contributed significantly to the development of the anisotropic elasticity theory of dislocations, the Korringa-Kohn-Rostoker and discrete variational method approaches for calculating electronic states in ordered solids, the coherent potential approximation for obtaining the electronic states in disordered solids, and cluster methods for treating a range of problems in the theory of condensed matter. We are using these techniques to gain an understanding of the properties of technologically interesting materials in their normal states and after radiation damage.

Studies on Disordered Systems Using Clusters of Muffin-Tin Potentials[1]

J. S. Faulkner, W. H. Butler, G. S. Painter, and W. A. Coghlan

Calculations of the electronic states of clusters of atoms have been carried out in recent years for representing the states of ordered or disordered solids. We propose a boundary condition to be put on the cluster that should simulate the effect of the system outside the cluster better than the ones presently in use.

Non-Muffin-Tin Effects in the 4*d* Transition Metals Rb, Nb, and Pd (ref. 2)

G. S. Painter, J. S. Faulkner, and G. M. Stocks[3]

While the effects of corrections to the muffin-tin model of the crystal potential have been investigated fairly thoroughly for various insulators and semiconductors, relatively few complete calculations have been done for transition metals. The Hartree-Fock-Slater muffin-tin approximate potential is usually adequate for characterization of the general properties of free-electron metals. In the transition-metals series, however, where the Fermi energy typically lies in a partially filled *d* band, it is recognized that small nonuniform energy shifts can appreciably alter the properties, dependent upon the details of the

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[1]Abstracted from *J. Phys. (Paris) Colloq.* 4: 85–87 (1974).

[2]Abstract of *Phys. Rev. B* 9: 2448–56 (1974).

[3]Now at the Department of Physics, The University of Bristol, Bristol, England.

topology of the Fermi surface. Here, we report the application of the recently proposed combined Korringa-Kohn-Rostoker discrete variational method for several crystals representative of the *4d* transition-metal series. Results are analyzed directly in terms of the Bloch wave functions, and various trends and general features of non-muffin-tin effects in the transition metals are determined.

Electronic States of Substoichiometric Compounds[4]

J. S. Faulkner, G. S. Painter, and W. H. Butler

Transition metal compounds form an interesting class of solids, but the crystals used in experiments rarely approximate the ordered form that is assumed in ordinary band theory calculations. A type of disorder that frequently occurs is substoichiometry, which can be described to a first approximation as the situation in which all of the metal ions are on their proper lattice sites, but many of the anion sites are vacant. Such systems may be treated by a straightforward extension of the coherent potential approximation in the tight-binding form. The parameters for the CPA are obtained from a Slater-Koster interpolation fit to the results of band structure calculations that make use of a very flexible KKR program that we recently put together.

Short-Range Order and the Electronic Structure of a One-Dimensional Liquid Metal[4]

W. Butler, H. K. Peterson,[5] and L. Schwartz[5]

The energy spectrum of a one-dimensional array of delta function potentials is studied as a function of the degree of short-range order in the arrangement of the scatterers. The integrated density of states $N(E)$ is evaluated numerically, and the results are used to compare alternate formalisms. In particular, we consider the quasi-crystalline approximation (QCA) and the more recent approaches of Gyorffy[6] and Schwartz and Ehrenreich.[7] The machine calculations show that, as the short-range order is increased $N(E)$ develops steplike structure similar to that associated with the existence of band gaps in the perfect crystal. Interestingly, we find that the simplest of the approximation schemes, the QCA, reproduces the essential features of this structure, and in addition, yields a reasonable overall picture of the electronic spectrum.

---

[4]Abstract of a talk presented at the March Meeting of the American Physical Society, Philadelphia, Pa., March 25–28, 1974.

[5]Department of Physics, Brandeis University, Waltham, Mass.

[6]B. L. Gyorffy, *Phys. Rev. B* 1: 3290 (1970).

[7]L. Schwartz and H. Ehrenreich, *Ann. Phys. (N. Y.)* 64: 100 (1971).

The Electronic Structure of Alloys from Cluster Calculations[8]

W. H. Butler

The Coherent Potential Approximation[9] is an excellent single-site approximation. It is inadequate, however, for treating the details of the density of states in the strong scattering regime. In this regime a cluster approach, in which one treats the local environment of an atom exactly and the more removed region in an approximate but self-consistent fashion, seems most reasonable. This program has so far been carried out only for model systems. We also show how one can use clusters of muffin-tin potentials to determine qualitatively the nature of the electronic structure of disordered transition metal alloys.

Multiple Scattering Cluster Calculations of the Densities of States of Some Transition Metal Compounds[4]

G. S. Painter, J. S. Faulkner, W. A. Coghlan, and W. H. Butler

The multiple scattering formulation of Lloyd[10] for calculating the density of states for a cluster of muffin-tin scatterers has been applied to several compounds. A summary is given of investigations in progress to characterize the bonding mechanisms in nonstoichiometric transition metal carbides and oxides. Studies of the influence of interstitial oxygen on the electronic structure of metallic niobium will also be reviewed. The results of this work are further analyzed by the use of comparative LCAO calculations for the cluster of atoms.

Self-Consistent Cluster Theory of Disordered Alloys[11]

W. H. Butler

Several techniques for extending the single-site coherent-potential approximation (CPA) to account for particular local configurations of atoms are evaluated. We show that attempting to define a site-diagonal medium by requiring consistency between the Green's function evaluated at the center of the cluster and the external medium may lead to unphysical results. If, however, one requires consistency between a site at the boundary and the medium, one can obtain (at least in one dimension) a site-diagonal medium that reproduces the cellular CPA exactly.

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[8]Invited paper presented at the March Meeting of the American Physical Society, Philadelphia, Pa., March 25–28, 1974.

[9]P. Soven, *Phys. Rev.* 156: 809 (1967).

[10]P. Lloyd, *Proc. Phys. Soc. London* 90: 809 (1967).

[11]Abstracted from *Phys. Rev. B* 8: 4499–4509 (1973).

Difference Equation Solutions for Non-Steady-State Diffusion of Charged Particles in Solids[12]

W. H. Butler and A. T. Fromhold, Jr.[13]

The hopping transport of charged particles through a solid is described by means of difference equations based on the concept of classical thermal motion over discrete energy barriers. Homogeneous electric fields and concentration gradients are considered to be the driving forces for transport. Transient and steady-state currents are derived, and the concentration profiles are obtained for the mobile charged defect species. For the case of a slab geometry the discreteness of the potential barriers leads to a nonlinear dependence of current on voltage in the high electric field limit, with a more rapid increase of current with voltage than would be expected from an extrapolation of the low-field linear dependence. The field-dependent relaxation of a non-steady-state defect concentration profile to the corresponding steady-state profile can be nearly exponential in the limit of large fields. Tracer distributions for the cases of semi-infinite and unbounded diffusion mediums are likewise affected appreciably in the high field limit. The velocity of the peak is increased over that obtained by linear extrapolation from the low field limit. We conclude that the combination of high electric fields with the natural microscopic discreteness of a solid-state diffusion medium can result in readily observable nonlinear electric field effects that increase approximately exponentially with the atomic separation distances of the discrete barriers in hopping transport. Some of this nonlinear behavior can be retained in differential equations derived from the difference equations by means of Taylor series expansions of the carrier concentration with respect to position.

The Electronic Structure of Disordered Transition Metal Alloys[14]

W. H. Butler

Theories of disordered alloys are discussed, including the Rigid Band Model, the Virtual Crystal Approximation, the Virtual Bound State Model, and the Coherent Potential Approximation (CPA). We shall show that the CPA is a very good single-site approximation, which agrees with the other approximations in the appropriate limits. We shall discuss the results of applying the CPA to alloy systems. We shall show that both

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[12]Abstracted from *J. Phys. Chem. Solids* 35: 1099–1114 (1974).

[13]Physics Department, Auburn University, Auburn, Alabama.

[14]Abstract of an invited talk presented at the meeting of the Southeastern Section of the American Physical Society, Winston-Salem, North Carolina, November 10–12, 1974.

rigid band behavior and virtual bound state behavior occur in transition metal alloys. Finally we shall show how one may predict qualitatively (without doing a full CPA calculation) the nature of the density of states of a disordered alloy.

Elastic Interaction of Small Defects and Defect Clusters in Hexagonal Crystals[15]

M. H. Yoo

An analysis is given of the elastic interaction between small defects and/or defect clusters in hexagonal crystals. Simple explicit expressions for the interaction energy are obtained directly from the Green's function given by Kröner. Some typical calculated results are presented for various combinations of axially symmetric defects — e.g., a pair of point defects, a pair of dislocation loops, and a point defect and a dislocation loop. Applications to a study of Frenkel defect recombination processes in irradiated hexagonal crystals are discussed.

Suppression of Void Formation in Zirconium[16]

M. H. Yoo

An analysis is given of the effects of anisotropic thermal expansion coefficients and elastic constants on radiation-induced void formation in zirconium and other hexagonal metals. Upper bound values of thermal stress in polycrystalline metals are estimated. A cyclic temperature fluctuation at high irradiation temperature causes changes in internal stresses. These changes suppress nucleation and growth of voids as a result of the enhanced recombination of point defects at grain boundaries and also retard void growth due to the induced self-stress field. Such effects are more pronounced in highly anisotropic zirconium and magnesium. Effects of hydrostatic pressure on void formation are also investigated. Other metallurgical variables for void formation are discussed, and an experiment to test the current concept is suggested.

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[15]Abstracted from *Phys. Status Solidi (b)* 61: 411–18 (1974).

[16]Abstract of paper published in *Zirconium in Nuclear Applications, Amer. Soc. Test. Mater. Spec. Tech. Publ.* 551, pp. 297–307, American Society for Testing and Materials, Philadelphia, 1974.

Elastic Interaction and Recombination Rate of Frenkel Defects in Hexagonal Crystals[17]

M. H. Yoo

An analytic expression for the elastic interaction energy between a pair of point defects in an anisotropic hexagonal medium is obtained by the Green's function method. The parameters involved in the expression are the strength and the symmetry of each point defect and the elastic constants of the medium. Effects of the elastic interaction on the direct recombination rate of Frenkel defects are studied by seeking a steady-state solution of the diffusion equation for highly mobile interstitials around a vacancy. Temperature dependence and possible roles of various impurity atoms are discussed. The degree of anisotropy in the strain field and the diffusion coefficient of interstitials alter the recombination rate most significantly.

Pressure-Induced Superconductivity in Cesium[18]

G. M. Stocks,[3] G. D. Gaspari,[19] and B. L. Gyorffy[3]

Pressure-induced variations in the electron-phonon coupling constant λ of cesium are calculated from first principles. We find that λ increases with pressure and predict that cesium should be superconducting with an observable transition temperature T_c at 35 kbar.

2. X-RAY DIFFRACTION

H. L. Yakel

The following abstracts and research summaries present a concise picture of the activities of the Group during the period of this report.

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[17]Abstract of talk presented at the Spring Meeting of the Metallurgical Society of AIME, Pittsburgh, Pa., May 19–23, 1974.

[18]Abstract of a paper published in *J. Phys. F: Metal Phys.* 2: L-123–28 (1972).

[19]Department of Physics, University of California, Santa Cruz, California.

The Short-Range Structure of BCC Solid Solution Alloy of Titanium and of Zirconium

B. S. Borie and H. L. Yakel

Efforts to extend our understanding of the short-range structure of bcc solid solutions of titanium and of zirconium bearing the omega phase have continued. Our goal continues to be the development of a detailed interpretation of the diffuse neutron intensity in reciprocal space for such an alloy. Using a model for a completely transformed alloy, we had previously found that by introducing a certain characteristic fault in the omega structure we could reproduce the experimentally observed shifts of the diffuse omega Bragg maxima. However, under each of the sharp bcc reflections (characteristic of the untransformed material) there are more or less equiaxed diffuse maxima, which our model failed to generate.

We generalized our model to include untransformed material. The missing diffuse peaks now appear, though not with their experimentally observed relative strengths. This discovery, that the presence of untransformed material will cause the previously absent diffuse maxima, is not well understood.

To illuminate the meaning of this interesting and unexpected result, we have pursued several leads. First, we have examined in detail the special simplified form taken by the expression for the diffuse scattering at the sharp bcc maxima. The relative strengths of these maxima may be related to the details of how the transformed material is dispersed in the bcc matrix. If this is so, we will ultimately succeed in extracting from the intensity distribution in reciprocal space an additional parameter related to the transformation to complete a description of the short-range structure.

Our method for computing the diffuse intensity distribution from a specific atomic arrangement yields a convergent sum of complex quantities, which are functions of position in reciprocal space. Until now we have had to approximate it by an arbitrarily truncated termwise summation with the aid of the computer. We have found a way to represent the relevant sum in closed form. This result reduces computer time by an order of magnitude and eliminates all truncation errors in the computed intensity distributions.

Another possibility is that there may be contributions from interference effects among the variants of the system. For simplicity in all our previous calculations they have been omitted. We have developed a theory to account for these effects. We find that they are not negligible and that they probably contribute to both the diffuse part of the intensity distribution and the sharp bcc maxima. We have not yet developed the diffuse component of this recent result. However, the resultant contribution to the bcc reflections completes a description of their integrated intensities, allowing us to say from the measurements, within the terms of our model, what fraction of the crystal is untransformed. This had not previously been possible.

Calibration of Polyethylene (Lupolen) as a Wavelength-Independent Absolute Intensity Standard[20]

L. B. Shaffer[21] and R. W. Hendricks

The unified theory of absolute intensity measurements in small-angle x-ray scattering has been experimentally verified by determining for polyethylene (Lupolen) a calibration constant that is independent of collimation system and wavelength. Consistent values of this constant were determined for three x-ray wavelengths (Ag Kα, Mo Kα, and Cu Kα) by gas scattering and multiple-foil attenuator experiments. The agreement of the results implies that all the experimentally significant parameters involved in these absolute intensity calibrations have been identified. In addition, they show Lupolen to be a more general secondary standard than had been previously recognized.

A Pulse-Matching Method for Estimating the Gas Amplification Factor in Proportional Counters[22]

R. W. Hendricks

The gas amplification factor in a proportional counter may be estimated by a comparison technique using an oscilloscope. A known capacitor is put in parallel with the input from the detector at the first group of the preamplifier, and a unipolar output signal is generated by applying a voltage step from a precision pulse generator. This signal is then matched on the oscilloscope to a similar signal from the main amplifier, as created by a monoenergetic x-ray pulse falling on the counter.

To obtain the correct value for the gas amplification factor, the differences in rise-times between the charge input from the detector and the pulse generator must be taken into consideration. This correction is obtained from a calibration chart determined from a computer solution of the differential equations that describe the pulse shapes for the pulse generator and the proportional counter at various points in the preamplifier-amplifier system. The calculations assume (1) zero electron collection time in the detector, (2) a charge-sensitive low-noise preamplifier, and (3) a main amplifier having simple *RC* pulse-shaping networks. The errors introduced by a spread in the electron collection time are discussed. Experimental results that test and illustrate the use of the method are given.

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[20]Abstracted from *J. Appl. Cryst.* 7: 159–63 (1974).

[21]Visiting consultant on leave from Anderson College, Anderson, Ind.

[22]Abstracted from *Nucl. Instr. Methods* 106: 579–88 (1973).

Positron-Annihilation Studies of Voids in Neutron-Irradiated Aluminum Single Crystals[23]

W. Triftshäuser,[24] J. D. McGervey,[25] and R. W. Hendricks

We measured the angular correlation of positron-annihilation radiation from single crystals of aluminum in which voids had been induced by neutron irradiation. The angular-correlation curve is narrower than that for aluminum containing vacancies. The data fit well a curve computed on the assumption that the positrons are annihilated at the void surfaces. After the sample was heated to 325°C the angular-correlation curve became identical to that of unirradiated aluminum.

Misalignment of Flux Lines in a Type II Superconductor Studied by Neutron Small-Angle Diffraction[26]

G. Lippmann,[24] J. Schelten,[24] R. W. Hendricks, and W. Schmatz[24]

The mutual misalignment of flux lines was measured by neutron small-angle diffraction with the incident beam parallel to the flux lines. The resolution function of the apparatus for this new geometry was evaluated, and various physical contributions to the broadening of the observed diffraction peaks are discussed. The resulting misalignment angles were largest (≈4°) at low flux densities and decreased to a constant value (8′ ± 5′) at flux densities larger than $0.5H_{c2}$.

X-Ray Zero-Angle Scattering Cross Section of Water[27]

R. W. Hendricks, P. G. Mardon,[28] and L. B. Shaffer[21]

The absolute x-ray scattering cross section for water has been determined at small angles for specimen thicknesses t in the range $0.5 \leq \mu t \leq 6.2$ at 20°C and in the temperature range 6 to 75°C for $\mu t = 1.0$ (μ = absorption coefficient). The data determined as a function of specimen thickness are in agreement with a new determination of second-order

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[23]Abstracted from *Phys. Rev. B* 9: 3321–24 (1974).

[24]Institut für Festkörperforschung, Kernforschungsanlage, Jülich, Germany.

[25]Case Western Reserve University, Cleveland, Ohio.

[26]Abstracted from *Phys. Status Solidi (b)* 58: 633–41 (1973).

[27]Abstracted from paper accepted for publication in *Journal of Chemical Physics*.

[28]Process Technology Division, Atomic Energy Research Establishment, Harwell, England.

multiple scattering. After correction for multiple scattering, the temperature-dependent results are in good agreement with the zero-angle scattering cross section computed from fluctuation theory. This result precludes certain cluster models of the structure of liquid water.

The Crystal Structure of a Boron-Rich Boron Carbide[29]

H. L. Yakel

Structural changes accompanying extreme boron enrichment of boron carbide were deduced by use of Mo Kα x-ray diffraction data from a twinned aggregate containing about 9 at. % C. The crystal system is rhombohedral, space group $R\bar{3}m$, as for B_4C, but rhombohedral cell parameters show significant expansions compared with those of material with 20 at. % C. Fourier, difference Fourier, and least squares calculations produced a final weighted R value of 8.92% and a σ_1 of 0.975 for a structure that retains (B_{12}) icosahedra and randomly replaces one-fourth of the [CBC] chains with B_4 rhombuses. Terminal atoms of the latter are fixed along a triad axis and have a fivefold coordination, being bonded to two central atoms and three icosahedral atoms. Central atoms may lie in one of six pairs of opposed equivalent positions in a plane normal to the triad axis and have a threefold coordination, being bonded to two terminal atoms and one icosahedral atom. Possible reasons for the location of the boron-rich terminus of the boron carbide phase field are advanced on the basis of the proposed structure.

3. FUNDAMENTAL CERAMICS

G. P. Smith

This group does research on nonmetals in support of various energy programs. Activities reported here cover basic research on the mechanical properties of UO_2 (a nuclear fuel material), pyrolytic carbons (such as are used to coat fuel materials), transition metal tellurides (which play an important role in the degradation of metal containers exposed to fission products), and impurity atoms in crystalline systems (which serve as model materials with respect to various applications).

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[29]Abstract of paper to be presented at the Summer Meeting of the American Crystallographic Association, Pennsylvania State University, University Park, Pa., Aug. 19–23, 1974.

On the Observation of Lattice and Grain Boundary Dislocations in UO_2 Deformed at High Temperatures[30]

C. S. Yust and J.T.A. Roberts[31]

The microstructure of a series of deformed polycrystalline uranium dioxide specimens was studied by transmission electron microscopy. The lattice and grain boundary dislocation structures observed correlate with three temperature regimes of deformation. At temperatures below the brittle-to-ductile transition temperature, T_c, the specimen is completely brittle, and all grains are essentially free of dislocations. The grain boundary structures observed in these brittle specimens are very regular in appearance. In the temperature range between the high-temperature transition, T_t, and T_c, where small degrees of plastic deformation are recorded before fracture, the first evidence of dislocation activity within the grains is noted, while the boundary structures remain regular. Above T_t, appreciable plasticity occurs before fracture, and extensive dislocation networks form in the grains of the polycrystal. In addition, the structures in the grain boundaries become very irregular in appearance. A qualitative discussion of the deformation process in polycrystalline UO_2 is derived from the observations of this study.

Structure of Pyrolytic Carbon Coatings[32]

C. S. Yust

Investigation of a series of fuel-sphere pyrolytic carbon coatings by transmission electron microscopy has been initiated to provide structural data on a variety of these coatings. The data can be correlated with other measured coating properties such as density, anisotropy, and fracture stress. The initial experimental samples are a series of coatings prepared under fixed propene gas concentration (20 vol %) at temperatures ranging from 1100 to 2000°C. The initial transmission electron micrographs show that the coating structures prepared at 1250 and 1400°C are distinctly different. The distinction is evident in both bright-field and dark-field electron micrographs, as well as in the electron diffraction pattern. In general, the 1250°C coating is composed of large, dense, well ordered structural units, 0.5 to 1.0 μm in diameter, containing large regions of common orientation within which very small (∿25 Å diam) coherently diffracting volumes are observed. In contrast, the coating prepared at 1400°C is composed of spherelike units of highly disordered material, 0.5 to 1.0 μm in diameter, of much lower apparent density, and containing a

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[30]Abstract of *J. Nucl. Mater.* 48: 317–29 (1973).

[31]Materials Science Division, Argonne National Laboratory, Argonne, Ill.

[32]Work performed in Institut für Reaktorwerkstoffe, KFA, Jülich, West Germany.

relatively uniform distribution of coherently diffracting volumes significantly larger than those observed in the 1250°C specimen. The differences observed in these structures are consistent with the density and anisotropy differences measured for these coatings.

Thermodynamic and Chemical Properties of Transition Metal Tellurides

J. Brynestad

A program has been established to study the thermodynamic, chemical, and structural properties of metal-rich phases of nickel and chromium tellurides and the metal-rich end of the ternary system Ni-Cr-Te. This program is closely coordinated with simultaneous studies of the interaction of tellurium with Hastelloy N, which are directed at understanding the intergranular embrittlement of Hastelloy N caused by tellurium.

Polarized Electronic Absorption Spectrum of Ni^{2+} in $Ca_2MgSi_2O_7$

L. M. Toth and G. P. Smith

The optical absorption spectrum of Ni^{2+} (0.5 mole %) doped in akermanite, $Ca_2MgSi_2O_7$, was measured at 2, 80, and 298 K. Akermanite has a tetragonal structure of space group $P\bar{4}2_1m$, in which it is presumed that the Ni^{2+} ions substitute on four-coordinated Mg sites of S_4 symmetry. The assignment of the Ni^{2+} spectrum is based on a distorted tetrahedral structure $NiO_4{}^{6-}$ and is consistent with a species of S_4 symmetry. Polarized infrared and Raman spectra of undoped akermanite have been measured[33] to provide a direct determination of the phonon frequencies giving rise to the vibronic structure that is observed in the electronic spectrum. The absence of extensive structure in the 2-K spectrum indicates either that other sites in the crystal are also occupied by Ni^{2+} or that the $NiO_4{}^{6-}$ species cannot be viewed as a distinct molecular entity. In view of the cross-linked nature of the akermanite structure, the latter explanation is preferred.

4. CRYSTAL PHYSICS

G. W. Clark

The growth of crystals of high-melting refractory materials is our central theme. Frequently very specific crystals (composition, phase, purity, perfection, size, etc.) are required to characterize physical properties uniquely or are required in technical devices for their optimum

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[33]The infrared and Raman measurements have been made in cooperation with J. B. Bates, Solid State Division.

operation; such suitable crystals are often difficult to obtain. Hence, we are conducting a continuing program to devise and improve methods of crystal growth, to develop increased understanding of crystal growth processes and kinetics, and to provide crystals needed in research. Crystals are grown by several methods: by internal centrifugal zone growth, by temperature-gradient zone melting, from molten-salt solvents, from supercritical aqueous systems, by edge-defined film-fed growth, and by the general Verneuil method. During this report period, our crystals were shared for investigating electron spin resonance; optical, magnetic, and elastic properties; deformation; diffusion; field emission; and electronic oscillator quality. Also, we are investigating selected physical properties, both those related to the crystal growth process and those important for characterizing new compounds and eutectic structures.

Unidirectional Solidification in the System ThO_2-Tungsten

A. T. Chapman[34] and G. W. Clark

The ultimate refractory metal oxide-metal eutectic system for use at high temperatures is ThO_2-tungsten, since both components have melting points well above 3000°C. We have successfully induction melted ThO_2-tungsten mixtures initially fabricated as a ThO_2-W-WO_3 rod contained within a pure ThO_2 cylinder. After preheating this duplex rod above 2000°C a stable internal molten zone was formed when the sample was exposed to a 3–4 MHz rf field. Following unidirectional solidification most samples revealed regions of a eutectic structure consisting of tungsten fibers aligned in the oxide matrix. Multiple experiments indicated that the increased oxygen potential available in H_2O/H_2 and CO_2/CO gas mixtures promoted better eutectic structures than found in ThO_2-tungsten samples solidified in a $N_2 + H_2$ atmosphere. Unfortunately, as yet, we found no set of growth conditions that approached "steady-state" conditions and provided and maintained sufficient tungsten solubility in molten ThO_2 to give extensive areas of uniform composite growth.

Metal Oxide-Metal Binary and Ternary Eutectics

G. W. Clark

We have been searching for metal oxide-metal eutectic systems that demonstrate coupled growth of metal fibers or lamellae in one or more oxide phases by directional solidification. Last year we reported discovering seven new binary systems $CaO{\cdot}Cr_2O_3$-W, $CaO{\cdot}Cr_2O_3$-Mo, $MgO{\cdot}Cr_2O_3$-W, $Al_2O_3(Cr_2O_3)$-W, $Al_2O_3(Cr_2O_3)$-Mo, $MgO{\cdot}Al_2O_3(Cr_2O_3)$-W, and $MgO{\cdot}Al_2O_3(Cr_2O_3)$-Mo. Also, six ternary systems were found in which one oxide phase was continuous and the other oxide and metal phases grew coupled as fibers

[34]Consultant from the Georgia Institute of Technology.

or blades. These systems were $CaO \cdot Cr_2O_3$-CaO-W, Cr_2O_3-ZrO_2-W, Cr_2O_3-ZrO_2-Mo, Cr_2O_3-HfO_2-Mo, $MgO \cdot Cr_2O_3$-ZrO_2-Mo, and MgO-ZrO_2-W. We are currently seeking to establish the requirements for steady-state growth and determine the composition and temperature dependence for these systems.

Studies on the Origin of the 3400-cm^{-1}-Region Infrared Bands of Synthetic and Natural α-Quartz

P. A. Staats[35] and O. C. Kopp[36]

Boron was incorporated into synthetic quartz, thereby producing by perturbing an OH vibration an infrared band at 3594 cm^{-1} whose absorption coefficient varies with boron content. We could not confirm the correlations of infrared band with alkali ions, as reported by others. Ions such as Al^{3+}, Y^{3+}, Ti^{4+}, and Be^{2+} produced no new sharp absorption bands in the infrared spectra. We concluded that the impurity perturbation of the OH vibration was strongly influenced by the impurity site and that probably the tunnel sites were more important than interstitial or lattice sites. Mass-spectrometric analyses of 16 crystals were done. We determined infrared spectra of natural quartz, undoped synthetic quartz, and boron-doped synthetic quartz.

The Validity of the Pressure Correction in Inclusion Thermometry Using Fluid Inclusions in Hydrothermally Grown Quartz

O. C. Kopp[36]

We have undertaken a joint investigation with Edwin Roedder of the U.S. Geological Survey, Reston, Virginia, to evaluate the validity of inclusion thermometry when the pressure corrections are large. The crystals used in this study were grown in the pressure range 0.7 to 1.8 kilobars. In spite of relatively large temperature corrections (74 to 173°C) the observed differences between the actual temperatures measured during growth and those determined by inclusion thermometry ranged from only −11 to +24°C. The data are now being evaluated statistically and will be presented at the International Mineralogical Association Meeting in Berlin in September, 1974.

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[35]Physics Division.

[36]Consultant from the Department of Geology, University of Tennessee, Knoxville.

Electron Paramagnetic Resonance of $^{239}Pu^{3+}$ and $^{243}Am^{4+}$ in CeO_2 and of $^{241}Pu^{3+}$ in ThO_2 (ref. 37)

W. Kolbe,[38] N. Edelstein,[38] C. B. Finch, and M. M. Abraham[39]

The g values of the $5f^5$ ions Pu^{3+} and Am^{4+} in CeO_2 cubic symmetry sites have been measured by EPR techniques and found to be 1.33 ± 0.001 and 1.3120 ± 0.0005, respectively. For the case of Pu^{3+}, the cubic crystal-field parameters were calculated and compared with those previously obtained for other fluorite-structure lattices. The EPR spectrum was also observed for ^{241}Pu in ThO_2, and the ratio of the magnetic moments $^{241}\mu_I/^{239}\mu_I$ determined to be 3.52 ± 0.06.

EPR Determination of the Nuclear Spins and Magnetic Moments of ^{243}Cm and ^{247}Cm (ref. 40)

M. M. Abraham,[39] L. A. Boatner,[41] C. B. Finch, R. W. Reynolds,[41] and W. P. Unruh[42]

EPR spectroscopy was employed in making the first direct measurements of the nuclear spins of ^{243}Cm and ^{247}Cm ($I = 5/2$ and $I = 9/2$, respectively). Estimated nuclear moments are $|0.4\mu_N|$ for ^{243}Cm and $|0.36\mu_N|$ for ^{247}Cm.

Czochralski Growth and Characterization of Single-Crystal Akermanite ($Ca_2MgSi_2O_7$) (ref. 43)

C. B. Finch, G. W. Clark, L. A. Harris, and C. S. Yust

High-purity, optically transparent single crystals of the tetragonal silicate akermanite ($Ca_2MgSi_2O_7$) were grown by the Czochralski method in sizes up to 1 cm diam × 3 cm long. The boules were pulled in air, argon, and nitrogen from stoichiometric melts contained in platinum crucibles at 1450 to 1500°C. The best growth was obtained in argon or nitrogen at pull

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[37]Abstracted from *J. Chem. Phys.* 60: 607–09 (1974).

[38]Lawrence Berkeley Laboratory, University of California, Berkeley.

[39]Solid State Division.

[40]Abstracted from *Phys. Lett.* 44A: 527–28 (1973).

[41]Advanced Technology Center, P.O. Box 6144, Dallas, Texas.

[42]On leave from the Department of Physics, University of Kansas, Lawrence.

[43]Abstracted from paper accepted for publication in the *Journal of Crystal Growth.*

rates of 3 mm/hr or less, and spectrochemical analyses indicate a crystal purity exceeding 99.99%. Several properties of the resulting samples are given, including x-ray diffraction, optical, and compression data. The room-temperature uniaxial compressive strength of akermanite was determined to be 35 kg/mm^2 (343 MPa) in the [001] and 5.5 kg/mm^2 (54 MPa) in the [100]. Boules were also grown doped individually with various limited amounts of Mn^{2+}, Fe^{2+}, Ni^{2+}, Nd^{3+}, Eu^{3+}, or Gd^{3+}.

Czocharlski Growth of Single-Crystal Gehlenite ($Ca_2Al_2SiO_7$)

C. B. Finch and G. W. Clark

Single-crystal boules (6 mm diam × 2 cm) of the tetragonal silicate gehlenite ($Ca_2Al_2SiO_7$) were grown by the Czochralski method at 1600°C. The best growth was obtained from Pt—20% Rh crucibles in air at lift rates of 2 mm/hr or less. The growth parameters of gehlenite have not yet been optimized, and boules are more prone to growth imperfections and thermal cracking than is isostructural akermanite ($Ca_2MgSi_2O_7$) grown by the same method. Both gehlenite and akermanite are of interest as potentially useful optical materials or phosphor hosts.

Charge-Transfer Spectra of Fe^{3+} and Mn^{2+} in Synthetic Forsterite (Mg_2SiO_4) (ref. 44)

R. A. Weeks,[39] J. C. Pigg,[39] and C. B. Finch

The optical absorption spectra of nominally pure and manganese-doped Czochralski-grown single crystals of Mg_2SiO_4 have been measured from 1 to 7.5 eV. The intensity of weak optical absorption bands at 4.78, 6.0, and 6.6 eV in the nominally pure crystal has the same dependence upon irradiation with gamma rays and subsequent thermal treatments as does the intensity of the electron paramagnetic resonance (EPR) spectrum of Fe^{3+} located in the forsterite M(2) site. On the basis of this correlation, we assume that these optical bands are due to the Fe^{3+} ion. Oscillator strengths, calculated on the basis of the concentration of Fe^{3+} ions, are 0.2, 1.0, and 0.5 for the 4.78, 6.0, and 6.6 eV bands, respectively. These strengths are those expected for charge-transfer bands. The optical absorption spectrum of the manganese-doped crystal has an intense band at 6.5 eV and an indication of another above 7.3 eV. If the 6.5-eV band is due to a transition involving Mn^{2+}, its oscillator strength is 0.1, and hence this band is attributed to a charge-transfer transition involving Mn^{2+}. Tentative estimates of Dq are $Dq(Fe^{3+}) \approx 1050\ cm^{-1}$ and $Dq(Mn^{2+}) \approx 850\ cm^{-1}$.

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[44]Submitted to *American Mineralogist*.

Radioluminescence of $SrCl_2$ Doped with Cm^{3+} and Es^{2+}

J. P. Young[45] and C. B. Finch

Crystals of $SrCl_2$ doped with the transuranics Cm^{3+} and Es^{2+} emit a strong, blue radioluminescence at 4000 Å. This effect has been attributed to the presence of an F-center associated with Cl^- deficiency (Sr excess) in the host. The crystals also exhibit less intense emission peaks at higher wavelengths, some of which are not quenched at temperatures as high as 400°C. The material may prove useful as a possible light source enabling the colorimetric determination of uranium in a molten-salt reactor.

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[45]Analytical Chemistry Division.

PART 2. PHYSICAL PROPERTIES AND TRANSPORT PHENOMENA

1. SURFACE PHENOMENA

J. V. Cathcart

The long-term aims of the research of the Surface Phenomena Group are to identify the various mechanisms by which alloying elements influence the oxidation behavior of the host metal and to develop a systematic set of guidelines that would provide a generalized basis for the design of oxidation-resistant alloys. These goals are being pursued through two programs, one dealing with the oxidation of a series of uranium-base alloys and the other involving several tantalum- and niobium-base alloys. The former project is concerned with the measurement of defect concentrations and diffusion coefficients in low-temperature oxide films as functions of alloy content. The refractory alloy investigation deals with several phenomena, including internal oxidation, grain boundary embrittlement, and the diffusion of both oxygen and the alloying elements in the parent alloy.

Oxidation of Uranium-Base Alloys

Current Investigations — J. V. Cathcart and G. F. Petersen

In general the defect flux across a growing oxide scale (and, hence, the oxidation rate) is determined by the product of the defect concentration gradient, ΔC, and the chemical diffusion coefficient, $\tilde{D}$, for the oxide. The presence of impurity atoms in the scale may change either of these important parameters, and, using a method called the "interrupted oxidation technique,"[1] we measure ΔC and $\tilde{D}$ as a function of alloy content and composition for the oxide on a series of uranium alloys. Data have been gathered at temperatures from 150 to 300°C for binary alloys of uranium with niobium, molybdenum, and zirconium. Analysis of the data is in progress.

Gaseous Oxidation of Uranium Alloys[2] — J. V. Cathcart

The gaseous oxidation of uranium and its alloys is a complex process. Material transport across the oxide scale occurs predominately by anion diffusion, leading to the development of large stresses in the oxide. The thermodynamic stability of UO_2 causes the oxygen potential in a compact layer of UO_2 to be so low that many constituents of uranium alloys remain

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[1]A. J. Rosenburg, "Separation of the Defect Concentration and Diffusion Coefficient in Diffusion-Limited Scaling Reactions," *J. Electrochem. Soc.* 107: 795–99 (1960).

[2]Abstracted from paper presented at Conference on the Physical Metallurgy of Uranium Alloys, Vail, Colo., Feb. 11–14, 1974; to be published in the proceedings.

unoxidized and embedded in the UO_2 scale. The resulting scale morphology is frequently intricate and in many instances of high-temperature oxidation plays a critical role in the oxidation process.

The oxidation properties of uranium and a variety of its alloys are reviewed in light of the above ideas. Only high-temperature oxidation is considered. Materials covered include binary alloys of uranium and more complex formulations. The details of oxygen diffusion in UO_2 are discussed because of the relevance of this process to the oxidation of uranium alloys. Evidence is given for stress generation during oxidation, and mechanisms for stress generation and relief are described.

Oxidation of Refractory Metal Alloys

R. E. Pawel and J. J. Campbell

Diffusion processes represent a particularly important part of the oxidation reaction for most metals and alloys. Aside from diffusion in the oxide layers themselves, certain refractory metal alloys exhibit internal oxidation, which involves diffusion and atomic rearrangement in the metal ahead of the advancing oxide-metal interface. Pertinent to our studies of this phenomenon in tantalum alloys containing tungsten and hafnium, we investigated the diffusion of hafnium into tantalum single crystals at temperatures from 1400 to 2000°C. The results of this work, performed in cooperation with the Diffusion in Solids Group, are described in more detail in Sect. 2-2 of this report.

Preparatory to determining oxygen diffusion coefficients in Ta-W and Ta-W-Hf alloys, we used our flexure method to measure oxygen diffusion in polycrystalline tantalum at temperatures from 750 to 900°C. The data were in general agreement with values obtained by different techniques.

We have begun an oxidation study of a Nb—1% Zr alloy by measuring the oxidation kinetics over the range 400 to 900°C in dry oxygen at atmospheric pressure.

Some Oxidation Characteristics of Ta—10% W and Ta—10% W—2.5% Hf Alloys[3] — R. E. Pawel and J. J. Campbell

As part of a program on the oxidation behavior of refractory metal alloys, the early stages of oxidation of the alloys Ta—10% W and Ta—10% W—2.5% Hf—0.012% C (T-222) were studied over the range 500 to 800°C in dry oxygen at atmospheric pressure. Both alloys exhibited a period of protective oxidation followed by an accelerating rate due at the lower temperatures to the initiation and lateral growth of regions consisting of porous, nonprotective oxide. At the higher temperatures, the oxide film was more resistant to this form of breakdown resulting in comparatively longer periods of protection. The strain induced in these alloys during oxidation

[3]Abstract of a paper to be presented at the Fall 1974 AIME Meeting as part of MSD-IMD Symposium on "The Influence of Stresses and Mechanical Properties of Oxide Scales on the Oxidation Behavior of Metals."

was measured by the flexure technique. The kinetics of flexure in the protective stage of oxidation over the above temperature range was determined and compared with the kinetics of oxidation. While high stresses undoubtedly existed in the oxide layers themselves, the major contributor to flexure under these conditions was judged to be oxygen solution. The experimental results are examined in terms of the differences (and similarities) in the kinetics and morphology of the reactions for the individual alloys and for unalloyed tantalum.

Diffusion in Finite Systems with a Moving Boundary[4] — R. E. Pawel

Important characteristics of many oxidation reactions are associated with the infusion of oxygen into the metal in advance of the oxide-metal interface. The calculation of the resulting oxygen profiles generally involves the solution to the diffusion equation for a finite system with a moving boundary. While we could obtain at least approximate analytical solutions in this case for the simplest initial and boundary conditions, it is apparent that these solutions are not easily applicable to many reactions of interest.

In this paper, we present the approximate analytical solutions to the finite-geometry moving-boundary problem and compare results from these solutions with those obtained by a finite-difference method based on a simple model of the system. The methods are reasonably accurate and the results suggest that the latter method would be an especially useful approach in examining diffusion behavior under more complicated conditions.

Oxygen Diffusion in Beta Zircaloy During Steam Oxidation[5] — R. E. Pawel

Analytical and finite-difference methods developed to deal with diffusion in finite systems with moving boundaries were used to compute oxygen concentration profiles in the beta phase of Zircaloy specimens oxidized in steam. The results for isothermal exposures at temperatures from 927 to 1371°C are presented and discussed in terms of the properties and observations from a previously reported experimental data set. The computational results furnish a basis for the argument that the (prior) beta phase oxygen content and distribution may contribute significantly to the observed mechanical properties of Zircaloy specimens. The application of the computational methods was also extended to the case of oxygen diffusion in beta Zircaloy during temperature excursions. The exact shapes of the resulting profiles in these cases were shown to depend not only upon the shape of the temperature transient, but also upon the specific model chosen to represent the beta-to-alpha transformation behavior during cooling.

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[4]Abstracted from *J. Nucl. Mater.* 49: 281–90 (1974).

[5]Abstracted from *J. Nucl. Mater.* 50: 247–58 (1974).

2. DIFFUSION IN SOLIDS

T. S. Lundy

In addition to the items subsequently presented in this section, our efforts involved measurements of the self-diffusion of cations in oriented crystals of rutile (TiO_2) and development of techniques for determination of oxygen diffusion in various refractory systems.

MATANO — A Computer Code for the Analysis of Interdiffusion and Intrinsic Diffusion Information in Binary Systems[6]

P. T. Carlson

MATANO is a FORTRAN IV computer program, which was written for the IBM 360/75 and 360/91 systems to determine interdiffusion and intrinsic diffusion coefficients from the experimental concentration penetration profiles of infinite and semi-infinite diffusion couples.

The program uses analytical techniques to generate a smooth curve through data point sets. Its computational scheme is divided into experimental and theoretical parts so that diffusion coefficients may be deduced from experimental concentration profiles and/or predicted from a prior knowledge of tracer diffusion and thermodynamic information.

To calculate experimental interdiffusion and intrinsic diffusion coefficients, the program first converts the x-ray intensity data to units of concentration and then fits low-order polynominal splines to the concentration-penetration data of each species in a couple by the method of least squares and generates a mathematical expression that defines each profile. The regenerated profile is integrated with the use of Simpson's rule to locate the Boltzmann-Matano plane and then differentiated and integrated at incremental penetration distances to determine interdiffusion coefficients as functions of composition.

The location of the Kirkendall reference plane, determined experimentally, is input data and the intrinsic diffusion coefficients of each species are determined at the composition of this plane by three independent analyses to provide an internal crosscheck. Values of the experimental vacancy wind parameters are calculated at the composition of the Kirkendall plane. The inclusion of disk plotting subroutines supplements the standard printed output with plots of the concentration profiles of each species, as well as plots of the calculated interdiffusion coefficients as functions of composition.

The use of the theoretical portion of MATANO requires a knowledge of tracer diffusion and thermodynamic information at a particular temperature. The computational scheme calculates, prints out, and plots interdiffusion coefficients, intrinsic diffusion coefficients, and the vacancy wind parameters predicted from the theories of Darken and Manning.

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[6]Abstracted from ORNL report in preparation.

The output and flexibility of MATANO provides the user with a rapid summary of both experimental and theoretically predicted diffusion information in tabular as well as graphical formats for a given binary system.

Atomic Mobilities and Vacancy Wind Effects for Diffusion in Ternary Silver-Zinc-Cadmium Solid Solutions[7]

P. T. Carlson, M. A. Dayananda,[8] and R. E. Grace[9]

Diffusion in the silver-zinc-cadmium system was investigated at 600°C with semi-infinite, vapor-solid couples to determine the atomic mobilities and contribution of the vacancy wind effect to the intrinsic diffusion fluxes of silver, zinc, and cadmium in the alpha phase region.

Ternary alloy chips of selected compositions in the alpha phase region were used as vapor sources in contact with diffusion sinks of pure silver and a silver-zinc binary alloy; the couples were annealed 3 to 8 days at 600°C. Inert markers placed at the initial vapor-solid interface determined the position and movement of the Kirkendall reference plane for intrinsic diffusion coefficient determination. The annealed couples were sectioned, metallographically polished, and analyzed by point-to-point counting techniques on an electron microprobe by measuring the x-ray intensities of the Zn Kα and the Cd Lα lines. The measured intensities were converted to compositions by a standard microprobe conversion procedure with the use of both binary and ternary alloy standards.

Atomic mobilities and vacancy wind parameters were computed at selected compositions in the alpha phase along isoconcentration lines of 11 at. % Zn, 18 at. % Zn, 5 at. % Cd, and 9 at. % Cd. An appreciable vacancy wind effect on intrinsic diffusion fluxes is strongly suggested in this system from the observation of maxima in the zinc concentration profiles of several couples, such as that shown for a typical couple in Fig. 2.1. The maxima were more pronounced for couples employing higher concentrations of cadmium in the vapor source. The apparent diffusion of zinc up its concentration gradient, or uphill diffusion, is indicative of strong thermodynamic interactions between the diffusing species. A significant consequence of the theory of a vacancy wind effect is that the cross terms in the general thermodynamic diffusion equation arise entirely from the vacancy wind effect. Furthermore, if the nondiagonal elements of the phenomenological coefficient matrix are appreciable compared with the diagonal elements, they can effectively reverse the direction of flow of a given species and result in uphill diffusion.

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[7]Abstracted from paper submitted for publication in *Metallurgical Transactions*. Experiments were performed at Purdue University and theoretical analyses were done at ORNL.

[8]School of Materials Engineering, Purdue University, West Lafayette, Indiana.

[9]Department of Interdisciplinary Engineering Studies, Purdue University, West Lafayette, Indiana.

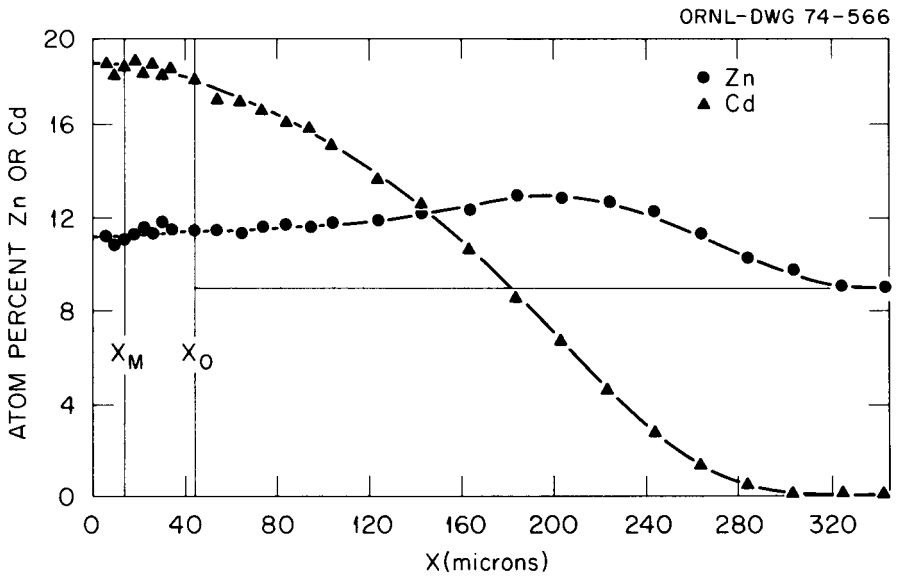

Fig. 2.1. Concentration Profiles of Zinc and Cadmium for the Alloy 4/Alloy B Diffusion Couple Diffused for 5 Days at 600°C.

Significant thermodynamic interaction between zinc and cadmium is also reflected in the values of the intrinsic diffusion coefficients where the cross coefficients,[10] D^3_{12}, were found to be greater than or equal to the diagonal coefficients, D^3_{11}. This indicates a strong vacancy wind effect since the D^3_{ii} and D^3_{ij} coefficients can be directly related in a functional sense to the phenomenological coefficients, L_{ii} and L_{ij}.

The atomic mobilities of zinc and cadmium were smoothly increasing functions of composition; the mobility of zinc was greater than that of cadmium at all compositions, as can be seen from the typical plot given in Fig. 2.2. The fluxes arising from the vacancy wind effect contribute significantly to the total intrinsic fluxes of each component for diffusion into pure silver and silver-zinc binary sinks. Calculations that assume negligible interactions among the diffusing species and, hence, negligible vacancy wind effects are not valid for diffusion in the silver-zinc-cadmium system. This investigation represents the first reported observation of the vacancy wind effect in intrinsic diffusion in a ternary system.

[10] 1 = Zn, 2 = Cd, 3 = Ag.

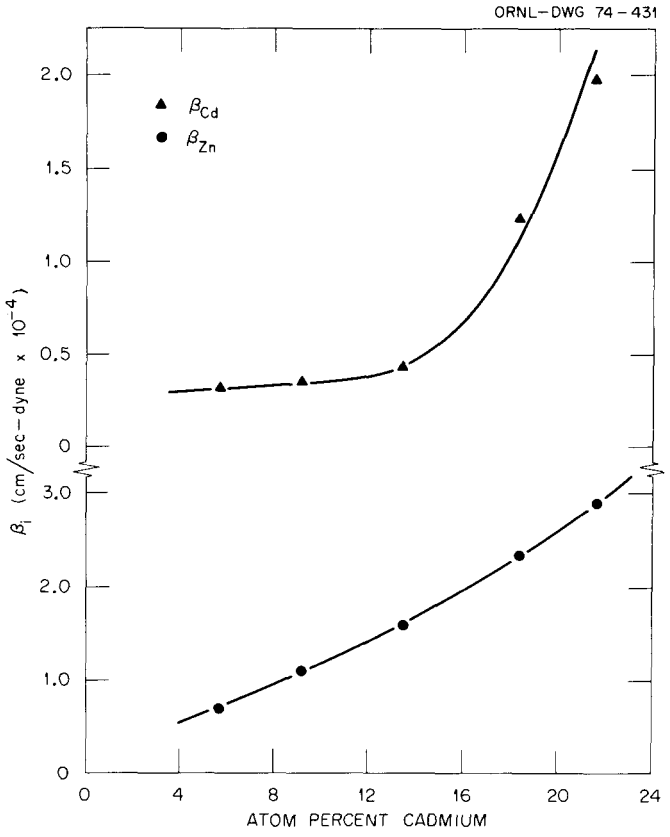

Fig. 2.2. Atomic Mobilities as Functions of Cadmium Concentration for Alpha Ag-Zn-Cd Alloys with 11 at. % Zn.

Interdiffusion and Intrinsic Diffusion in Binary Vanadium-Titanium Solid Solutions[11]

P. T. Carlson and L C Manley, Jr.

Diffusion processes in the presence of chemical concentration gradients are being investigated in binary vanadium-titanium solid solutions with the use of infinite solid-solid diffusion couples. Interdiffusion coefficients, intrinsic diffusion coefficients, and vacancy wind parameters are being determined across the entire composition range of the vanadium-titanium solid solution phase field at temperatures from 900 to above 1600°C. Solid-solid diffusion couples of various compositions are being used with overlap of concentration ranges so as to obtain a reliable set of coefficients at all temperatures. Intensity-penetration profiles

[11]Abstracted from paper submitted for publication in *Metallurgical Transactions* and presented orally at the 1974 Spring Meeting of the Metallurgical Society of AIME, May 20–23, Pittsburgh, Pa.

of the diffused couples were measured with an electron microprobe by simultaneously monitoring the Ti Kα and V Kα line intensities and subsequently converting to concentration units. Inert markers placed at the original weld interfaces of the couples were used to measure the location and movement of the Kirkendall reference plane. The Boltzmann-Matano analysis was applied to the diffusion profiles to determine the experimental interdiffusion coefficients, while the analyses of Darken and Heumann were used to determine the intrinsic coefficients at the composition of the Kirkendall plane. Particular emphasis was placed on the observation in this system of vacancy wind effects, which occur when diffusion proceeds by the vacancy mechanism. The net flux of vacancies gives rise to additional terms in the classical Darken expression for the intrinsic flux: the flux of the faster species is enhanced while that of the slower species is retarded. With the use of published tracer diffusion and thermodynamic information, vacancy wind parameters, as well as interdiffusion and intrinsic diffusion coefficients, were calculated from experiment and theory and compared. The results indicate that interdiffusion and intrinsic diffusion coefficients range from 10^{-14} to 10^{-8} cm^2/sec for the temperature range 900 to 1600°C, and the coefficients increase with increasing titanium content. The vacancy wind phenomenon is especially interesting in the vanadium-titanium system because no net vacancy motion occurs at certain temperatures and compositions. Figure 2.3 shows that at 1350°C the intrinsic diffusion coefficients for

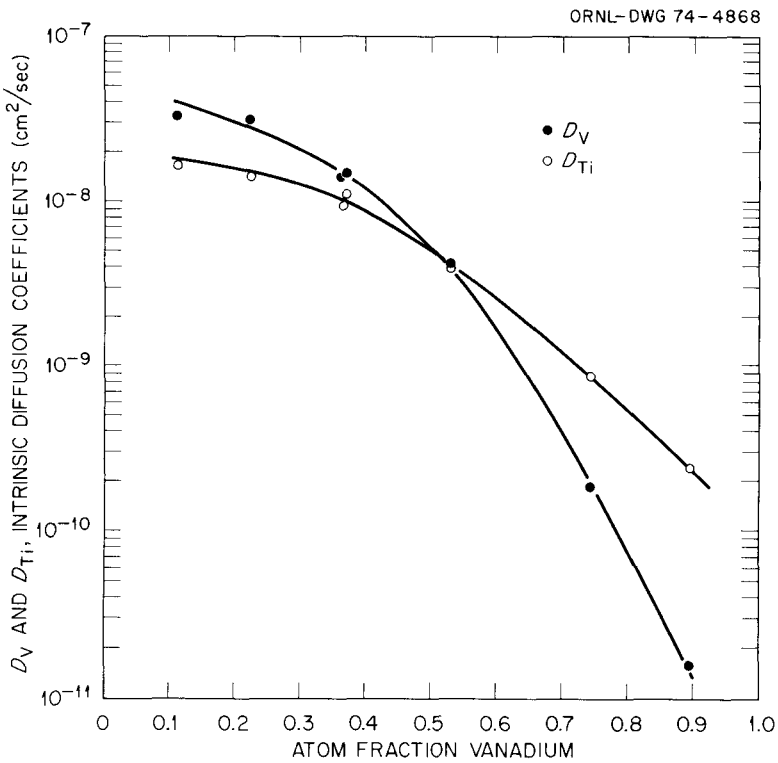

Fig. 2.3. Experimental Intrinsic Diffusion Coefficients of Vanadium and Titanium for Vanadium-Titanium Alloys at 1350°C.

vanadium and titanium are equal at 52 at. % V. The intrinsic diffusivity of vanadium is greater below 52 at. % V, and less than that of titanium at higher vanadium concentrations. The theoretically predicted vacancy wind parameters are shown as functions of composition in Fig. 2.4. Below 52 at. % V, the faster diffusing vanadium is enhanced since $1 + V_V > 1$ and the slower diffusing titanium is retarded since $1 - V_{Ti} < 1$, while at the composition of 52 at. % V, the effects of vacancy wind are zero. The experimentally determined vacancy wind parameters are shown in Fig. 2.5 and display good agreement with the theoretically predicted values. The crossover point of equal diffusivities is shifted to higher vanadium concentrations as the temperature is increased. The results for the vanadium-titanium system indicate the ability to reliably predict the effect of the vacancy wind phenomenon on intrinsic diffusion fluxes. The availability of accurate diffusion coefficients for both matter and vacancies is critical to the ability to predict the extent of void formation and migration in a given material.

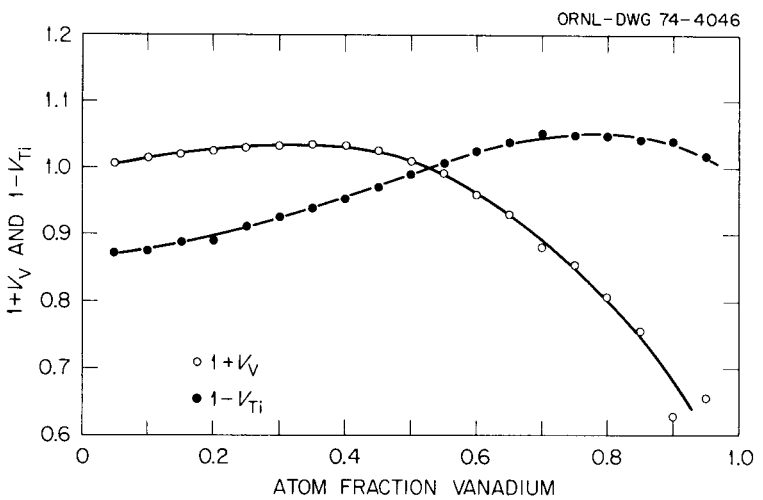

Fig. 2.4. Theoretically Predicted Vacancy Wind Parameters for Vanadium and Titanium as Functions of Vanadium Concentration at 1350°C.

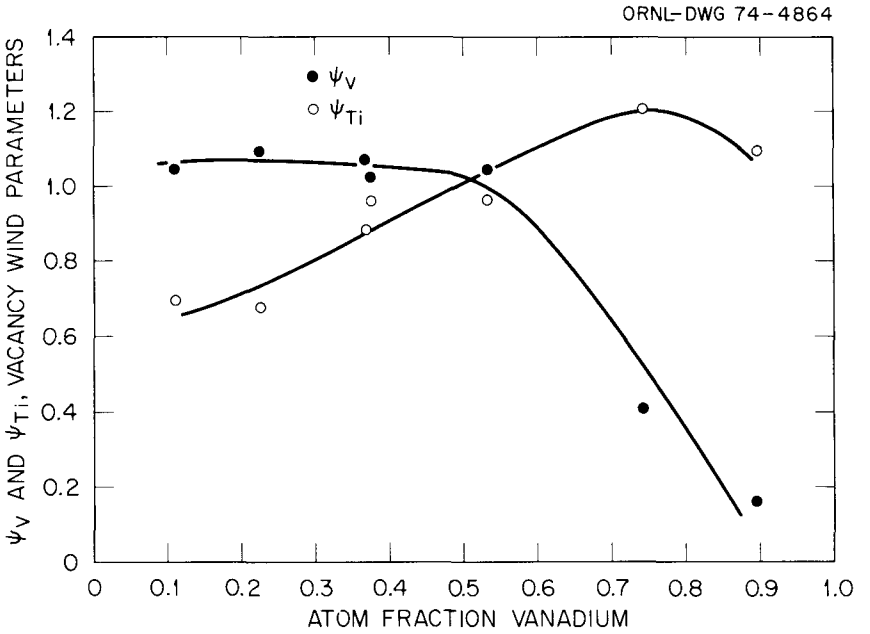

Fig. 2.5. Experimentally Determined Vacancy Wind Parameters for Vanadium and Titanium as Functions of Vanadium Concentration at 1350°C.

Thermotransport of Cadmium and Thallium in Sodium and Potassium Chlorides

P. T. Carlson and L C Manley, Jr.

The thermotransport of monovalent and divalent impurities in single-crystal ionic materials is being investigated with the intent of obtaining a better understanding of the heat of transport in nonmetallic systems. The thermotransport of ^{115m}Cd and ^{204}Tl tracers in single crystals of sodium and potassium chlorides, respectively, is being investigated. Thin disks of single-crystal NaCl and KCl were doped with the respective tracers and annealed in temperature gradients of 800 to 2000°C/cm for periods up to 100 days. The annealed specimens were sectioned with a rotary microtome and the sections counted with suitable detectors in conjunction with a multichannel analyzer to yield activity profiles. The relatively small isothermal diffusion coefficients at the temperatures of interest necessitated a non-steady-state analysis of the data, based on the exact solution to the differential equation describing thermotransport. A computer code was developed for the determination of the heats of transport from both steady-state and non-steady-state distributions. The results for ^{204}Tl diffusion in KCl indicate a preferential migration to lower temperatures with a heat of transport of 25 ± 5 kcal/mole. The thermotransport of ^{115m}Cd in NaCl is characterized by a heat of transport of −2 ± 1 kcal/mole, and consequently cadmium preferentially migrates toward higher temperatures. These investigations give an insight for the understanding of segregation that occurs in nuclear ceramics under the influence of large temperature gradients.

Hafnium Diffusion in Monocrystalline Tantalum

R. E. Pawel and T. S. Lundy

Cooperative efforts with the Surface Phenomena Group have continued with research on the tracer diffusion of ^{181}Hf in monocrystalline tantalum at temperatures from 1200 to 2000°C. Diffusion coefficients are determined from the thin-film solution to Fick's second law in conjunction with the anodizing-and-stripping technique and standard gamma activity analyses. The results indicate that hafnium diffuses slightly faster in tantalum than do either niobium or tantalum tracers and that short-circuiting phenomena are more important for hafnium at higher temperatures than for the other two tracers. A least squares fitting to the Arrhenius equation for seven determinations of the hafnium volume diffusion coefficients at temperatures from 1397 to 2000°C yields

$$D = (0.136 \pm 0.004)\exp[-(92{,}900 \pm 1200)/RT]\ \mathrm{cm}^2/\mathrm{sec}\ .$$

Lower temperature penetration profiles are characterized by segmented Gaussian behavior with the portion near the original tracer plane source related to volume diffusion and deeper penetrations due to atomic migration along short-circuit paths such as dislocation pipes.

Diffusion Under High Pressures

T. S. Lundy

Vendor delays, including a fire at the fabrication plant, have plagued our efforts to acquire a suitable high-pressure, high-temperature system for use in research on diffusion in the refractory metals at temperatures up to 1500°C and hydrostatic pressures of 1 to 15 kilobars (0.1—1.5 GPa). Most of these problems seem to have been worked out now, and the system should be delivered soon. Vendor performance regarding construction of the furnace was unsatisfactory, and we have agreed to build the furnace after receipt of the high-pressure system.

From a fundamental research viewpoint, we are interested in using the system to determine activation volumes for the atomic jump process for tracers in the bcc metals Ta, Nb, and W. The activation volume represents the increase in volume relative to that of the perfect crystal upon the formation and migration of the defect responsible for diffusion. In addition, we believe that changes in tracer penetration behavior versus pressure at given temperatures may allow quantitative assessment of short-circuiting phenomena that are so important at lower temperatures where most engineering applications take place.

3. PHYSICAL PROPERTIES RESEARCH

D. L. McElroy

The mechanisms that govern physical properties of a solid indicate important characteristics of the solid and phenomena occurring therein. We seek an improved understanding by critical data analyses of accurate physical property measurements on selected solids from 4.2 to 2600 K. The physical properties of interest include thermal conductivity, λ; electrical resistivity, ρ; Seebeck coefficient, S; specific heat capacity, C_p; and thermal expansion coefficient, α.

Apparatus Development

Differential Dilatometer[12] — T. G. Kollie and T. G. Godfrey

A fused quartz differential dilatometer was interfaced with a computer to measure automatically the thermal expansion of solids from 300 to 1000 K. Measurements on National Bureau of Standards certified fused silica, copper, and tungsten demonstrated an uncertainty of about $\pm 1 \times 10^{-7}$/K in the determination of α. Measurements of α between 300 and 800 K on commercially pure copper were within ±0.3% of our data on NBS certified copper. Creep-induced errors in the NBS data between 600 and 800 K may have caused a 1.5% difference between the NBS values and our values. Between 300 and 600 K, the two data sets agree to ±0.4%.

Computer-Operated Data Acquisition System (CODAS) — T. G. Kollie, T. G. Godfrey, and S. H. Jury[13]

CODAS capacity was extended by addition of a second processor and teletypewriter, a 16-channel multiplexer, an analog-to-digital converter, and computer-controlled relays. This allows CODAS to operate two experiments simultaneously and independently.

A transient technique for simultaneously determining the λ and thermal diffusivity of solids was developed. This technique relies totally on CODAS to conduct the experiment and analyze the results. An accuracy of ±2% is indicated by results on NBS gum rubber and Pyroceram. Three such systems are under construction to operate from 0°C to about 200°C.

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[12] T. G. Kollie, D. L. McElroy, J. T. Hutton, and W. M. Ewing, "A Computer Operated Fused Quartz Differential Dilatometer," pp. 129–146 in *Thermal Expansion — 1973 (Lake of the Ozarks)*, AIP Conf. Proc. 17, ed. by R. E. Taylor and G. L. Denman, American Institute of Physics, New York, 1974.

[13] Consultant, University of Tennessee.

Guarded Linear Heat Flow Apparatus[14] — J. P. Moore, D. L. McElroy, and R. S. Graves

A guarded-longitudinal-heat-flow technique for measuring λ, ρ, and S on small rods between 300 and 1000 K is described. The low λ of the insulation permitted use of the thin-rod approximation for calculating λ. Determinate errors were ±0.4% for ρ, ±0.14 μV/K for S with respect to platinum, and ±1.3% and ±2.2% for λ at 300 and 1000 K, respectively. The apparatus was tested with an Armco iron reference specimen, which had a λ known to within ±1.9%. With brazed connections between the specimen heater and heat sink, results were within the determinate error. Measurements with the heater and heat sink threaded to the reference produced ρ and S results within the determinate error, but the λ results were 2 to 6% above the reference values. The cause of this discrepancy may be related to the interfaces.

The λ, ρ, and S of two grades of nearly isotropic graphite were measured from 300 to 1000 K both before and after neutron irradiation in HFIR to a fluence of 5.0×10^{21} neutrons/cm^2 (>50 keV). Nominal irradiation temperatures were 823, 923, and 1023 K. The thermal resistance, $1/\lambda$, of the unirradiated graphites was proportional to T from 500 to 1000 K. Neutron irradiation decreased λ at 300 K by a factor of 4.5 and increased ρ at 300 K by 2.5, in general agreement with previous investigations. Irradiation changed S from small negative values to large positive ones. The product of thermal conductivity and electrical resistivity was nearly constant with fluence from 2.6×10^{21} to 5.0×10^{21} neutrons/cm^2.

Heat Flow Analysis[15] — S. H. Jury,[13] T. G. Godfrey, and J. P. Moore

End effects on the flow of heat, mass, or electrical energy through a cylindrical rod were investigated analytically. Analytical solutions evaluated on an IBM 360/91 computer compared favorably with the results of a finite difference heat conduction code (HEATING-3).

Platinum and Hastelloy X rods were subjected to an electrical current applied via two electrodes, one covering one end and the application of the other being the subject of investigation. End effects based on voltage

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[14]Abstract of two reports: J. P. Moore, D. L. McElroy, and R. S. Graves, *A Technique for Determining Thermal and Electrical Conductivity and Absolute Seebeck Coefficient Between 300 and 1000 K*, ORNL-4986 (in press); J. P. Moore, R. S. Graves, and D. L. McElroy, "Thermal and Electrical Conductivities and Seebeck Coefficients of Unirradiated and Irradiated Graphites from 300 to 1000°K," *Nucl. Technol.* 22: 88–93 (1974).

[15]Abstract of two reports: S. H. Jury, D. Arnurius, T. G. Godfrey, D. L. McElroy, and J. P. Moore, "End Effects on the Flow of Heat, Mass, or Electrical Energy Through a Cylindrical Rod," accepted for publication in the *Journal of the Franklin Institute;* T. G. Godfrey, D. L. McElroy, and Z. L. Ardary, "Thermal Conductivity of Oriented Fibrous Carbon Insulation from 300 to 1300°K in Nitrogen and Argon at One Atmosphere," *Nucl. Technol.* 22: 94–107 (1974).

deviations of 0.1% or more were found to extend for a length-to-diameter ratio ranging from 0.6 to 1.9, depending upon whether the small electrode position being investigated was at the center of the end or on the periphery of the end. Calculated results from HEATING-3 showed excellent agreement with the experimental results.

This analytical solution also represents a contribution to a neglected area of mathematical physics and as such should prove equally useful in other areas that are dynamically analogous. The discontinuous infinite integral technique of solution has proven to be a very powerful one.

In addition, HEATING-3 analyses of thermal configurations of the radial heat flow apparatus provided valuable insight about assumed boundary conditions. Equipment modifications suggested by these calculations were applied for measurements to 1300 K.

Metals

Irradiation Effects in Tungsten-Rhenium Alloys[16] — R. K. Williams, J. O. Steigler, and F. W. Wiffen

Tungsten-base alloys containing 5, 11, and 25% Re were irradiated in the EBR-II at temperatures from 600 to 1500°C. All compositions were irradiated to fluences of 4.3 to 6.1 × 10^{21} neutrons/cm^2 (>0.1 MeV), and three 25% Re samples were irradiated to 3.7 × 10^{22} neutrons/cm^2 at temperatures from 700 to 900°C. Postirradiation examination included measurement of electrical resistivity at room temperature and lower temperatures, x-ray diffraction, optical metallography, microprobe analysis, and transmission electron microscopy, including selected-area diffraction analysis. Irradiation-induced resistivity *decreases* observed in most of the samples suggested second-phase precipitation. Complete results confirmed the precipitate formation in all samples, in disagreement with existing equilibrium phase diagrams for the tungsten-rhenium system. Electron diffraction showed the precipitates to be consistent with the cubic rhenium-rich chi phase and inconsistent with the sigma phase. Large variations in precipitate morphology and distribution were observed between the different compositions and irradiation conditions. For the 5 and 11% Re alloys, spherically symmetric strain fields surrounded the disk-shaped precipitate particles, and persisted even after the particles had etched out. These strain fields are believed to arise from rhenium concentration gradients.

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[16]Abstract of paper presented at the Sixth Annual Spring Meeting Institute of Metals Division, The Metallurgical Society of AIME, May 20–23, 1974, Pittsburgh, Pa.

Molybdenum — R. K. Williams, R. S. Graves, and J. P. Moore

The improved accuracy of the longitudinal heat flow apparatus[17] has been used to obtain λ, ρ, and S data on two molybdenum-base alloys, containing 1.07 and 2.06 wt % Nb. The precision of the results was comparable to that of the earlier results on two high-purity molybdenum specimens. Analysis of the data is incomplete, but the results appear to support our previously reported[18] lattice thermal conductivity values for pure molybdenum. Measurements on molybdenum-base alloys containing zirconium are in progress.

Chromium — J. P. Moore and R. K. Williams

Measurements of λ, ρ, and S of high-purity chromium from 280 to 1000 K were completed using a high-temperature longitudinal heat flow technique.[14] In addition, S and ρ of a companion specimen were measured in a separate apparatus from 300 to 1200 K. All data from the present study agree with our previously published results from a third technique[19] to within experimental uncertainties. Although λ and ρ have small (~1%) local maxima and minima near the antiferromagnetic-paramagnetic transition (T_N) at 311 K, $\lambda\rho/T$ is smooth, which confirms our previous conclusion[20] about the behavior of λ and S of pure chromium near T_N. Above T_N from 320 to 1200 K, many aspects of the properties are similar to those of the other VIB transition metals, molybdenum and tungsten.

Heat Transport in Alkali Halides

J. P. Moore, R. K. Williams, and R. S. Graves

At this time, only small specimens of some alkali halide compounds suitable for measurement of heat conduction can be fabricated. This

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[17] J. P. Moore, R. K. Williams, and R. S. Graves, "Precision Measurements of the Thermal Conductivity, Electrical Resistivity and Seebeck Coefficient from 80 to 400 K and Their Application to Pure Molybdenum," *Rev. Sci. Instrum.* 45: 87–95 (1974).

[18] R. K. Williams and W. Fulkerson, "Separation of the Electronic and Lattice Contributions to the Thermal Conductivity of Metals and Alloys," pp. 389–456 in *Thermal Conductivity, Proc. 8th Conf.*, ed. by C. Y. Ho and R. E. Taylor, Plenum Press, New York, 1969.

[19] J. P. Moore, R. K. Williams, and D. L. McElroy, "Physical Properties of Chromium from 77 to 400°K," pp. 297–310 in *Thermal Conductivity, Proc. 7th Conf. Gaithersburg, Maryland, Nov. 13–16, 1967, Nat. Bur. Std. Spec. Publ.* 302, ed. by D. R. Flynn and B. A. Peavy, Jr., National Bureau of Standards, Washington, D.C., September 1968.

[20] J. P. Moore, R. K. Williams, and D. L. McElroy, "Comments on 'Lorenz-Function Enhancement Due to Inelastic Processes Near the Neel Point of Chromium'," *Phys. Rev. Lett.* 24, 587–88 (1970).

restricts λ measurements to a longitudinal technique, which is inaccurate when λ of the specimen is low compared to that of the insulation surrounding the specimen. Corrections for the heat shunted through the insulation have been determined by making measurements on a Pyroceram thermal conductivity standard, which has a λ near that of the alkali halides of interest. These corrections have been applied to data on the rubidium compounds, and the resulting λ data on RbI, RbCl, and RbBr have been analyzed.

Polycrystalline samples of the compounds CsCl, CsBr, and CsI have been fabricated by the method developed for production of RbI samples. In this technique, high-purity powder, about 65% dense, is sealed in a Teflon-lined copper can, heated under vacuum to drive off moisture, and swaged to a 3 or 4 to 1 reduction. After dejacketing, the samples are annealed at 300 to 400°C to remove some very fine grained material. The samples are typically 98 to 99% dense, have infrared extinction coefficients (at 10 μm) of 3 to 6/cm, and consist of equiaxed grains with small pores at the grain boundaries. The material can be machined to produce cylindrical samples, and we have been able to drill deep small-diameter holes for thermocouple wires.

Specific Heat Capacity

The Heat Capacity of Ni_3Fe (ref. 21) — T. G. Kollie and C. R. Brooks[22]

The heat capacity, accurate to about ±1%, of Ni_3Fe has been measured from 300 to 1670 K using pulse calorimetry, with heating rates from 20 to 60 K/sec. The specimen was heat treated in the calorimeter to induce various degrees of long-range order, varying in Bragg-Williams parameter S from 0 to 0.96. The heat capacity was measured for various initial values of S. The rapid heating allowed measurements on the ordered structures of Ni_3Fe at temperatures where they were metastable. Below 750 K, the heat capacity-temperature curve was lowered as S increased. The Curie transformation for a high value was 70 K above that for a disordered condition ($S = 0$). The order-disorder transformation was suppressed to 230 K above the equilibrium transformation temperature by use of a heating rate of 60 K/sec.

Aluminum — T. G. Kollie

Measurements of C_p of aluminum between 300 and 900 K were attempted using the direct heating pulse calorimeter. Data taken during cooling after a pulse overcorrected for the radiative heat loss during heating because of the high λ and low ρ of aluminum. Completion of these measurements awaits development of a finite-difference computer code to properly evaluate these heat losses.

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[21]Abstract of T. G. Kollie and C. R. Brooks, "The Heat Capacity of Ni_3Fe: Experimental Data from 300 to 1670 K," *Phys. Status Solidi (a)* 19: 545–54 (1973).

[22]Consultant, University of Tennessee.

Technetium — T. G. Kollie and J. O. Scarbrough

CODAS has been interfaced with a low-temperature pulse calorimeter for measurement of C_p of technetium from 4 to 12 K. These data will allow calculation of the Debye temperature and the electronic contribution (γT) to the heat capacity. Calibration of the equipment is complete, and initial measurements indicate the need for a new specimen holder design to ensure that the beta decay heat of technetium is dissipated uniformly in the specimen and its holder.

Coefficient of Thermal Expansion

T. G. Kollie and T. G. Godfrey

The α's of monoclinic and cubic Eu_2O_3 were measured from 300 to 1000 K. Between 800 and 900 K the monoclinic α deviated from theoretical extrapolation of the 300 to 800 K data, and above 900 K the data were not repeatable. Similar anomalies have been found by others in the C_p of monoclinic Eu_2O_3. The cubic Eu_2O_3 sample exhibited anomalous decreases in sample lengths during isothermal treatments between 600 and 1000 K. However, after holding at 1000 K the lengths did not change with time at temperature below 900 K.

The expansion of single-crystal and natural NaCl was determined from 300 to 1000 K and 300 to 500 K, respectively. The α results were within 1% of literature data, even at high temperatures where α increases rapidly. On heating, the brine in natural NaCl caused 2% deviations from the single-crystal data. Most of this volume change was due to plastic deformation, and data taken during cooling agreed with that for the single crystal.

4. SUPERCONDUCTING MATERIALS

C. C. Koch

We are studying the effects of metallurgical variables on the properties of superconducting materials. The most highly structure-sensitive superconducting property appears to be current-carrying capacity in an applied magnetic field. It is sensitive to microstructural variables such as grain size, dislocation density and distribution, and morphology, composition, and volume fraction of second-phase particles. Meaningful correlation of structure and properties requires detailed knowledge of both. Consequently, some of our effort is devoted to obtaining basic metallurgical information on phase diagrams, transformation kinetics and products, and the microstructures that result from them in superconducting alloy systems. We correlate current-carrying capacity with the "model" microstructures.

We also study the influence of metallurgy on the basic superconducting property T_c, the superconducting-normal transition temperature. This property is controlled by alloying and, in compound superconductors, by degree of long-range chemical order. In conjunction

with this part of our program we make low-temperature specific heat capacity measurements.

We have recently begun studies of power frequency ac losses in superconductors and of the relationship of mechanical strain under operating conditions to the superconducting properties of commercial Nb-Ti/Cu composites.

Fluxoid Pinning in Niobium Containing Small Amounts of Yttrium or Gadolinium[23]

C. C. Koch and D. M. Kroeger

The fluxoid pinning of niobium containing small amounts of either yttrium or gadolinium (≤ 2 at. %) was determined by dc magnetization measurements on specimens in the heavily deformed, partially recovered, and recrystallized conditions. Optical and electron microscopy were used to characterize the samples.

Magnetic hysteresis of niobium was increased by the additions of yttrium or gadolinium. In the cold-worked condition, increased pinning was due to planar sheets and stringers of yttrium (gadolinium) produced by the deformation and parallel to the fluxoid axes. Fluxoid pinning was a function of yttrium (gadolinium) concentration. In the recrystallized samples, the high-angle grain boundaries were the most important pinning sites, and the pinning was independent of yttrium (gadolinium) content. The yttrium (gadolinium) enhanced the pinning effectiveness of grain boundaries by: (1) inhibiting grain boundary migration and thus increasing the grain boundary surface area (decreasing grain size), and (2) reducing preferred orientation in the recrystallized state, thus providing greater average grain misorientation, which for niobium with its anisotropic superconducting parameter (κ) produces an additional pinning effect.

Superconducting Transition Temperatures of Chemically Vapor Deposited Tungsten-Rhenium Alloys[23]

D. S. Easton, C. C. Koch, D. M. Kroeger, and J. W. Cable[24]

Superconducting transition temperatures (T_c) have been measured on a series of chemically vapor deposited (CVD) tungsten-rhenium alloys. The samples were prepared by hydrogen reduction of tungsten and rhenium hexafluorides in the composition range 3.5 to 95 at. % Re. The maximum T_c found was 11.4 K at 42 at. % Re, which is considerably higher than about 5 K reported for arc-melted tungsten-rhenium in this composition range. As-deposited samples had higher transition temperatures than comparable

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[23]Abstracted from a paper submitted for publication.

[24]Solid State Division.

arc-melted alloys for the entire composition range, regardless of crystal structure. Two reasons suggested for the enhancement of T_c are: (1) the formation of a Cr_3Si type A15 structure and (2) the presence of impurities trapped in the lattice. After high-temperature annealing the T_c was decreased to that of arc-melted samples. This is attributed to the decomposition of metastable phases to the equilibrium structures and/or the precipitation of gas or gaseous compounds in the form of bubbles. Long-range order parameters of samples with the A15 phase were low and showed the unusual feature of exhibiting increasing order with increasing departure from stoichiometry.

High Critical Current Densities Found for Thick $Nb_{12}Al_3Ge$ Superconducting Deposits Made by High-Rate Sputtering[25]

S. D. Dahlgren[26] and D. M. Kroeger

Superconducting critical current densities up to 5.2×10^5 A/cm^2 at 75 kOe and 4.2 K, and up to 4.4×10^5 A/cm^2 at 100 kOe were measured for a 66-μm-thick $Nb_{12}Al_3Ge$ deposit made at a rate of 1 μm/min by high-rate sputtering. Plots of current density against transverse magnetic field were either linear or nearly linear, and the current density dropped only 33% over the field range investigated (50 to 100 kOe). The sample that carried the highest current densities was deposited at 20°C and heat treated 1 day at 750°C. Current densities for another sample from the same deposit, but heat treated 20 days at 750°C, were about 14% lower than for the sample heat-treated 1 day. The deposits were bcc when deposited at 20°C. Heat treatment at 750°C produced a single metastable A15 phase containing grains 350 Å in diameter; the small grain size presumably accounted for the high current density. Current densities measured with the field perpendicular to the deposit plane were only about 14% lower than with the field parallel to the deposit plane, indicating that the A15 phase grains formed by heat treatment at 750°C were nearly equiaxed. The high current density results and the availability of high-rate sputtering equipment encourage fabrication of practical quantities of sputter-deposited Nb-Al-Ge superconducting products. Estimated fabrication rates and costs were reasonable.

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[25]Abstract of *J. Appl. Phys.* 44: 5595–98 (1973).

[26]Battelle-Northwest, Metallurgy Development Department, Richland, Wash.

New Apparatus Development

C. C. Koch, D. M. Kroeger, J. P. Charlesworth,[27] and D. S. Easton

Considerable effort was expended during the reporting period to improve existing experimental facilities as well as to develop new facilities for ac loss measurements and for the study of the effect of mechanical strain on superconducting properties.

Equipment for Measuring Critical Current Density, J_c

The technique for measuring J_c by the ac harmonic analysis method was improved by a novel compensation method. The same experimental arrangement was also utilized to measure J_c by the ac method first developed by A. Campbell, which provides information on the field distribution in a superconductor. Our ability to measure J_c resistively was enhanced by obtaining a 500-A power supply. We now can measure J_c by four techniques: (1) resistive measurement, (2) dc magnetization, (3) ac harmonic analysis, and (4) ac total flux measurement (Campbell technique). A comparison of the results from these differing techniques on the same sample is in progress.

Equipment for Measuring AC Losses in Superconductors

An apparatus was constructed to allow us to accurately measure low-level losses in superconductors at frequencies of 20 to 500 Hz and magnetic fields up to 2000 Oe.

Equipment for Measuring J_c in Superconductors Under Mechanical Stress

As part of a program to assist the Engineering Sciences Group in the Thermonuclear Division in design of large superconducting solenoids we are assembling equipment to allow us to measure J_c in transverse fields up to 70 kOe at 4.2 K under well characterized mechanical stress. This involves obtaining a split-pair superconducting solenoid, modifying an existing Instron tensile machine, and obtaining appropriate Dewars, power supplies, etc.

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[27]Visitor from AERE, Harwell.

PART 3. DEFORMATION AND MECHANICAL PROPERTIES

C. J. McHargue

The physical and mechanical properties of technological materials are highly sensitive to their structure. We provide a basic understanding of the relationship between structure and these material properties. Specific projects include a study of the effect of oxygen absorption on the mechanical properties of refractory alloys, the thermodynamics of solid solutions, x-ray studies of ordering in solid solution alloys, phase transformation in a ternary uranium-base alloy, and an investigation of the depth dependence of texture in rolled niobium

Low-Pressure Oxygen Absorption by Refractory Metals

R. W. Carpenter

The mechanical properties, including fracture, of bcc refractory alloys are very strongly influenced by oxygen, either in solution or present as a second-phase constituent. A tantalum-tungsten-hafnium ternary solid solution alloy was chosen as a model system to investigate the oxygen absorption behavior of this class of alloys.

The oxygen absorption kinetics and oxygen gradients in this alloy containing hafnium were experimentally determined after oxidation in low-pressure oxygen at 1273 K. At this temperature oxygen diffuses rapidly in the alloy, but metallic alloying elements, such as hafnium, are essentially immobile. The structure of the alloy after oxidation consisted of a homogeneous array of hafnium-oxygen clusters in the bcc lattice. A typical example of this microstructure is shown in the transmission electron micrograph (Fig. 3.1). The kinetic and gradient observations were analyzed in terms of three different models.[1] They were:

1. oxygen absorption rate determined by a time-invarient oxygen mass flow through the gas-solid interface into the alloy;
2. oxygen absorption rate proportional to alloy surface oxygen concentration, which increases linearly with time;
3. oxygen absorption rate controlled by the diffusivity of oxygen within the alloy.

Model 1 correctly predicted the observed absorption kinetics and oxygen gradients. The oxygen profiles developed in specimens with a finite-thickness slab geometry are shown as a function of time in Fig. 3.2., computed from this model. The average oxygen concentration is proportional to time and inversely proportional to specimen thickness. Model (2) predicts nearly the same result for thin specimens and long oxidation times but deviates from the observed behavior at other experimental conditions. In addition, model (2) does not predict the observed linear time

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[1]R. W. Carpenter and C. T. Liu, "Surface Reaction Controlled Oxygen Absorption: Kinetics and Concentration Gradients," submitted for publication.

Photo 1913-73

Fig. 3.1. Electron micrograph of the alloy after oxidation at 1273 K. The average oxygen concentration is 3000 ppm. The intensity modulations, called the TWEED structure, indicate the presence of small coherent hafnium-oxygen clusters. $\bar{g}$ = [020], (001) is foil plane. Scale at lower right shows 500 Å.

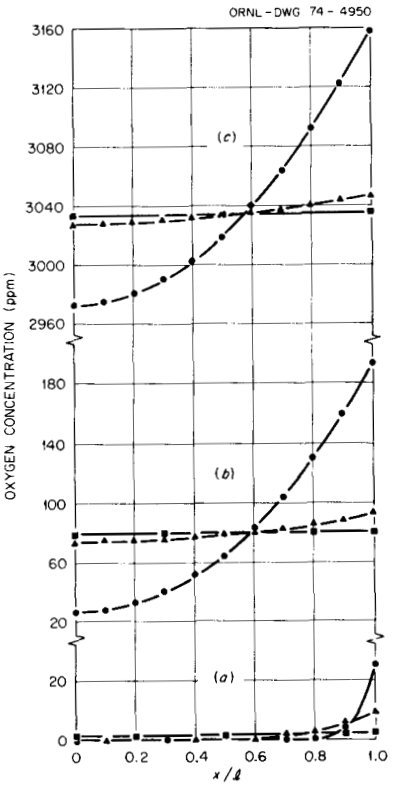

Fig. 3.2. Oxygen concentration profiles computed from model (1) for different times. The different values of the oxygen diffusion coefficient used show its effect on the profile; this diffusion coefficient has no effect on the average oxygen concentration. ●, $D = 3.9 \times 10^{-8}$ cm^2/sec; ▲, $D = 3.9 \times 10^{-7}$ cm^2/sec; ■, $D = 3.9 \times 10^{-6}$ cm^2/sec; (a) 1 min; (b) 1 hr; (c) 38 hr. On the abscissa $x/\ell = 0$ and $x/\ell = 1$ correspond to the specimen center plane and gas-solid interface, respectively.

dependence of the average oxygen concentration. Model (3) predicts an absorption rate much larger than observed.

At higher oxidation temperatures, such as 1673 K, where hafnium as well as oxygen is mobile in the alloy lattice, the absorption kinetics

are the same as described above, but the oxygen gradient is quite different. The gradient differs because metastable hafnium dioxide precipitation occurs concurrently with oxygen absorption, and the alloy consists of two phases.[2]

Solution Thermodynamics

Robin O. Williams

We have previously shown that the unreliability of the entropy deduced from the temperature dependence of the free energy must, in general, be the result of the systematic errors in the free energy measurements.[3] Further, such free energy measurements contain essentially no information about the possible temperature dependence of the entropy or the heat of mixing, a factor that also complicates establishing reliable entropy values.

We are extending this work in several respects. First we have examined a collection of thermodynamic data to establish the relation between the uncertainties in the heat of mixing, the entropy, and the free energy. An outstanding feature of this work is that the uncertainties in the free energy are often greater than the uncertainties based upon the scatter in the data.

We have been examining the relation between the entropy and the heat of mixing. It presently looks as if the entropy is primarily configurational and is represented by the Guggenheim quasi-mechanical theory.

We are also investigating the possibility of formulating simple mathematical models to represent the temperature dependence of solutions that are essentially regular.

Short-Range Order and the Development of Long-Range Order in Ni_4Mo (Ref. 4)

Bhaven Chakravarti,[5] Cullie J. Sparks, Jr., Edgar A. Starke, Jr.,[5] and Robin O. Williams

The short-range-ordered structure present in quenched Ni_4Mo and the initial development of the long-range-ordered structure have been studied by single-crystal x-ray diffuse scattering techniques. The diffuse x-ray intensities were measured in a volume in reciprocal space for the fcc

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[2]C. T. Liu, R. W. Carpenter, and H. Inouye, "Oxygen Distribution in Internally Oxidized Ta–8% W–2% Hf Alloy," submitted for publication.

[3]R. O. Williams, *The Nature of the Limitations of Second Law Determinations of Solution Thermodynamics*, report in preparation.

[4]To be published in the *Journal of Physics and Chemistry of Solids*.

[5]Metallurgy Program, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332.

alloy. This resulted in a quantitative determination of the three-dimensional local-order parameters and the "size effect" coefficients for the first- and second-order displacement effects. The local-order parameters were used for a computer simulation of the atomic configuration.

The local atomic arrangements for the quenched alloy consist of a strong preference for no molybdenum atoms as first neighbors to molybdenum. The atoms tend to satisfy the long-range-ordered structure of Ni_4Mo in which molybdenum has no molybdenum atoms as first neighbors, only one molybdenum atom as a second neighbor, and six or more as third neighbors. A rodlike morphology consisting of a strong preference for (200) and (400) Mo-Mo pairs exists on ordering for 5 min at 650°C. This result is similar to the rodlike morphology present in dilute nickel-tungsten alloys and explains the [100] streaking of the short-range-order peaks observed in the diffraction studies.

Recent Observations of Phase Transformations in a U-Nb-Zr Alloy[6]

R. A. Vandermeer

Dilatometry, x-ray diffraction, hardness, and optical microscopy were used to characterize a polycrystalline uranium alloy containing nominally 7.5 wt % Nb and 2.5 wt % Zr. Upon quenching this alloy from the bcc phase at 800°C, a metastable modification corresponding to a slight tetragonal distortion of the bcc lattice along with a short-range atom displacement ordering was revealed by x-ray diffraction. Minor compositional variations, differing quenching rates, and low-temperature aging cause important alterations in this metastable structural state. Slight depletion of the alloying elements increases the degree of tetragonality. Aging the quenched alloy initiates transformations to other anisotropic structural states. Altogether four transformation stages have been detected by dilatometry. At temperatures as low as 150°C, two stages, one beginning after a few minutes and the other after a few hours, produce a volume expansion of the alloy and a dramatic increase in hardness. The first stage has been attributed to solute clustering. The second stage corresponds to an isothermal martensitic shear transformation, resulting in a second phase that may be described as a monoclinic modification of the α-uranium orthorhombic structure. Cellular or discontinuous precipitation is observed at temperatures between 500 and 620°C. Light rolling deformation of the quenched alloy causes an immediate crystal reorientation in certain grains. Metallographic and x-ray evidence hints that this reorientation is accomplished by mechanical twinning at low strains.

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[6]Abstract of paper presented at the Conference on the Physical Metallurgy of Uranium Alloys, Vail, Colorado, Feb. 11–14, 1974; to be published in the proceedings.

Deformation Zone Geometry and Texture Gradients in Cold-Rolled Niobium[7]

R. A. Vandermeer and J. B. Bernal[8]

Depth-dependent variations in texture have been observed in cold-rolled niobium sheets and plates. The geometry or shape of the zone of plastic deformation in rolling was shown to be an important parameter controlling these texture gradients and inhomogeneous plastic flow. A useful index of the deformation zone geometry is the ratio of zone contact length to mean zone thickness. In this work niobium plates were rolled at room temperature to a total reduction of 60%, with the shape of the zone of plastic deformation maintained constant for each pass through the rolling mill. Zone shapes corresponding to index ratios between 0.5 (high body rolling) and 2 (high surface friction forces) were studied. Pole figures were obtained from selected depths in the thickness direction of the rolled plates by means of the Schulz x-ray reflection technique. Mildly depth-dependent variations in texture were noted for index ratios of at least 1, but strong textural differences, even to the point of a transition to a modified texture in the intermediate regions of the plates, were evident for index ratios below 1. Analysis and description of the resulting preferred orientation were related to the nature of the stress state and deformation patterns imposed upon the plates.

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[7]Summary of paper presented at Spring Meeting of AIME held at the University of Pittsburgh, May 20–23, 1974.

[8]On temporary assignment from Comisión Nacional de Energía Atómica, Buenos Aires, Argentina.

PART 4. RADIATION EFFECTS

J. O. Stiegler

The Radiation Effects Program has as its goal the development of materials showing improved radiation resistance for use in LMFBR, CTR and advanced research reactor applications as well as the understanding of the mechanisms involved in radiation effects at intermediate to elevated temperatures. We use both neutron irradiations and electron and ion bombardment simulation techniques.

Basic Studies on simple materials are carried out in work supported by the AEC Division of Physical Research. Corresponding studies of potential reactor alloys are supported by the AEC Division of Reactor Research and Development and Division of Controlled Thermonuclear Research. In this section we report only the activities supported at least partially by the Division of Physical Research.

Effects of Impurity Level on Electron Radiation Damage in Aluminum at Elevated Temperatures[1]

J. O. Stiegler and K. Farrell

An increase in the general level of impurities affects the formation of loops and the evolution of a dislocation structure in aluminum and nickel during bombardment with 650-kV electrons at elevated temperature in the high-voltage electron microscope. In aluminum at 220°C, decreasing purity increases the concentration of loops, reduces their growth rates, increases the time to reach a steady-state dislocation structure, and reduces the cutoff temperatures for loop formation, indicating that impurities enhance the nucleation of loops and suppress their growth. Annealing and reirradiation experiments just below the cutoff temperature suggest that some loops may be reinitiated at their original sites. This implies nucleation at impurities or stable submicroscopic defects. Large, multiply faulted loops accompanied by considerable dislocation movement develop in the higher purity materials near the surface after prolonged irradiation.

Alignment of Dislocation Loops in Irradiated Metals[2]

J. O. Stiegler and K. Farrell

Dislocation loops created during neutron irradiation of aluminum at 55°C and nickel at 300°C, assume an ordered arrangement. Clusters of loops

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[1]Abstract of pp. 341–47 in *High-Voltage Electron Microscopy* (Proc. 3rd Intern. Conf.), ed. by P. R. Swann, C. J. Humphreys, and M. J. Goringe, Academic Press, London, 1974.

[2]Summary of *Scr. Met.* 8: 651–56 (1974).

form the lattice points of an fcc lattice parallel to but much larger than the metal lattice. The arrangement is highly imperfect and is disrupted in the vicinity of dislocation lines. It does not exist in nickel irradiated at 350°C. There seems to be no spatial relationship between the loop lattice and radiation-induced voids. The observations are compared with an ordered loop arrangement reported for ion-bombarded nickel.

Postirradiation Properties of the 6061-T6 Aluminum HFIR Hydraulic Tube[3]

K. Farrell and A. E. Richt[4]

A tube of 6061 aluminum alloy in a T6 temper, precipitation-hardened with Mg_2Si, was examined after irradiation in the core of the High Flux Isotope Reactor to fluences as high as 1.3×10^{23} neutrons/cm^2 (>0.1 MeV) and 3.1×10^{23} neutrons/cm^2 (thermal) in contact with the cooling water, which is at about 55°C. The alloy displayed less than 0.02% swelling from cavity formation, and these cavities are believed to be gas-filled. Tensile tests in the range 55 to 200°C showed radiation-induced increases in yield and ultimate stresses of 50 to 80%; elongation was reduced from the range 10 to 15% to about 5% at 55°C and to about 3% at test temperatures above 100°C. These changes are attributed primarily to a radiation-induced precipitate of transmutation-produced silicon; about 7 wt % Si was formed. This silicon also caused significant swelling. The fracture mode was changed from transgranular tearing around inclusions to a mixture of transgranular tearing and ductile intergranular separation. The formation of a rim of intergranular cracks at the originally oxidized surfaces of the tube during testing is believed to contribute to the loss in elongation at elevated temperatures.

Effect of High Helium Content on Stainless Steel Swelling[5]

F. W. Wiffen and E. E. Bloom

We investigated the combined effects of high displacement rate and high helium generation rate on the dimensional stability of structural materials proposed for use in fusion reactors. The use of an austenitic stainless steel for the first wall has been examined in at least one conceptual design. Operation with stainless steel at a neutronic wall loading of 1.87 MW/m^2 would result in 30 displacements per atom (dpa)

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[3]Abstract of a paper presented at the Seventh ASTM International Symposium on Effects of Radiation on Structural Materials, Gatlinburg, Tenn., June 11–13, 1974. To be published in Proceedings.

[4]Now with Union Carbide Corp., South Charleston, W. Va.

[5]Summary of *Trans. Amer. Nucl. Soc.* 17: 143–44 (1973).

and generation of 460 ppm He per year of reactor operation.[6] Irradiation of this alloy in the EBR-II produces about 45 dpa but less than 20 ppm He per year. Thus data on swelling obtained in EBR-II do not take into account possible effects of the high helium generation rate in fusion reactor operation. In contrast, irradiation of type 316 stainless steel in the HFIR results in about 60 dpa/year, with about 1900 ppm He produced in the first year.[7]

Specimens of annealed and 20%-cold-worked type 316 stainless steel were irradiated in the HFIR at temperatures between 370 and 680°C to fluences from 3.8 to 8.7×10^{22} neutrons/cm^2 (>0.1 MeV). The microstructure of a sample irradiated at 574°C to 4.2×10^{22} neutrons/cm^2 was examined by electron microscopy. Areas away from the grain boundary contained about 1×10^{14} cavities/cm^3, identical to the concentration predicted[8] for similar EBR-II irradiations. In contrast to samples from EBR-II, this sample also contained a high concentration of cavities on grain boundaries and at precipitate interfaces.

Immersion density measurements are given in Table 4.1. Also given is the predicted swelling for solution-treated material based (1) on EBR-II results,[8] where helium production would be less than 30 ppm, and (2) on the calculated helium content[9] and the assumption that all helium is contained in bubbles at equilibrium pressure in a concentration of 10^{14}/cm^3.

Comparison of the measured and calculated swelling shows that high helium concentrations have the following effects on irradiation-produced swelling of type 316 stainless steel.

1. Swelling of 5 to 15% could be expected in a solution-treated stainless steel fusion reactor first wall in ten years operation at temperatures in the range 350 to 680°C. Cold work will significantly reduce this swelling for temperatures below 650°C.

2. The swelling observed agrees reasonably with that calculated for equilibrium helium bubbles in the one case for which the cavity density is known. This suggests that all cavities are gas bubbles that contain helium at equilibrium pressures, as opposed to the voids produced in EBR-II.

3. Since calculated swelling due to helium bubbles is generally greater than observed swelling, bubble concentrations must be higher than 10^{14}/cm^3.

4. The temperature dependence of swelling is different from that observed in fast reactor irradiations with low helium concentrations. There is no upper temperature cutoff for swelling for high-gas-content material. At constant fluence, swelling will probably increase with increasing temperature above 600°C because of decreasing bubble concentrations and the accompanying significant increase in bubble size.

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[6]P. Bonanos and W. G. Price, Jr., Plasma Physics Laboratory, Princeton University, personal communication, June 1973.

[7]W. N. McElroy and H. Farrar IV, p. 187 in *Radiation-Induced Voids in Metals*, J. W. Corbett and L. C. Ianniello, eds., AEC Symp. Ser. 26, CONF-710601 (April 1972).

[8]H. R. Brager and J. L. Straalsund, *J. Nucl. Mater.* 46: 134 (1973).

[9]R. S. Barnes, *J. Nucl. Mater.* 11: 135 (1964).

Table 4.1. Swelling in Type 316 Stainless Steel Irradiated to High Fluences in the HFIR

Irradiation Temperature (°C)	Fluence, >0.1 MeV (neutrons/cm^2)	Calculated Displacements[a] (dpa)	Calculated Helium Content[b] (ppm)	Swelling, %			
				Calculated		Measured	
				In Fast Reactor Without Helium[c]	Due to Helium Alone[d]	Solution Treated	20% Cold Worked
379	7.05×10^{22}	97	4020	0.71	11	6.7	1.6
456	7.69	107	4820	5.27	16	8.7	0.80
528	8.27	114	5450	7.94	21	8.3	2.6
535	3.79	52	1930	1.17	5.8	3.5	0.52
574	4.21	58	1791[e]	1.05	5.5	3.3	
602	8.71	119	5940	4.50	28	8.0	3.3
679	8.74	121	6090	1.67	34	14.1	16.8

[a]The dpa calculated from Lindhard model. H. T. Kerr and R. Q. Wright, Oak Ridge National Laboratory, personal communication, May 17, 1973.

[b]Atomic concentration calculated by the formula $C = 9.8\ K \times 10^{-19}(\phi t)^{1.68}$ atoms He/g Ni, where ϕt is thermal fluence. Formula from McElroy and Farrar, p. 187 in *Radiation-Induced Voids in Metals*, J. W. Corbett and L. C. Ianniello, eds., AEC Symp. Ser. 26, CONF-710601 (April 1972), with normalization factor $K = 0.65$ determined by measured value (e) for 574°C.

[c]Calculated from empirical formula for swelling of neutron-irradiated solution-treated type 316 stainless steel given by Brager and Straalsund, *J. Nucl. Mater.* 46: 135 (1973). Equations derived from irradiations that resulted in <30 ppm He.

[d]Approximate swelling for all helium in equilibrium bubbles, with constant bubble concentration of $10^{14}/cm^3$. Taken from Barnes, *J. Nucl. Mater.* 11: 135 (1964).

[e]Measured helium content.

5. The tendency for cavities to form on grain boundaries may lead to intergranular failure in stressed components well before maximum swelling is realized.

The Effects of Large Concentrations of Helium on The Mechanical Properties of Stainless Steel[10]

E. E. Bloom and F. W. Wiffen

Austenitic stainless steels and nickel-base alloys have been suggested as possible first-wall construction materials for fusion reactors.[6,11] At a neutronic wall loading of 1.87 MW/m^2 approximately 30 displacements per atom (dpa) and 460 ppm (atomic) He will be produced in stainless steel per year of operation. As little as 10 ppm He can have pronounced effects on mechanical properties, particularly high-temperature (>0.5 T_m) ductility and fracture behavior. The combined effects of very large amounts of helium and high dpa values are unknown and are difficult to investigate. Irradiation of stainless steel in EBR-II produces about 45 dpa but less than 20 ppm He per year. High helium concentrations can be studied with alpha-particle injection techniques, but the displacement damage, which acts synergistically with helium to influence properties, is not created in such experiments. In the High Flux Isotope Reactor helium is produced by a two-step nuclear reaction with thermal neutrons [$^{58}Ni(n,\gamma)^{59}Ni$ followed by $^{59}Ni(n,\alpha)^{56}Fe$], with effective cross sections of 4.2 and 7.4 barns, respectively. In nickel-containing alloys the helium concentration increases[7] as $(\text{fluence})^{1.68}$ and irradiation of stainless steel results in about 60 dpa and 1900 ppm He in the first year. The neutron spectrum and flux available in HFIR thus allow us to approximate displacement and helium production in a stainless steel fusion reactor first wall.

Specimens of annealed and 20%-cold-worked type 316 stainless steel were irradiated in HFIR at temperatures between 370 and 780°C to fluences from 2.2×10^{21} to 8.7×10^{22} neutrons/cm^2 (>0.1 MeV). The density and microstructural changes in these samples were reported previously.[5] Post-irradiation tensile and creep-rupture tests have been conducted near the respective irradiation temperatures. The response of this alloy to irradiation in HFIR can be compared to that observed in previous irradiation experiments[12] conducted in EBR-II.

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[10]Abstract of a paper presented at the First Topical Meeting on the Technology of Controlled Nuclear Fusion sponsored by the American Nuclear Society, April 16–18, 1974, San Diego, Calif. To be published.

[11]G. L. Kulcinski et al., "Materials Radiation Damage Limitations in the Wisconsin Fusion Reactor Design," *Trans. Amer. Nucl. Soc.* 17: 133 (1973).

[12]D. Fahr, E. E. Bloom, and J. O. Stiegler, "Post-Irradiation Tensile Properties of Annealed and Cold-Worked Type 316 Stainless Steel," pp. 167–77 in *Irradiation Embrittlement and Creep in Fuel Cladding and Core Components*, The British Nuclear Energy Society, London, 1973.

Samples irradiated in the annealed condition to about 100 dpa and 4000 ppm He exhibited an increase in yield strength at temperatures between 350 and about 600°C. The increase was smaller, however, than observed in EBR-II-irradiated samples, even though the dpa level was one-half to one-fifth as much in the latter. The ultimate tensile strength at all temperatures was less than the unirradiated value, except at 350°C. The material exhibited little strain hardening, and the total elongation was reduced to below 1% over the entire temperature range.

Samples irradiated in the 20%-cold-worked condition exhibited a decrease in both yield and ultimate tensile stress at all temperatures. The total elongation was only slightly affected by irradiation at 350°C (reduced from 5 to about 4%). However, with increasing irradiation and test temperature, the elongation decreased, becoming zero at 650°C. Limited results on the tensile properties of 20%-cold-worked material as a function of dpa and helium concentration suggest that the decrease in ultimate tensile strength is a result of a decrease in the true fracture stress, which can be more closely correlated with helium concentration than dpa level. For tensile tests at 575°C and creep-rupture tests at 550°C the effects of helium on strength properties and ductility became very pronounced above about 100 ppm.

Effect of Neutron Irradiation on the Microstructure and Properties of Titanium-Stabilized Type 316 Stainless Steels[13]

E. E. Bloom, J. M. Leitnaker, and J. O. Stiegler

Four heats of vacuum-melted type 316 stainless steel with titanium additions of 0.23, 0.33, 0.46, or 0.60 wt % were irradiated in thermal and fast reactor neutron spectra. Samples were irradiated in the Oak Ridge Research Reactor at 650°C to 0.9×10^{21} neutrons/cm^2 (thermal) and 1.4×10^{21} neutrons/cm^2 (>0.1 MeV) and in the EBR-II at temperatures in the range 450 to 850°C to peak fluences of 4.4×10^{22} neutrons/cm^2 (>0.1 MeV). Postirradiation examination included immersion density measurements, transmission electron microscopy, tensile tests, and creep rupture tests. Irradiation effects in these alloys have been compared with standard type 316 stainless steel irradiated under similar conditions.

The swelling as a function of titanium content passed through a minimum for annealed samples irradiated to a fluence of 2.0×10^{22} neutrons/cm^2 (>0.1 MeV). At both 500 and 600°C the 0.33 and 0.23 wt % Ti alloys swelled the least. Furthermore, the alloy containing 0.23 wt % Ti swelled significantly less than expected for standard type 316 stainless steel. For example, the alloy containing 0.23 wt % Ti irradiated in the annealed condition showed density decreases of 0.4 and 0.2% for irradiation at 500 and 600°C, compared with expected values of 0.9 and 0.5% for standard type 316 stainless steel. Slight densification occurred when the alloy

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[13]Abstract of a paper presented at the Seventh ASTM International Symposium on Effects of Radiation on Structural Materials, Gatlinburg, Tenn., June 11–13, 1974. To be published in the proceedings.

containing 0.23 wt % Ti was irradiated in the 20%-cold-worked condition at 500 and 600°C to 3.1×10^{22} neutrons/cm^2 (>0.1 MeV).

For irradiation and test conditions where helium embrittlement limited the mechanical properties (i.e., irradiated and tensile or creep-rupture tested at 0.5 T_m or higher), the 0.23 and 0.33 wt % Ti alloys had the higher maximum ductilities. As expected from previous results on types 304 and 316 stainless steel, the changes in tensile and creep-rupture properties of the 0.23 wt % Ti alloy were sensitive to preirradiation heat treatment, irradiation temperature, and fast neutron fluence. Annealed samples that were irradiated in the temperature range where voids and dislocations form and were tested at temperatures where these defects control tensile behavior (≤500°C) exhibited the expected increase in yield strength and decrease in uniform strain. For these conditions the ductility was similar to that of standard type 316 stainless steel. When irradiated and tested at conditions that would produce intergranular fracture in a standard alloy, the alloy containing titanium generally exhibited higher ductility. Phenomenologically the higher ductility was associated with a decreased tendency for grain boundary fracture.

Chemical analysis, x-ray diffraction, and electron diffraction of precipitates present in the titanium-containing samples show that the first addition of titanium removes essentially all the sulfur and nitrogen from solution as $Ti_4C_2S_2$ and Ti(C,N), respectively. Thermodynamic analysis shows that at the 1050°C preirradiation annealing temperature titanium removes additional carbon, as TiC in proportion to the amount of titanium remaining after precipitation of the sulfur and nitrogen. During irradiation additional TiC is precipitated, the amount decreasing with decreasing temperature because of kinetic limitations.

Effect of Heat Treatment on Fast-Neutron Damage in Type 316 Stainless Steel[14]

E. E. Bloom, J. M. Leitnaker, and J. O. Stiegler

Irradiation-induced swelling and loss of ductility are two major problems in the use of type 316 stainless steel as a fuel cladding and core structural alloy in LMFBRs. Cold working reduces swelling because the high dislocation density generated during working acts as a sink for both vacancies and interstitials.[15] This alloy exhibits low creep-rupture

[14]Summary of *Trans. Amer. Nucl. Soc.* 17: 203–04 (1973).

[15]J. L. Straalsund, H. R. Brager, and J. J. Holmes, p. 142 in *Radiation-Induced Voids in Metals*, J. W. Corbett and L. C. Ianniello, eds., AEC Symp. Ser. 26, CONF-710601 (April 1972).

ductility because of intergranular fracture after irradiation in either the cold-worked or annealed condition.[16] We have recently conducted experiments to determine whether the postirradiation ductility can be improved by control of the preirradiation microstructure while swelling resistance is still maintained by virtue of a high sink density.

Samples of type 316 stainless steel were given various combinations of cold work and thermal aging before irradiation in EBR-II at 500 and 600°C to fluences of 2.5 to 3×10^{23} neutrons/cm^2 (>0.1 MeV). Post-irradiation examination included immersion density measurements, transmission electron microscopy, and creep-rupture tests at 550°C at 55,000 psi and 650°C at 25,000 psi. The various preirradiation heat treatments, irradiation conditions, and results of creep-rupture tests are listed in Table 4.2.

Samples that were cold worked 20% and then aged 10 hr at 550 or 650°C to recover the dislocation structure exhibited relatively small density changes (≤0.2%) when irradiated at either 500 or 600°C. Density increases of 0.5 and 0.6% were observed for 500 and 600°C irradiation temperatures for material that had been aged 100 hr at 700°C (to remove some carbon from solution) and then cold worked 20%. A two-step aging treatment (24 hr at 480°C and 100 hr at 700°C), which recovered the cold-worked structure and caused carbide precipitation, resulted in density decreases of 0.5% at 500°C and 1.0% at 600°C.

For equal irradiation and test temperatures this alloy exhibits minimum creep-rupture ductility at 550 and 650°C in the annealed and 20% cold-worked conditions, respectively.[16] These temperatures result in a combination of maximum matrix hardening and tendency for grain boundary fracture. Aging the 20%-cold-worked material 10 hr at 550 or 650°C before irradiation generally resulted in lower rupture life and higher creep rate compared with the cold-worked condition. These two treatments gave improved ductility, particularly for irradiation and test conditions that give low-ductility intergranular fracture (e.g., annealed samples irradiated at 500°C and tested at 650°C or 20%-cold-worked samples irradiated at 600°C and tested at 650°C). The two-step aging treatment (20% cold work + 24 hr at 480°C + 100 hr at 700°C) resulted in relatively low strength properties, particularly at 550°C, and no better ductilities than from the 10-hr aging treatments at 550 or 650°C. Aging 100 hr at 700°C before 20% cold work did not appear to change significantly the post-irradiation creep properties.

These results indicate that the irradiation-induced swelling and loss of creep-rupture ductility are sensitive to the preirradiation microstructure of the alloy. Combinations of cold work and thermal aging treatments can be used to optimize *both* swelling resistance and ductility.

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[16]E. E. Bloom and J. O. Stiegler, "Effect of Irradiation on the Microstructure and Creep-Rupture Properties of Type 316 Stainless Steel," pp. 360–80 in *Effects of Irradiation on Substructures and Mechanical Properties of Metals and Alloys, Amer. Soc. Test. Mater. Spec. Tech. Publ.* 529, American Society for Testing Materials, Philadelphia, September 1973.

Table 4.2. Effect of Preirradiation Heat Treatment on the Postirradiation Creep-Rupture Properties of Type 316 Stainless Steel

Preirradiation Treatment[a]	Irradiation Temperature (°C)	Neutron Fluence, >0.1 MeV (neutrons/cm^2)	Test Temperature (°C)	Stress		Results		
				(psi)	(MPa)	Rupture Life (hr)	Creep Rate (%/hr)	Creep Elongation (%)
	500	3.0×10^{22}	550	55,000	379	161	0.0074	1.8
	500	3.0	650	25,000	172	450	0.0032	4.5
20% CW	510	1.2	550	55,000	379	2157	0.00096	5.2
20% CW + 10 hr at 550°C	500	3.0	550	55,000	379	1260	0.0024	5.7
	500	3.0	650	25,000	172	138	0.021	8.0
20% CW + 10 hr at 650°C	500	3.0	550	55,000	379	549	0.0058	4.8
	500	3.0	650	25,000	172	951	0.0073	16.7
100 hr at 700°C + 20% CW	500	2.5	550	55,000	379	>4300	<0.0004	>1.7
20% CW + 24 hr at 480°C + 100 hr at 700°C	500	2.5	550	55,000	379	2.5		6.4
	600	3.0	650	25,000	172	458	0.014	15.4
20% CW	565/630	2.3	650	25,000	172	718	0.0004	1.4
20% CW + 10 hr at 550°C	600	3.0	550	55,000	379	57	0.042	3.5
	600	3.0	650	25,000	172	114	0.038	7.8
20% CW + 10 hr at 650°C	600	3.0	550	55,000	379	57	0.047	3.9
	600	3.0	650	25,000	172	111	0.032	6.7
100 hr at 700°C + 20% CW	600	2.5	650	25,000	172	291	0.0013	1.0
20% CW + 24 hr at 480°C + 100 hr at 700°C	600	2.5	650	25,000	172	95	0.069	9.8

[a]1 hr at 1050°C followed by indicated treatment. CW = Reduction in area by cold swaging.

Auger Electron Spectroscopy of Fracture Surfaces in Irradiated Type 304 Stainless Steel[17]

E. E. Bloom and R. E. Clausing

Auger electron spectroscopy was used to study surfaces in type 304 stainless steel fractured after irradiation at 370°C to 1.4 to 10×10^{22} neutrons/cm^2. At the temperatures and strain rates employed, the irradiated samples failed primarily along grain boundaries, although in some cases areas of ductile transgranular fracture were also present. Helium release during fracture was monitored with a residual gas analyzer. The concentration of helium at the boundaries was approximately proportional to the fast neutron fluence to which the samples had been irradiated, being about 0.08 helium atom per grain boundary site at 1×10^{23} neutrons/cm^2. Phosphorus was more concentrated at grain boundaries than on ductile fracture surfaces. Sulfur segregated to surfaces during and after the fracture, making its determination less conclusive. The fact that it segregated to surfaces, however, suggests that it could segregate to grain boundaries. Grain boundary fracture surfaces of irradiated samples were depleted in chromium and enriched in iron and nickel compared with ductile fracture surfaces of unirradiated samples broken at the same temperature.

Applications of Thermodynamics for Predicting and Understanding the Performance of Fast Breeder Reactor Oxide Fuels[18]

J. M. Leitnaker

Intergranular corrosion of stainless steel cladding during fast reactor operation appears to be a limiting factor in design of fuel pins. Presently available data can be treated by a simple rate equation with reasonable agreement and the implication that the rate limiting mechanism is the same in all cases studied. Since some fuel pins perform better than others, the possibility exists that intergranular corrosion can be greatly reduced, thereby increasing the fuel pins' reactor life. Data needed to complete the understanding of this corrosion are described.

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[17]Abstract of a paper presented at the Fourth Bolton Landing Conference on Grain Boundaries in Engineering Materials, to be published in the proceedings. Also published as ORNL-TM-4495.

[18]Abstracted from *J. Nucl. Mater.* 51: 95–105 (1974).

The Crystal Structure of "TiC" Precipitate Formed During Irradiation

J. M. Leitnaker

Electron diffraction observation of a "TiC" precipitate formed during neutron irradiation led to the conclusion that it is not cubic as is TiC obtained from solution-annealed (1050°C) stainless steel. The relationship of some spots with the hexagonal Ti(C,H), and its relationship, in turn, to hexagonal V_2C, which has an orthorhombic modification, led to the successful indexing of the "TiC" precipitate's diffraction patterns on an orthorhomic lattice. Parameters used were: a_0 = 5.05, b_0 = 6.18 and c_0 = 5.33 Å. Not enough hydrogen formed by an (n,p) reaction is present in the samples to form Ti(C,H). It is reasonable to suppose that chromium may be involved in the precipitation, yielding a compound isoelectronic with orthorhombic V_2C.

Some Implications of Size and Spatial Distributions of Voids

J. M. Leitnaker and W. E. Lever[19]

Gaussian Distribution

Plots of void diameter distribution from several irradiated stainless steel samples led to the conclusion that the diameters follow a Gaussian distribution if the smallest voids are eliminated from the plot. Further, the plot of void number density against diameter does not extrapolate through zero at zero diameter. Since it appears from other data that nucleation has not ceased, we conclude there are two distinct void growth mechanisms operating. Additional data should allow us to separate these mechanisms more clearly.

Inhomogeneous Void Densities

Statistical examination of void density concentration data reveals that the voids are often inhomogeneously distributed throughout a given sample. A particularly striking example is shown in Fig. 4.1. The area of low void density is associated with what we deduce are pre-existing TiC precipitate particles. This observation is of potential technological importance.

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[19]Computer Sciences Division.

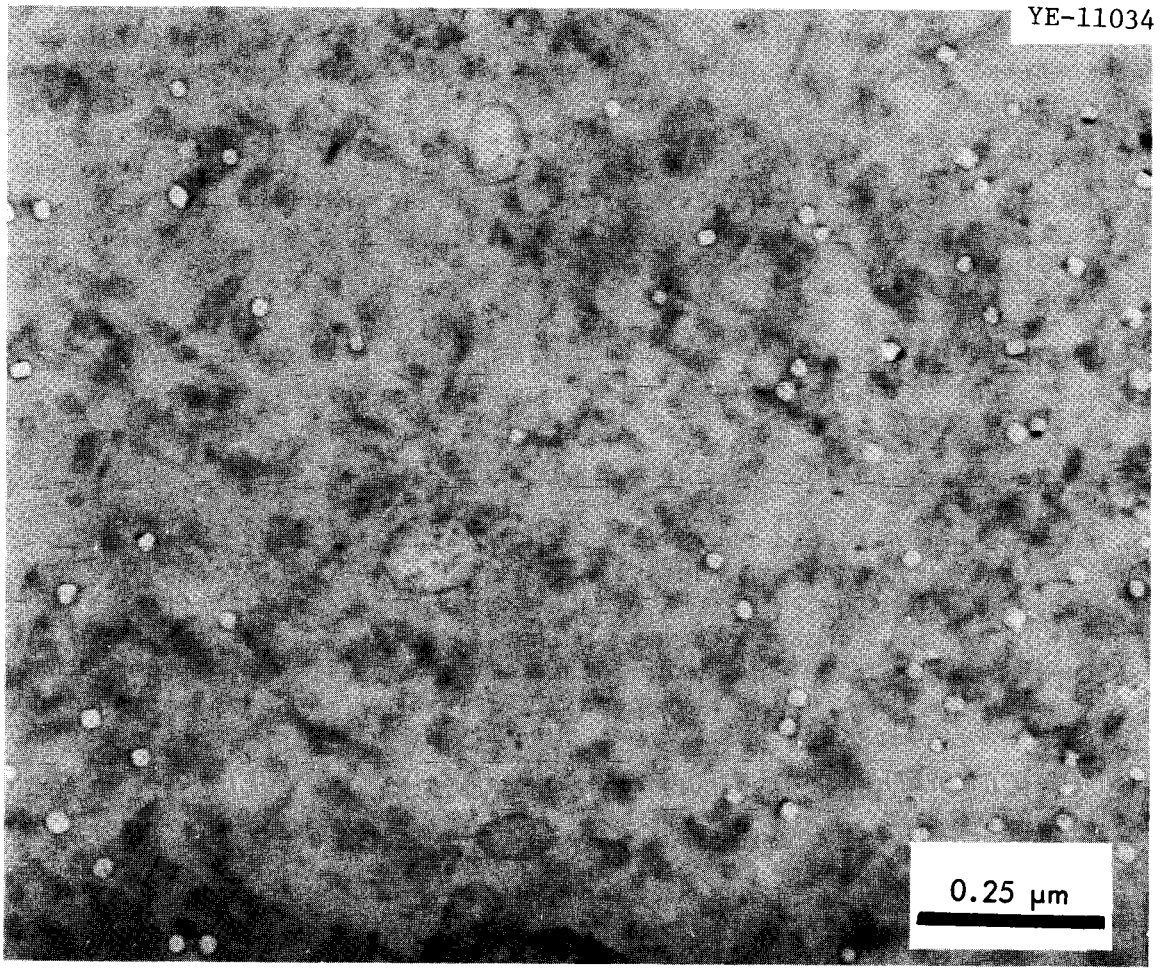

Fig. 4.1. Inhomogeneous Distribution of Voids in a Specimen of Type 316 Stainless Steel Containing 0.33 wt % Ti Irradiated to a Fluence of 1.1×10^{22} Neutrons/cm^2 (<0.1 MeV) at 450°C.

PRESENTATIONS AT TECHNICAL MEETINGS

Compiled by Brenda J. Johnson and Stephanie B. Davison

European Physical Society Topical Conference on "Lattice Defects in Ionic Crystals," Marseille-Luminy, France, July 2–6, 1973

T. S. Lundy* and W. A. Coghlan, "Cation Self Diffusion in Rutile"

G. P. Williams, Jr., Thomas J. Turner,* and Charles S. Yust, "Optical Absorption and Dielectric Loss in Deformed CaF_2"

National Science Foundation and The Japan Society for the Promotion of Science Joint Seminar on "Thermodynamics and Phase Relations of Nuclear Materials," Ames, Iowa, July 9–12, 1973

J. M. Leitnaker,* "Applications of Thermodynamics for Predicting and Understanding the Performance of Fast Breeder Reactor Oxide Fuels"

Information Exchange Meeting on LMFBR/FFTF Control Elements Between Principal Investigators (AI, GE-BRD, HEDL, and ORNL) and WARD Demo Plant Designers, Richland, Washington, July 23–24, 1973

R. G. Donnelly,* "Europia Development — Status and Plans"

International Conference on Positron Annihilation, Helsinki, Finland, August 1973

J. D. McGervey,* R. W. Hendricks, and W. Triftshäuser, "Angular Correlation of Annihilation Radiation from Aluminum Containing Voids"

ASNT Topical Symposium, "Radiography and Complementary Nondestructive Testing in the Nuclear Industry, Portland, Ore., Aug. 1–3, 1973

R. W. McClung,* K. V. Cook, B. E. Foster, and C. V. Dodd, "Development of NDT Methods for LMFBR Steam Generators"

First Annual National Science Foundation Trace Contaminants Conference Oak Ridge National Laboratory, Oak Ridge, Tenn., Aug. 7–10, 1973

J. C. Ogle* and C. J. Sparks, Jr., "Quantitative Trace Element Analysis with X-Ray Fluorescence"

AIME Conference on "Defects and Defect Clusters in BCC Metals and Their Alloys," Gaithersburg, Md., Aug. 14–16, 1973

F. W. Wiffen,* "The Tensile Properties of Fast Reactor Neutron Irradiated BCC Metals and Alloys"

Third International Conference on the Strength of Metals and Alloys, Cambridge, England, Aug. 20–25, 1973

R. W. Carpenter* and C. T. Liu, "Intercrystalline Fracture in an Internally Oxidized BCC Alloy"

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*Speaker

Symposium on Zirconium in Nuclear Applications, sponsored by ASTM and AIME Nuclear Metallurgy Committee, Portland, Ore., Aug. 21–24, 1973

M. H. Yoo,* "Suppression of Void Formation in Zirconium"

The 22nd Annual Denver Conference on Applications of X-Ray Analysis, Denver, Colo., Aug. 22–24, 1973

C. J. Sparks, Jr., J. C. Ogle,* O. B. Cavin, and L. A. Harris, "Quantitative Analysis and Trace Element Determinations by X-Ray Fluorescence with Monochromatic Primary Radiation and Si(Li) Detectors"

Third International Conference on High Voltage Electron Microscopy, Oxford, England, Aug. 27–30, 1973

J. O. Stiegler and K. Farrell,* "Effects of Impurity Level on Electron Damage Radiation in Aluminum at Elevated Temperatures"

Symposium on Materials Performance in Operating Nuclear Systems, Ames, Iowa, Aug. 28–30, 1973

K. Farrell, R. T. King,* A. Jostsons, and E. L. Long, Jr., "Bowing of an Oak Ridge Research Reactor Component"

R. T. King,* K. Farrell, and A. E. Richt, "Creep Properties of Irradiated Aluminum Alloys from the HFIR Surveillance Program"

Central Electricity Generating Board Conference on Physical Metallurgy of Reactor Fuel Elements, Berkeley Nuclear Energy Laboratory, Berkeley, England, Sept. 2–7, 1973

J. O. Stiegler, J. M. Leitnaker, and E. E. Bloom, "Effects of Solute Interstitial Elements on Swelling of Stainless Steel" (presented by K. Farrell)

Third International Conference on X-Ray and Neutron Small Angle Scattering, Grenoble, France, Sept. 5–8, 1973

R. W. Hendricks,* J. Schelten, and W. Schmatz, "A Neutron Small-Angle Scattering Investigation of Voids in Neutron Irradiated Aluminum Single Crystals"

Meeting on Superconductivity and Lattice Instabilities, Gatlinburg, Tenn., Sept. 10–12, 1973

D. S. Easton,* D. M. Kroeger, and C. C. Koch, "Superconducting Transition Temperatures of Chemically Vapor Deposited Tungsten-Rhenium Alloys"

C. C. Koch,* J. O. Scarbrough, and D. M. Kroeger, "Effects of Interstitial Oxygen on the Superconductivity of Niobium"

Conference on Disordered Metallic Systems, Strasbourg, France, Sept. 10–15, 1973

J. S. Faulkner,* G. S. Painter, W. H. Butler, and W. A. Coghlan, "Studies on Disordered Systems Using Clusters of Muffin Tin Potentials"

1973 Meeting Nondestructive Testing Subcommittee of the Air Transport Association of America, Houston, Tex., Sept. 11–13, 1973

K. V. Cook, R. W. McClung,* and H. L. Whaley, "Advances in Electrodischarge Machining of Reference Flaws"

International Union of Pure and Applied Chemistry Symposium, Prague, Czech., Sept. 18–21, 1973

K.H.G. Ashbee* and C.K.H. DuBose, "Plate-Shape Voids in Neutron Irradiated Boron Carbide"

ASM-IMS Symposium on Specimen Preparation Techniques for Optical and Electron Metallography, Beverly Hills, Calif., Sept. 23–24, 1973

C.K.H. DuBose,* "Review of Current Electrolytic Thinning Techniques for Transmission Electron Microscopy"

R. J. Gray,* "Ferrofluid Etching"

R. J. Gray,* R. S. Crouse, and B. C. Leslie, "Decorative Etching"

International Conference on Creep and Fatigue in Elevated Temperature Applications, Philadelphia, Pa., Sept. 23–28, 1973

R. T. King, J. O. Stiegler, and G. M. Goodwin, "Creep Properties of a Type 308 Stainless Steel Pressure Vessel Weld with Controlled Residual Elements" (rapporteur — W. D. Doty — U.S. Steel)

American Institute of Mining, Metallurgical and Petroleum Engineers Fall Meeting, Chicago, Ill., Oct. 1–4, 1973

R. W. Carpenter,* "Internal Oxdiation of Ta–8 W–2 Hf (T-111) Alloy"

W. A. Coghlan* and M. H. Yoo, "A Computer Model for Vacancy and Interstitial Diffusion Near Prismatic Dislocation Loops"

C. T. Liu* and H. Inouye, "Determination of the Distribution and Solubility Limit of Interstitials in Substitutional Solid Solutions"

American Society for Metals Annual Meeting, Chicago, Ill., Oct. 1–4, 1973

H. R. Brager* and J. O. Stiegler, "Effects of Irradiation on Precipitates in Types 316 and 304 Stainless Steel"

Annual Fall Conference of the American Society for Nondestructive Testing, Chicago, Ill., Oct. 1–5, 1973

B. E. Foster, S. D. Snyder, R. W. McClung,* and W. J. Godzinsky, "High Voltage Radiography and Dual Microdensitometry for Evaluating Stressed Rock Specimens"

J. H. Smith and C. V. Dodd,* "Optimization of Eddy-Current Measurments of Coil-to-Conductor Spacing"

American Society for Metals Metals Congress, Chicago, Ill., Oct. 2, 1973

F. W. Wiffen,* "Formation of Void Order in Neutron Irradiated BCC Metals"

SAMPE 5th National Technical Conference, Kiamesha Lake, N.Y., Oct. 9–11, 1973

G. M. Slaughter,* "Recent Developments in the Brazing of Advanced Aerospace Materials"

British Nuclear Energy Society Conference on "Nuclear Fuel Performance," London, England, Oct. 15–19, 1973

J. H. Coobs, W. P. Eatherly, and J. L. Scott, "Testing and Irradiation Performance of Coated Particle Fuels for HTGR's" (presented by H. J. de Nordwall, Chemical Technology Division)

The Fall Meeting of the Nuclear Division of the American Ceramic Society, San Francisco, Calif., Oct. 30—Nov. 2, 1973

R. L. Beatty,* "Oxidizing Diluent for Fluidized-Bed Pyrolytic-Carbon Deposition"

D. A. Canonico, N. C. Cole, and G. M. Slaughter, "Direct Brazing of Ceramics, Graphite, and Refractory Metals" (presented by J. M. Leitnaker)

A. T. Chapman,* J. Brynestad, J. C. Wilson, and G. W. Clark, "Redox Equilibria in Molten Uranium Dioxide"

A. E. Pasto,* M. M. Martin, and R. G. Donnelly, "Fabrication of Europium Sesquioxide"

J. D. Sease,* F. C. Davis, and A. L. Lotts, "Remote Fabrication of High-Temperature Gas-Cooled Reactor Fuels"

Proceedings of the 13th International Conference on Thermal Conductivity, Lake Ozark, Mo., Nov. 5—7, 1973

J. P. Moore,* R. K. Williams, and R. S. Graves, "Precision Measurements of the Thermal Conductivity, Electrical Resitivity, and Seebeck Coefficient from 80 to 400 K and Their Applicaton to Pure Molybdenum"

Proceedings of the 1973 International Thermal Expansion Conference, Lake Ozark, Mo., Nov. 7—9, 1973

Thomas G. Kollie,* D. L. McElroy, J. T. Hutton, and W. M. Ewing, "A Computer-Operated Quartz Differential Dilatometer"

31st Pittsburgh Diffraction Conference, Pittsburgh, Pa., Nov. 7—9, 1973

Bernard Borie,* "The Short Range Structure of Ti and Zr BCC Solid Solutions Containing the Omega Phase"

Southeastern Section of the American Physical Society Meeting, Winston-Salem, N.C., Nov. 10, 1973

W. H. Butler,* "The Electronic Structure of Disordered Transition Metal Alloys"

Fall Meeting of the American Nuclear Society, San Francisco, Calif., Nov. 11—16, 1973

E. E. Bloom,* J. M. Leitnaker, and J. O. Stiegler, "Effect of Heat Treatment on Fast-Neutron Damage in Type 316 Stainless Steel"

R. A. Bradley and E. L. Long, Jr., "Matrix-Particle Interaction in HTGR Fuel Rods" (presented by A. L. Lotts)

D. D. Cannon,* F. C. Davis, R. A. Bradley, and J. D. Sease, "Development of a Laboratory Slug Injection Machine for Fabrication of HTGR Fuel Rods"

D. A. Canonico,* "Fracture Behavior of Thick-Walled Vessels"

N. C. Cole* and G. M. Slaughter, "Brazing of Molybdenum"

J. E. Cunningham* "History of Development of Fuel Elements for MTR"

F. C. Davis,* D. D. Cannon, J. D. Sease, and R. A. Bradley, "Production Slug Injection Machine for Fabrication of HTGR Fuel Rods"

J. H. DeVan* and R. L. Klueh, "The Effect of Oxygen on the Corrosion of Vanadium and V—20% Ti by Liquid Lithium"

W. P. Eatherly, J. L. Scott,* and W. H. Cook, "Graphite for Controlled Thermonuclear Reactor Applications"

T. G. Godfrey,* D. L. McElroy, and Z. L. Ardary, "Thermal Conductivity of Oriented Fibrous Carbon Insulation"

F. J. Homan,* "Timing the Use of Recycle Fuel in the HTGR"

R. T. King, R. G. Berggren, N. C. Cole,* G. M. Goodwin, and G. M. Slaughte "Elevated Temperature Properties of Shielded Metal-Arc Type 308 Stainless-Steel Weld Metals with Varying Ferrite Contents"

R. T. King. G. M. Goodwin, and J. O. Stiegler, "Heterogeniety of Properties and Microstructure in a Thick Stainless-Steel Weldment" (presented by N. C. Cole)

R. L. Klueh,* "Effect of Carbon on the Creep-Rupture Properties of 2 1/4 Cr-1 Mo Steel"

R. E. McDonald, J. R. DiStefano, and A. J. Moorhead,* "Fabrication Procedures for Molybdenum"

J. P. Moore, R. S. Graves, and D. L. McElroy, "Thermal and Electrical Conductivities of Unirradiated and Irradiated Graphite from 300 to 1000 K" (presented by T. G. Godfrey)

R. A. Olstad,* R. B. Fitts, A. R. Olsen, and E. L. Long, Jr., "A Thermally Severe Irradiation Test of Candidate HTGR Recycle Fuels"

F. W. Wiffen* and E. E. Bloom, "Effect of High Helium Content on Stainless-Steel Swelling"

F. W. Wiffen,* "Neutron Irradiation Damage in Niobium and Nb—1% Zr"

Winter Meeting of the American Society of Mechanical Engineers, Detroit, Mich., Nov. 12—15, 1973

H. E. McCoy, Jr., and R. D. Waddell, Jr., "Mechanical Properties of Several Products from a Single Heat of Type 304 Stainless Steel" (presented by R. T. King)

J. O. Stiegler, R. T. King,* and G. M. Goodwin, "Effect of Composition on Fracture Characteristics and Creep Ductility of Type 308 Stainless Steel Weld Metal"

American Society for Testing Materials E-9.08 Task Group Meeting on Cyclic Stress-Strain Analysis, Bal Harbor, Fla., Dec. 4, 1973

R. W. Swindeman,* "Uniaxial Cyclic Data Requirements for ORNL Structural Design Methods Program"

3rd New Zealand Science of Materials Conference, Institute of Physics and Physical Society, Christchurch, New Zealand, Dec. 10—14, 1973

A. Wolfenden,* K. Farrell, and J. O. Stiegler, "Some Effects of Impurities and Microstructure on Radiation-Induced Swelling Metals"

Conference on Surface Effect in Controlled Thermonuclear Fusion Devices and Reactors, Argonne National Laboratory, Argonne, Ill., Jan. 10–12, 1974

R. E. Clausing,* C. J. McHargue, and J. E. Spruiell, "Some Remarks on the Importance of Contaminants in High Temperature Applications of Niobium and Vanadium"

Forty-First Annual Meeting of the American Academy of Orthopedic Surgeons, Dallas, Tex., Jan. 17–22, 1974

R. J. Gray* and Paul Spray, "Metallographic Examinations of Retrieved Intramedullary Bone Pins and Bone Screws from the Human Body"

The Physical Metallurgy of Uranium Alloys Conference, Vail, Colo., Feb. 12–14, 1974, sponsored by the AEC Army Material and Mechanical Research Center

J. V. Cathcart,* "Gaseous Oxidation of Uranium Alloys"

R. A. Vandermeer,* "Recent Observations of Phase Transformations in a U-Nb-Zr Alloy"

H. L. Yakel,* "A Review of X-Ray Diffraction Studies in Uranium Alloys"

1974 National Association of Corrosion Engineers Corrosion Research Conference, Chicago, Ill., Mar. 4–8, 1974

J. P. Hammond,* P. Patriarca, G. M. Slaughter, and W. A. Maxwell, "Chloride Stress-Corrosion of Steam Generator Materials and Post-Test Analysis of an Incoloy 800 Loop"

H. S. Issacs,* "The Initiation and Early Growth of Pits"

J. W. Testor* and H. S. Issacs, "Multicomponent Ionic Effects in Localized Corrosion"

Philadelphia Meeting of the American Physical Society, Philadelphia, Pa., Mar. 25–28, 1974

W. H. Butler,* "The Electronic Structure of Alloys from Cluster Calculations"

J. S. Faulkner,* G. S. Painter, and W. H. Butler, "Electronic States of Sub-Stoichiometric Compounds"

G. S. Painter,* J. S. Faulkner, W. H. Butler, and W. A. Coghlan, "Multiple Scattering Cluster Calculations of the Densities of States of Some Transition Metal Compounds"

Annual Meeting of the Southeastern Section of the Geological Society of America, Atlanta, Ga., Apr. 2–3, 1974

Otto C. Kopp* and Edwin Roedder, "An Independent Check on the Validity of the Pressure Correction in Inclusion Geothermometry, Using Hydrothermally Grown Quartz"

First Topical Meeting on the Technology of Controlled Nuclear Fusion, American Nuclear Society, San Diego, Calif., Apr. 16–18, 1974

E. E. Bloom and F. W. Wiffen,* "The Effects of Large Concentrations of Helium on the Mechanical Properties of Stainless Steel"

General Meeting of the American Physical Society Meeting, Washington, D.C., Apr. 22–25, 1974

J. D. McGervey,* V. W. Lindberg, R. W. Hendricks, and W. Triftshäuser, "Positrons in Voids in Neutron-Irradiated Aluminum"

International Union of Crystallography Inter-Congress Conference on Anomalous Scattering, Madrid, Spain, Apr. 22–26, 1974

Cullie J. Sparks, Jr.,* "Contributions to X-Ray Diffuse Scattering Measurements from an Inelastic Scattering Associated with Anomalous Dispersion" (invited paper)

76th Annual Meeting and Exposition of the American Ceramic Society, Chicago, Ill., Apr. 28–May 2, 1974

R. L. Beatty,* "Carbonization and Coating of Fueled Resin Microspheres"

A. J. Caputo* and R. A. Bradley, "Carbonization of HTGR Fuel Rods"

J. R. DiStefano,* "Compatibility of Eu_2O_3 with Type 316 Stainless Steel and Sodium"

W. J. Lackey,* W. H. Pechin, and J. D. Sease, "Recent Progress in Coating HTGR Fuel Particles"

T. B. Lindemer* and R. A. Olstad, "Quantitative Correlation of Observed Fuel Migration in Irradiated HTGR Fuel Particles"

V. J. Tennery* and T. G. Godfrey, "Radiation Self Damage in (U,Pu)N Solid Solutions"

International Discussion Meeting on Studies of Lattice Distortions and Local Atomic Arrangements by X-Ray, Neutron and Electron Diffraction, Kernforschungsanlage, Jülich, West Germany, Apr. 29–May 3, 1974

B. Chakravarti, C. J. Sparks,* E. A. Starke, and R. O. Williams, "Three-Dimensional X-Ray Diffuse Scattering Study of Local Atomic Arrangements in Ni_4Mo"

R. W. Hendricks* and J. Schelten, "Measurement of Relative Void Surface Energies in Irradiated Metals by Small-Angle Scattering"

American Welding Society 55th Annual Meeting, Houston, Texas, May 6–10, 1974

D. A. Canonico,* "The Influence of Cooling Rate on the Microstructure of Annealed 2 1/4 Cr-1 Mo Steel"

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N. C. Cole,* R. G. Berggren, G. M. Goodwin, and R. T. King, "Properties and Structure of Type 308 Stainless Steel Welds with Varying Ferrite Contents"

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