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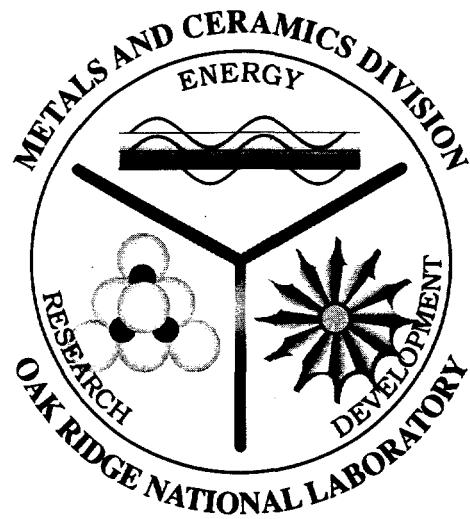
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## OAK RIDGE NATIONAL LABORATORY

**MARTIN MARIETTA**

Metals and Ceramics Division  
Progress Report for Period Ending  
December 31, 1993



MANAGED BY  
MARTIN MARIETTA ENERGY SYSTEMS, INC.  
FOR THE UNITED STATES  
DEPARTMENT OF ENERGY

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METALS AND CERAMICS DIVISION PROGRESS REPORT FOR  
PERIOD ENDING DECEMBER 31, 1993

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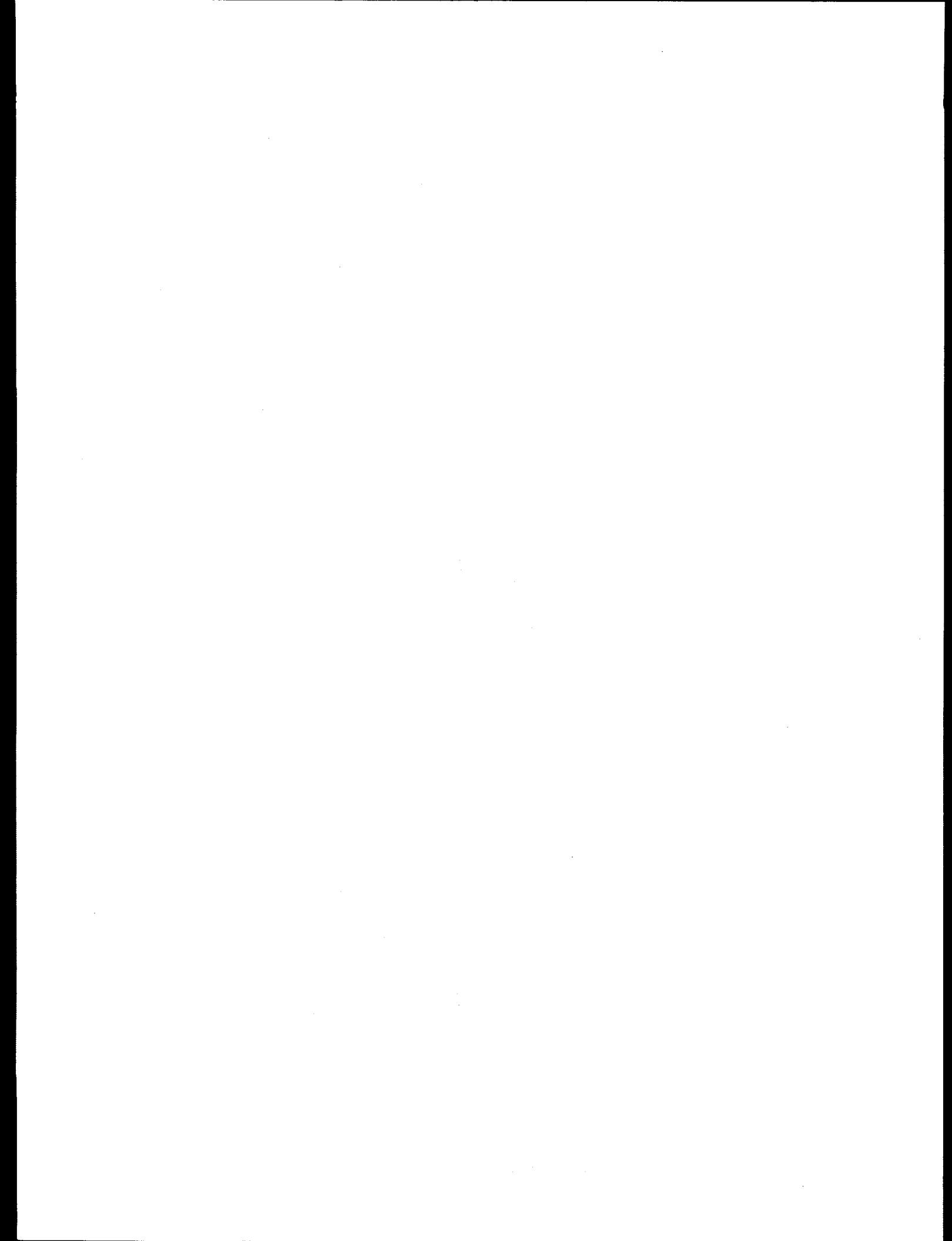
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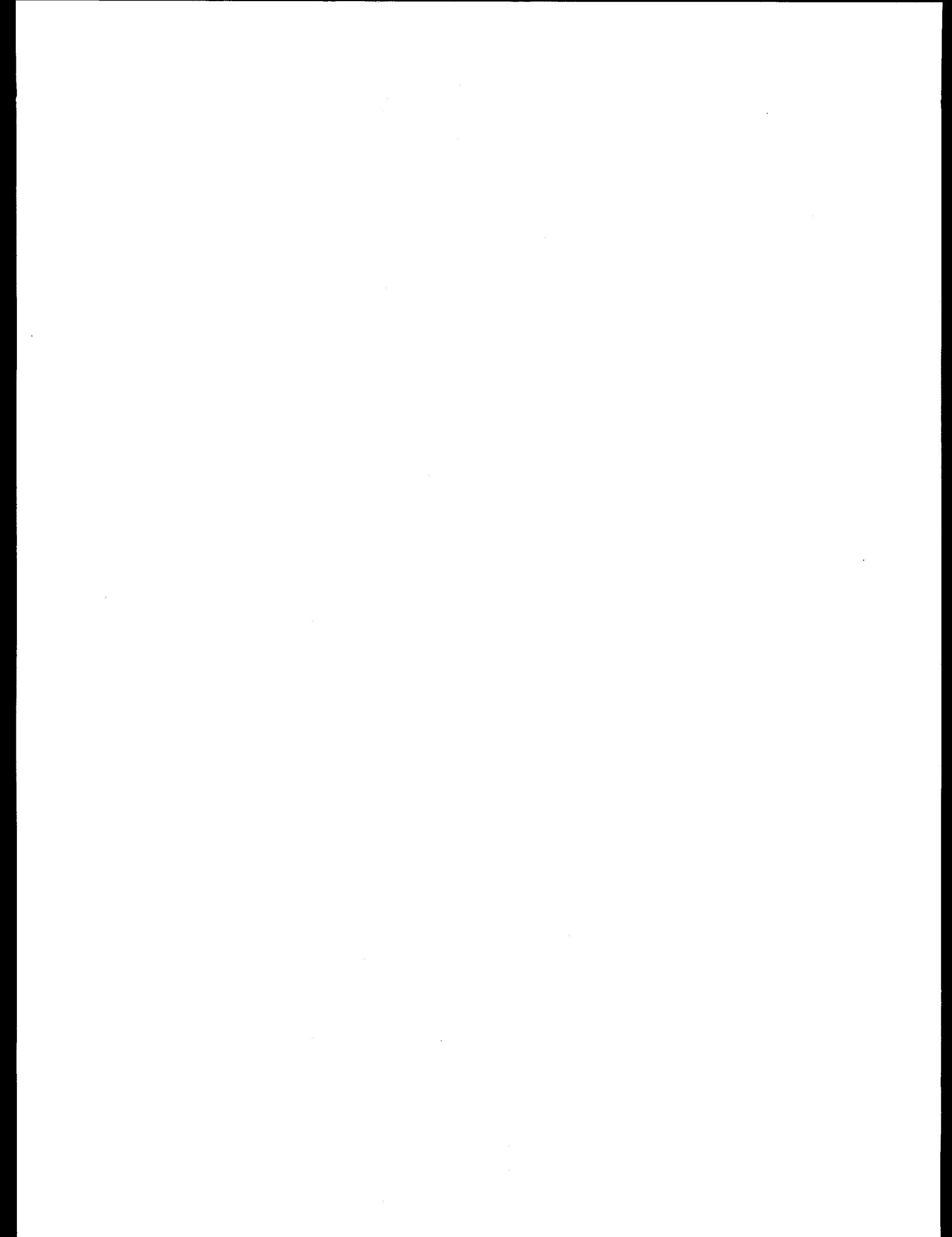


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## OVERVIEW

This report provides a brief overview of the activities and accomplishments of the Metals and Ceramics (M&C) Division during the period October 1992 through December 1993. The division is organized to provide technical support, primarily in the area of high-temperature materials, for the various technologies being developed by the U.S. Department of Energy (DOE). Activities span the range from basic research (through applied research and engineering development) to industrial interactions (through cooperative research and a strong technology transfer program). The division is organized in functional groups that encompass nearly all of the disciplines needed to develop and to apply materials in high-temperature applications. Sections 1 through 5 describe the different functional groups; Sect. 6 provides an alternative view of the division in terms of the major programs, most of which cross group lines; Sect. 7 summarizes external interactions including cooperative research and development programs and technology transfer functions; and Sect. 8 describes the division's involvement in educational activities. Appendices describe the organizational structure, note personnel changes, present honors and awards received by division members, and contain listings of publications completed and presentations made at technical meetings.

During the reporting period, the need arose for several organizational changes. To better position the division to compete for non-defense opportunities in DOE, Ron Bradley was appointed Associate Director of the M&C Division with responsibility for the division's programs involving extensive external interaction with industry, in particular, the Energy Efficiency and Renewable Energy materials programs, Fossil Materials, and Cooperative Research and Development Agreements (CRADAs) with industry. Vic Tennery assumed the new position of Manager of the High Temperature Materials Program, which includes responsibility for the HTML User Program, the HTML Fellowship Program, and the International Energy Agency Program. Arvid Pasto succeeded Vic Tennery as Manager of the High Temperature Materials Section, and Bob Lauf replaced Pasto as Group Leader of the Ceramic Processing Group. After serving as Assistant Manager for more than a year, Wayne Hayden replaced Gerry Slaughter, who retired at the end of the year, as the Engineering Materials Section Manager. Tom Zacharia was named Group Leader of the newly created Materials Process Modeling Group under the Engineering Materials Section. This new group will develop complex models related to materials processing, interact with experimental groups within the division on joint efforts involving materials process modeling, and interface with the Oak Ridge National Laboratory (ORNL) Center for Computational Science. Ed Kenik returned to full-time research and was replaced by Kathi Alexander as Group Leader of the Microscopy and Microanalytical Sciences Group.

So that the division might more effectively apply its materials expertise and facilities to pursuing opportunities in the environmental restoration and waste management areas, and to better accommodate existing reactor materials programs, the Nuclear Fuel Materials Section became part of the Ceramic Science and Technology Section under the leadership of Ron Beatty. To address a change in the focus of research from defects behavior to the science and technology associated with irradiated materials, the Defect Mechanisms Group became the Radiation Effects in Materials Science Group under Lou Mansur's leadership.

The collection of work supporting the different energy technologies in the division strengthens our overall research effort. Development of a new material is a lengthy and expensive undertaking that is often beyond the capabilities of any individual program. Cooperation between programs is enabling us to pursue alloy development activities more vigorously than would be possible otherwise. For example, the Basic Energy Sciences, Fossil Energy Materials, and Advanced Industrial Concepts Materials programs and the Martin Marietta Energy Systems, Inc., Technology Applications Program are all contributing to the development of iron-aluminide alloys. This collaboration has increased the scope of our program and has accelerated the development of these materials. CRADAs have allowed us to strengthen our ties to industry and the relevancy of our research and development (R&D) to the problems of many industrial segments. Fourteen of 28 new CRADAs were approved during the reporting period. Thirteen invited technology transfer presentations were made describing 20 M&C Division technologies. The division's technology transfer exhibit was displayed at seven meetings, and we responded to more than 100 inquiries for information and sample materials.

Several members of the division received major awards. Stan David received the Champion H. Mathewson Award and the Charles H. Jennings Memorial Award. Bob Lauf received the Federal Laboratory Consortium Award for Excellence in Technology Transfer. Bob also received an R&D 100 Award for "Thick-Film Hydrogen Sensor." Wayne Hayden was named Inventor of the Year for the Inventors Clubs of America, Gerry Slaughter was an honored recipient of the Allan Ray Putnam Service Award, and Man Yoo was awarded the Alexander von Humboldt Research Fellowship. Nine division staff members were also named fellows of major societies. Ron Beatty was named a fellow of the American Ceramic Society; Peter Blau, Linda Horton, and Vinod Sikka were named fellows of ASM International; Stan David was named a fellow of the American Association for the Advancement of Science; C. T. Liu was named a fellow of The Minerals, Metals & Materials Society; Cullie Sparks was named a fellow of the American Physical Society; and Stan David and Gene Goodwin were named fellows of the American Welding Society.

A report of this type can do little to capture the excitement of research in the division. It is, at best, an index of activities in our various groups. Contact authors of the various sections for more information on our work or, better still, plan on a visit to discuss it in more detail.

## 1. ENGINEERING MATERIALS

*H. W. Hayden*

This section is responsible for determining and evaluating the suitability of engineering materials for use in various energy systems; for developing and commercializing new engineering alloys; and for determining and developing improved fabrication, joining, and nondestructive testing (NDT) techniques to ensure the structural integrity of materials and components in specific applications. It comprises approximately 70 staff members, about half of whom are professionals. Research and development (R&D) activities are carried out in seven different laboratories, which bear the functional names Corrosion Science and Technology, Fracture Mechanics, Materials Joining, Mechanical Properties, Materials Processing, Nondestructive Testing, and Materials Process Modeling. Additionally, division support for the Heavy-Section Steel Irradiation (HSSI) Program and the High Flux Isotope Reactor (HFIR) Surveillance Program is administered through this section. Brief descriptions of work performed and major accomplishments of these groups and program functions are presented.

### 1.1 HEAVY-SECTION STEEL IRRADIATION PROGRAM — *W. R. Corwin*

Maintaining the integrity of the reactor pressure vessel (RPV) in a light-water-cooled nuclear power plant is crucial in preventing and controlling severe accidents and the potential for major contamination releases. The RPV is one of only two major safety-related components of the plant for which a duplicate or redundant backup system does not exist. It is imperative to understand and predict the capabilities and limitations of its integrity. In particular, it is vital to fully understand the degree of irradiation-induced degradation of the RPV's fracture resistance which occurs during service, since without that radiation damage, it is virtually impossible to postulate a realistic scenario that would result in RPV failure.

For this reason, the HSSI Program has been established by the U.S. Nuclear Regulatory Commission (USNRC) in the Metals and Ceramics (M&C) Division at Oak Ridge National Laboratory (ORNL) to provide a thorough, quantitative assessment of the effects of neutron irradiation on the material behavior and, in particular, the fracture toughness properties of typical pressure vessel steels as they relate to light-water-reactor (LWR) pressure-vessel integrity. Effects of specimen size, material chemistry, product form and microstructure, irradiation fluence, flux, temperature and spectrum, and postirradiation annealing and reembrittlement are being examined on a wide range of fracture properties including fracture toughness ( $K_{Ic}$  and  $J_{Ic}$ ), crack-arrest toughness ( $K_{Ia}$ ), ductile-tearing resistance ( $dJ/da$ ), Charpy V-notch (CVN) impact energy, drop weight nil-ductility-transition temperature (NDT), and tensile properties. Models based on observations of radiation-induced microstructural changes using the atom-probe field-ion microscope (APFIM) and the high-resolution transmission electron microscope (HRTEM) provide improved bases for extrapolating the measured changes in fracture properties to wider ranges of irradiation conditions. The principal materials examined within the HSSI Program are high-copper welds since their postirradiation properties are most frequently limiting in the continued safe operation of commercial RPVs.

Of particular interest are the efforts during the past year concerning the examination of irradiation effects on the fracture and crack-arrest toughness in the low upper-shelf (LUS) weld from the Midland Reactor. Irradiation of the first large capsule, containing about 100 kg of fracture mechanics specimens, to the primary target fluence of  $1 \times 10^{19}$  neutrons/cm<sup>2</sup> ( $E > 1$  MeV) was completed in the newly installed irradiation facility at the University of Michigan Ford Nuclear Reactor (FNR) and a second, large-capsule irradiation begun. Two smaller capsules were irradiated to  $5 \times 10^{18}$  neutrons/cm<sup>2</sup> ( $E > 1$  MeV) to examine fluence effects in LUS welds. Collaborative efforts with a Department of Energy (DOE)-funded program were completed to perform the extensive dosimetry experiments and neutron source-term and transport calculations needed to fully characterize the neutron exposure conditions within the reactor. All of the unirradiated LUS weld material characterization was completed. A wide range of concentrations of impurity-copper levels was documented that is likely to affect the irradiation-embrittlement response of the welds. A significant difference in the average unirradiated fracture toughness levels of nominally identical fabrication welds from different portions of the pressure vessel was also observed even though this difference was not evident from Charpy-impact testing.

Significant advances toward the development of a new fracture toughness testing procedure were made. The proposed test practice is designed to obtain useful fracture mechanics information from small specimens that are of a size suitable for insertion into surveillance capsules. It models transition-toughness data scatter using a three-parameter Weibull distribution model and employs a weakest-link statistical theory to adjust fracture toughness for specimen size effects. A "reference temperature" is determined that positions the master fracture toughness curve for the test material on the temperature coordinate, with affiliated confidence bounds and safety margin adjustments. This procedure, when finalized and codified by the American Society for Materials and Testing (ASTM), will represent the first standardized procedure for obtaining transition-temperature fracture toughness measurements.

Experimental and analytical studies to understand the effects of irradiation exposure parameters were continued. The rate-theory-based embrittlement model previously developed was improved by incorporating precipitation kinetics, and its predictive capabilities were then examined through comparison with existing embrittlement data bases. New experimental efforts were initiated to quantitatively examine the effects of various microstructural hardening centers. Macroscopic hardening is being evaluated by performing nanohardness measurements on a variety of materials exposed to different types of near-surface ion irradiation and comparing the results with observed hardening centers. The results of the hardening study will be used to improve the ORNL embrittlement model. Detailed dosimetry measurements of exposure conditions in the HFIR revealed extremely high gamma-ray flux levels at the surveillance positions that may be eventually determined to have caused the previously observed accelerated embrittlement in the HFIR surveillance specimens.

Significant international collaboration continued during the year. Interactions with the Russians on LWR embrittlement expanded to include interlaboratory testing of ductile-fracture (J-R) resistance-curve testing techniques and the hosting of a visiting scientist from the Kurchatov Institute, Moscow, at ORNL. Joint irradiation and annealing experiments of exchanged RPV materials were continued by both Kurchatov and ORNL. Negotiations with the Japan Atomic Energy Research Institute (JAERI) resulted in an agreed-upon collaborative research agreement for the examination of the RPV from the decommissioned Japanese Power Demonstration Reactor. The

metallurgical, fracture, and dosimetry studies being conducted on this material will provide an enhanced knowledge of embrittlement during actual service and further underpin the overall understanding of irradiation-rate effects on embrittlement. A new initiative was begun with the Nuclear Research Center (SCK/CEN) in Belgium, and the Technical Research Center (VTT) in Finland, that includes reconstituted and miniature impact specimen testing technology, improved means for assessing the irradiation-induced shift in fracture toughness predictions for surveillance applications, and fracture modeling. Finally, a collaborative research effort was initiated with AEA Technology, Risley Laboratories, England, to examine the fracture toughness of RPV materials for surveillance applications using a novel, miniature testing technique—the precracked, round-notched tensile specimen.

The largest area of research growth in the HSSI Program is in examination of the behavior of RPV steels following toughness recovery during annealing and subsequent embrittlement during reirradiation. The focus of this task is to establish the relationship between the changes in fracture toughness of the material needed for engineering evaluations of the vessel and the changes in Charpy impact properties which comprise the vast majority of existing data on annealing and reirradiation. Annealing and reirradiation studies of previously irradiated inventory remaining from earlier HSSI irradiation series were begun, and the design of additional facilities for efficient annealing and reirradiation of new material was nearly completed. This research is providing an improved basis for predicting and regulating the amount of residual toughness that will remain during the continued operation of a pressure vessel after thermal annealing, since annealing is the only practical means for reducing existing embrittlement in an operating pressure vessel.

Results from the HSSI studies will be integrated to aid in resolving major regulatory issues facing the USNRC that involve RPV irradiation embrittlement such as pressurized-thermal shock, operating pressure-temperature limits, low-temperature overpressurization, and the specialized problems associated with LUS welds. Taken together, the results of these studies also provide guidance and bases for evaluating both the aging behavior and the potential for plant-life extension of LWR pressure vessels.

## 1.2 CORROSION SCIENCE AND TECHNOLOGY — *J. R. DiStefano*

The Corrosion Science and Technology Group performs experimental and analytical tasks to support material selections for advanced energy system designs and evaluates components in industrial systems to determine causes of corrosion or failure. These tasks include evaluations of both metallic and ceramic materials in aqueous, gaseous, liquid-metal, and molten salt environments.

During 1993, several new pieces of equipment were acquired that expand the group's corrosion testing capability. Two high-temperature furnaces capable of operating at 1550°C were obtained and gas supply systems designed and constructed in order to evaluate materials for an advanced ceramic heat exchanger. A recirculating water supply system was added for two autoclave systems that enables us to conduct mechanical properties tests on materials in a dynamic aqueous environment. A separate autoclave system, complete with water circulation and mechanical testing capability, was acquired for evaluation of radioactive materials in a hot cell. The accurate measurement of electrochemical phenomena can be important for a range of applications. Recent advances in potential and current measuring instrumentation have heightened interest in

electrochemical noise analysis, and clearly identifiable noise signatures have been reported. Special digital equipment and software applicable to corrosion monitoring have been recently ordered.

A necessary element in the design of the reactor core for the Advanced Neutron Source (ANS) is the characterization of oxide growth on the aluminum alloy clad fuel elements and its impact for the proposed thermal and hydraulic parameters. We have continued to conduct an experimental program involving a test loop whereby prototypic specimens are exposed to high heat flux and coolant flow. This year, we completed testing of the 8001 Al alloy under conditions particularly important to the New Production Reactor-Heavy-Water Reactor (NPR-HWR) Project and discussed the results in the context of the ANS data base in the NPR closeout report. Experiments on 6061 Al and other alternate alloys were resumed after major repair and refurbishment of the loop system. The important features of corrosion for each alternate will be alloy evaluated and compared to the behavior established for 6061 Al.

Corrosion studies of Fe<sub>3</sub>Al-based alloys were continued as part of an alloy development initiative supporting the Fossil Energy Materials Program (FEMP). The alloys being investigated are of two general types. The first is the long-range-ordered Fe<sub>3</sub>Al alloy containing 28 at. % Al with variable additions of Cr, Nb, Mo, and Zr. The second type is a non-ordered alloy containing (in atomic percentages) 16% Al, 5% Cr, 1% Mo, 0.1% Zr, and balance Fe. With each alloy type, the corrosion evaluations are designed to establish the performance limits of the alloys in high-temperature air, combustion, and gasification environments and to determine the mechanisms governing corrosion as a function of alloy content. Although the rate constants in air at 1100 to 1300°C are similar to those of most alumina-forming alloys, the times to breakaway oxidation are considerably longer because of the much larger reservoir of Al in the alloys. Alloying elements other than aluminum have little effect on the oxidation rate constant; however, Zr significantly improves the scale adherence of the alloys, particularly above 1100°C. Tests of the alloys in the simulated gasifier atmosphere containing HCl were performed under subcontract to the National Physical Laboratory. Test results agree with in-house exposures to H<sub>2</sub>S-H<sub>2</sub>-H<sub>2</sub>O gas mixtures and indicate that sulfidation and oxidation of the alloys is not increased by the presence of HCl.

In support of the design of high-strength Cr-Cr<sub>2</sub>Nb alloys for the FEMP, the isothermal and cyclic oxidation behavior of a set of developmental compositions was examined. Alloying additions of Al (up to 18%) or Re (2%) did not improve the isothermal oxidation resistance of Cr-12% Nb, nor did reducing the level of impurities. However, other alloying elements that lead to decreased growth rates and better scale adherence of chromia on chromium and Cr-containing alloys may provide opportunities for improved oxidation resistance. Experiments with several developmental Cr-6 at. % Nb alloys indicated one compositional variation that afforded oxidation resistance as good as any of the Cr-Cr<sub>2</sub>Nb alloys examined previously. A study of the oxidation resistance of Cr-Nb alloys as a function of temperature revealed that the upper limit for effective protection in air by chromia-based scales grown on Cr-Cr<sub>2</sub>Nb alloys will be between 1000 and 1100°C.

A clear challenge in corrosion science is to generate a better understanding of the relationships between composition and the protectiveness of oxides so as to ensure adequate high-temperature corrosion resistance. Proof-of-principle experiments had previously demonstrated the potential of using depth-sensing submicron indentation (DSSI) for measuring elastic and plastic properties of thin oxide products. In 1993, a

more detailed examination using DSSI predicted lower oxide thickness-limits for adherence by scale-yielding than the approach used by others. This finding raised questions about the accuracy of the data being used for such predictions and the adequacy of using modified bulk oxide properties for modeling of scale behavior. Work was also initiated to integrate submicron mechanical testing with microstructural characterization to investigate the fundamental relationships among alloy composition, scale chemistry, structure, adherence, and mechanical properties.

A study of high-temperature environmental effects on fiber-reinforced SiC was continued as part of the Continuous Fiber-Reinforced Ceramic Composites (CFCCs) Supporting Technologies Program. Thermogravimetric analysis was used to study the oxidation reactions associated with air exposures of SiC composites with carbon and BN fiber coatings (interphases). The oxidation resistance of the SiC with carbon-coated Nicalon™ or Nextel® fibers was significantly worse than when BN interphases were used. Oxidation led to significant changes in the mechanical behavior of SiC composites with carbon interphases. Room-temperature flexure testing of Nicalon™/carbon/SiC previously oxidized in air at 950°C revealed that degradation of fracture resistance can be detected after very short exposure times (less than 1 h). Accordingly, a knowledge of reaction kinetics is important in characterizing environmental effects on fracture and damage and in predicting the rate of properties degradation. Technical coordination was also provided for projects at ORNL, Idaho National Engineering Laboratory (INEL), the University of Cincinnati, and Virginia Polytechnic Institute covering time-dependent behavior, environmental effects, fracture and damage, thermal shock, and performance simulation.

Studies continued to determine the extent of degradation of ceramic materials in high-temperature heat exchanger environments. An examination was completed on a second set of ceramic-ceramic composite samples that had been exposed in an industrial hazardous waste incinerator. Some interaction between the environment and the ceramic fibers was found to occur with some ceramic matrices. Two test systems were assembled to permit high-temperature, high-pressure studies of ceramic tubes in steam-methane reformer environments. Initial studies showed the extent of reaction of silicon carbide increased with increasing temperature and/or steam partial pressure.

An assessment of the materials research needs for fuel cells for stationary and transportation applications was performed by a staff member of the group. The intent was to define areas where materials development could make a significant contribution to the improvement in system performance of fuel cells. In association with Argonne National Laboratory (ANL), an "Ad Hoc Workshop on Ceramics for Li/FeS<sub>2</sub> Batteries" was held. This battery system is one of the candidate batteries chosen by the United States Automotive Battery Consortium. The format of the workshop allowed for interaction between DOE Headquarters, the battery developers, the national laboratories, and the ceramic industry, and laid the foundation for a plan for attacking the materials problems associated with this battery system.

The DOE has launched a broad-based technology program to upgrade the thermal efficiency and environmental quality of land-based gas turbine engines for power production. Using laboratory discretionary funding, we participated in the development of a national strategic plan to guide the development of alloys and coatings which will be needed to meet the performance goals of this Advanced Turbine System (ATS) Program. This plan is currently under review by U.S. turbine manufacturers and industrial

metals suppliers and will become the basis for federal and industrial materials development programs starting in fiscal year (FY) 1995.

An assessment of materials problems in the pulp and paper industry was completed. In coordination with a black-liquor recovery boiler manufacturer and a paper company, a proposal was written for a cooperative study of recovery boiler corrosion problems. In addition, laboratory examination of selected samples of composite tubes and black-liquor nozzles from paper mill recovery boilers was initiated.

Gas-metal reaction studies of V-5Cr-5Ti are being conducted to evaluate hydrogen contamination effects associated with plasma-first wall interactions and with prototypic diverter conditions in a fusion reactor system. Two different heats of the V-5Cr-5Ti composition are being studied—one supplied by ANL and the other a laboratory-scale heat made at ORNL. Hydrogen uptake is measured by weight change and by chemical analysis, and effects on mechanical properties are determined by room-temperature tensile tests. Although both heats show the same dependence of hydrogen uptake on hydrogen pressure, the effect of a given hydrogen concentration on mechanical properties is much greater for the ANL heat. The room-temperature ductility of the ANL heat is reduced to zero after 100 h of exposure to  $H_2$  at  $10^{-3}$  torr. The ORNL heat shows no significant ductility loss under the latter condition, but its ductility is reduced from 30 to 20% after 100 h of exposure at  $3 \times 10^{-2}$  torr. Oxidation studies at 500°C are now under way to determine whether simultaneous oxygen contamination and associated aging reactions at the exposure temperature may be contributing to the observed hydrogen effects. Lithium capsule tests are also under way to evaluate the compatibility of AlN coatings on V-5Cr-5Ti in the presence of lithium at 450, 500, and 550°C.

We have concluded experimental testing to evaluate the corrosion performance of Nb-1Zr and Nb-1Zr-0.1C for space reactor applications. Guidelines for the vacuum environment during ground testing were established to prevent attack of the materials by lithium contained in a piping system. Long-term oxidation/corrosion testing was completed to better define the conditions under which formation of  $ZrO_2$  within the alloy structure would mitigate corrosion effects.

Staff members have also provided support to various organizations within Energy Systems in the form of consulting, failure analysis, and materials recommendations. Corrosion of stainless steel tubing in a low-level (radioactive) waste (LLW) evaporator was studied, and alternate materials were recommended. A staff member served as a member of a team which regularly inspected the steel drums containing K-25 pond waste, and he provided input on the environmental degradation of the drums. We assisted the Oak Ridge K-25 Site in investigating the failures of four steel  $UF_6$  storage cylinders and completed an interim report on the failure incidents. A topical report is now being prepared to document the failure causes and remedial actions. Consulting work was provided to the Chemical Technology Division (CTD) with respect to the behavior of 316 stainless steel that was to contain boiling zinc bromide solution. It was determined that while zinc plated out of solution, the performance of the steel was not compromised.

### 1.3 FRACTURE MECHANICS — *R. K. Nanstad*

The Fracture Mechanics Group investigates the fracture resistance of structural materials, particularly steels for pressure-vessel applications. This requires expertise in experimental fracture mechanics and metallurgy. Programs are sponsored

by both the USNRC and the USDOE. In 1993, we emphasized the materials property needs for the Modular High-Temperature Gas-Cooled Reactor (MHTGR) under the NPR Program, Heavy-Section Steel Technology (HSST) and HSSI Programs, ANS, Magnetic Fusion Energy (MFE), FEM, Advanced Industrial Concepts (AIC) Materials, and the Materials for Light-Weight Vehicles Programs.

For the MHTGR-NPR Program, irradiation effects studies for the steel RPV and projects to investigate the high-temperature fracture mechanics properties of structural materials for the steam generator and reactor internals were continued. Mini-tensile and automated ball indentation (ABI) tests for yield strength and flow properties were completed for specimens previously irradiated and were in good agreement with those from standard tensile specimens. Irradiations of two capsules containing A 508, class 3 forging steel and both high- and low-copper welds were completed, as was a capsule containing the medium-copper A 533, grade B, class 1 plate steel. Irradiation of all three capsules was accomplished by Materials Engineering Associates at the University of Michigan's FNR. A specification for irradiation of specially designed capsules incorporating tailoring of the neutron spectrum was developed, and preliminary calculations demonstrated the feasibility of such experiments in the FNR. For the high-temperature fracture mechanics studies, test methods development continued, along with completion of procurement and final assembly of additional equipment such as high-temperature environmental chambers, induction heating systems, specially designed and constructed susceptors for specimen heating, and electronic controls for the two servohydraulic machines. The two systems, designed for fatigue crack-growth and fracture toughness testing at high temperatures and in a helium environment, were checked out and verified to function as designed.

For the HSST Program, the task regarding margin assessments during startup and shutdown operations in commercial LWRs continued. The results of the studies will be used by the USNRC to decide whether to relax the current requirements upon which pressure-temperature limits are based. The task involves identification and characterization of the fracture toughness of potential local brittle zones in weldments of reactor vessels, analysis of the significance of cleavage pop-ins, and a comparison of fracture toughness from standard fatigue precracks and arrested cracks. The project involves cooperative research with Battelle Columbus Laboratories and the University of Maryland. Variations in yield strength and flow properties in local brittle zones [heat-affected zones (HAZs)] in weldments of reactor vessels were measured using ABI tests. For our continuing investigation of the role of specimen size effects in elastic-plastic fracture mechanics, experiments were conducted to provide additional data for statistical analyses and for analyses of the limitations of small-scale yielding criteria in cleavage fracture toughness measurements. Development of a draft standard test method for transition region fracture toughness testing of pressure-vessel steels under the jurisdiction of the ASTM continued. The draft standard has been presented to the appropriate ASTM E8 task group and subcommittee for review. The draft standard relies on the positioning of a master curve for fracture toughness versus test temperature. The positioning of the master curve is accomplished by testing a number of specimens [e.g., six 1/2TC(T)] at a temperature predicted to result in a mean  $K_{Ic}$  of 100 MPa/m, and adjusting for specimen size. The method also includes procedures, then, for determining confidence curves. The procedure is considered particularly applicable to testing small fracture toughness specimens contained in surveillance capsules of nuclear power

reactors. Detailed fractographic studies were conducted to determine the cleavage origin location relative to the final precleavage crack tip as well as the length of the blunting zone on the cruciform specimens tested to evaluate biaxial loading effects. These measurements will be used to compare various methods of determining the crack-tip opening displacement and for determination of the maximum stress location as part of a project to investigate the effects of biaxial constraint on fracture behavior. We also initiated studies to develop base metal and stainless steel cladding with mechanical properties similar to those exhibited by irradiated pressure-vessel steels. These materials will be used in the conduct of structural tests within the HSST Program as part of the development of fracture mechanics models. Development of the base metal incorporated the use of heat treatment to obtain a higher yield strength and a relatively high transition temperature. Development of the cladding will likely incorporate the use of an austenitic material with low ductile tearing toughness and which will likely be overlaid on the base metal with explosive bonding to minimize the formation of a HAZ.

For the HSSI Program, studies continued on a number of tasks, the largest of which is the Tenth Irradiation Series concerning irradiation effects on the LUS weld from the reactor vessel of the canceled Midland Unit 1 nuclear plant. Other tasks include (1) investigation of thermal annealing as a means to recover radiation embrittlement; (2) the use of reconstituted and subsize Charpy impact specimens to determine embrittlement; (3) continuing investigation of the irradiation-induced shift and shape of the fracture toughness curves; (4) procurement and installation of a computer numerically controlled milling machine for machining of mechanical test specimens from irradiated material; (5) effects of irradiation on the dynamic fracture toughness of stainless steel cladding; (6) formal collaboration with researchers in Belgium, England, Finland, Japan, and Russia; and (7) effects of thermal aging on type 308 stainless steel welds and weld overlay cladding. Section 1.1 of this report contains a more comprehensive discussion of these tasks.

Fracture toughness tests were continued with advanced nickel aluminides for the AIC Materials Program and with advanced iron aluminides for the FEMP. For a cast  $\text{Ni}_3\text{Al}$  alloy, the impact properties were shown to decrease at high temperatures (800°C). High-temperature exposure also degraded subsequent room-temperature impact behavior. Efforts on the  $\text{Fe}_3\text{Al}$  alloy system have focused on reduced-aluminum alloys with aluminum content as low as ~8%. The effects of grain refinement are being examined in an effort to improve the transition temperature of these alloys. These testing programs have demonstrated the necessity of evaluating the fracture toughness properties of all the materials proposed for structural applications.

For the ANS Program, the irradiation program to determine mechanical property and fracture toughness data for 6061-T651 aluminum continued. Testing of the first group of specimens irradiated to  $1 \times 10^{22}$  neutrons/cm<sup>2</sup> (thermal) in the HFIR has revealed only a slight irradiation-induced increase of the yield strength and a slight decrease of the fracture toughness. Irradiation of the next capsule to a higher neutron fluence ( $8 \times 10^{22}$  neutrons/cm<sup>2</sup>) has been completed. This capsule is being disassembled in preparation for specimen testing. A series of weldments has been fabricated, and specimens have been machined from these weldments. A third capsule containing these specimens is presently being irradiated in the HFIR to  $8 \times 10^{22}$  neutrons/cm<sup>2</sup>. The results

from the irradiation experiments will be used to make judgments regarding replacement schedules for the core pressure boundary tube (CPBT) in the reactor.

For the International Thermonuclear Experimental Reactor (ITER) Program, miniature disk-shaped compact specimens (about 5 mm thick and 12 mm in diameter from several variants of type 316 austenitic stainless steel) have been irradiated to approximately 3 displacements per atom (dpa) in the HFIR at either 90 or 250°C. Testing of irradiated specimens has begun with successful tests having been conducted from room temperature to 250°C.

#### 1.4 MATERIALS JOINING — S. A. David

The Materials Joining Group continues to conduct R&D for the Basic Energy Sciences (BES), AIC Materials, Space Nuclear Power (SNP), Fusion Energy, and FEM Programs. In addition, the group provides a wide variety of joining-related services to the Oak Ridge Y-12 Plant and other divisions at ORNL.

The in-house BES Fundamental Welding Science Program investigates the physical metallurgy of weldments. Combined with mathematical modeling of transport and phase transformation phenomena, the program tries to achieve a predictive capability for weldment microstructure and properties. The program has as its objectives: (1) to understand the solidification behavior of weld metal and its relation to weld defect formation, (2) to obtain an insight into the fundamentals of phase stability in stainless steel weldments, (3) to develop correlations between welding conditions and weld metal structure and properties by means of modeling, (4) to develop and verify mathematical models describing transport and phase transformation phenomena in weld processes, and (5) to extend the fundamental principles of welding metallurgy to industrial practice in order to promote improved productivity and reduce costs.

A finite difference model has been developed to evaluate diffusion-controlled transformation behavior in ternary systems. This model has been applied to study the ferrite growth during isothermal aging of austenitic stainless steel welds. There is a good correlation between the model predictions and the observed experimental results. The model capability is being extended to consider various geometries as well as non-isothermal aging.

A new area of research on the formation and distribution of oxide inclusions in ferritic steel weldments was initiated. Ladle steel deoxidation reactions were extended to inclusion formation in steel weld metal. Theoretical time-temperature-transformation diagrams were calculated for various oxide inclusions based on overall kinetics approach using nucleation and growth rate expressions.

We are continuing to use single-crystal welds in order to increase our understanding of the factors that influence the development of fusion zone (FZ) microstructures. Electron beam (EB) welding and extensive metallographic characterization were used to study the formation and selection processes in dendritic microstructures that occur during the welding of two differently oriented Fe-15 Ni-15Cr single-crystal pieces. Six orientation

configurations were selected: an (001) surface with a [100] or [110] direction; an (011) surface with a [100], [011], or [211] direction; and a (111) surface with a [211] direction. Using all of the possible combinations of two different crystal orientations (i.e., 15 combinations), two crystal halves were welded along their boundary using an EB to form longitudinal bicrystal welds. Experimental and simulated metallographic sections have been directly compared for 6 of the 15 bicrystal weld configurations. The dendritic pattern observed in transverse metallographic sections was analyzed for each single-crystal half on the basis of a selection criterion that had been developed previously for single-crystal welds; i.e., at a given location in the melt pool, the selected <100> dendrite trunk has the minimum undercooling or, equivalently, the minimum velocity. In general, the bicrystal weld microstructure is a simple composite of microstructures expected in each of the two single-crystal halves. However, near the weld centerline, it was shown that grain competition between dendrites belonging to different single crystals also occurred according to this same minimum undercooling criterion. Some of these results are being extended to a defense program (DP) cooperative research and development agreement (CRADA) program with Westinghouse Electric Corporation.

For the Radioisotope Thermoelectric Generator (RTG) Space Program, test procedures have been established to assess the relative weldability of iridium alloys on a batch-to-batch basis. To date, over 40 production batches have been evaluated, showing a wide range of hot-cracking sensitivity as measured by the sigmajig test. Recycled iridium alloy has been shown to have a higher sensitivity to hot-cracking than virgin material for reasons that are not yet fully explained. The sigmajig test is also being used to characterize developmental iridium alloys.

The MFE Program has involved development of welding procedures and parameters and weldment properties evaluations for the ITER, with emphasis on thick-section welds. The electroslag welding process is being used to join stainless steel up to 8 in. (200 mm) thick in a single pass. Mechanical properties tests include yield strength, tensile strength, toughness, and fatigue crack-growth rate at room temperature and 77 K. Electrogas welding is also being evaluated for several ITER components.

The reference blanket module design for the ITER Project was changed this year to require continuous welds between modules instead of the multiple welded connections of previous designs. A review of welding processes was conducted, and the electrogas process was determined to have characteristics which may be applicable to this new design concept. Encouraging results were obtained from preliminary weldments made in 50-mm-thick stainless steel plates. Modifications to the welding process are being studied to adapt this process to the new design concept.

The AIC Materials Program emphasizes development of weldable FeAl-type alloys, both for use as monolithic structural materials and as weld overlay cladding on stainless or low-alloy steel substrates. Resistance to hot-cracking in these alloys was found to be variable and dependent upon alloy composition (particularly boron, carbon, and niobium). Excellent weldability was achieved with some compositions, and cold-cracking could be avoided for all compositions by the use of preheat and postweld heat treatment (PWHT).

Efforts are under way to develop a shielded metal arc (SMA) electrode formulation to deposit some of the desired alloy compositions, partially funded by a small business CRADA.

The FEMP has been concerned with improving the weldability of  $Fe_3Al$ -type alloys. A wide range of hot-cracking sensitivity was determined using the sigmajig test. Additional alloy compositions are being evaluated with the goal of optimizing both mechanical properties (primary creep strength) and weldability. As with the higher aluminum content alloys, preheat and PWHT are necessary to avoid cold-cracking. The role of hydrogen in the cracking mechanism is being studied extensively.

Fabrication of the SSN-21 SEAWOLF propulsor was completed this year. The propulsor was a very successful project for Martin Marietta Energy Systems, Inc. A detailed analysis and characterization of weld defects present in nickel-aluminum bronze were conducted, and the results correlated with ultrasonic test indications. The results of this study should produce significant cost savings for future propulsors due to relaxation of the nondestructive examination (NDE) acceptance criteria and reduction of repair welding. Technology transfer activities are under way to assist the new contractor building components for the SSN-22 propulsor. Additional projects for the Navy are being examined and estimates prepared as they are requested of Energy Systems.

Joining support continued for the ANS corrosion test project, which is examining the oxidation behavior of aluminum alloys under expected thermal-hydraulic conditions. Additional alloys having potential application for this reactor were fabricated into test specimens this year.

The general-purpose heat source (GPHS) iridium-clad vent set production at the Y-12 Plant was reinstated using the new procedures and corrective actions identified by the extensive program review during the previous year. Welding fabrication by the laser and EB processes has proceeded with excellent results. Technical surveillance was conducted monthly during 1993 to assure the continued production of high-quality components.

Ceramic brazing studies continue to be supported by the Ceramic Technology Project (CTP), and the emphasis of this activity during FY 1993 was on high-temperature brazing of silicon nitride. A study of bonding  $Si_3N_4$  with nonmetallic materials was continued by formulating a series of both  $MgO-Al_2O_3-SiO_2$  (MAS) and  $Y_2O_3-Al_2O_3-SiO_2$  (YAS) mixtures and examining their general melting and wetting characteristics. Two silicon nitride substrates were used: GS44 for the MAS compositions and PY6 for the YAS compositions. The results from the initial set of experiments showed that three general types of behavior were observed: (1) a stable droplet formed; (2) a stable droplet formed, then chemical reactions created a moderate level of porosity; or (3) relatively strong or rapid chemical reactions caused the droplet to foam. In all cases, adding  $Si_3N_4$  powder to the oxide mixtures reduced their chemical stability. For all mixtures except the one based on MAS3, a further addition of 5 wt % Si suppressed the tendency for chemical instability. Also, an assessment of ways that poppet-type valves could be configured with ceramic heads and metal stems was initiated. Several valve configurations were proposed, and efforts to analyze them using finite element techniques were initiated.

A major thrust of work on the welding and fabrication characteristics of Ni<sub>3</sub>Al alloys supported by the AIC Materials Program is the transfer of this technology to industry through CRADAs. Part of the welding task on this program involves participation in CRADAs with General Motors (GM) Corporation, Saginaw Division, and with Metallamics, Inc. As part of the GM CRADA, defects in sand-cast Ni<sub>3</sub>Al alloy heat-treating trays were repaired by welding with filler metals developed on the AIC Program. The repaired trays are currently undergoing evaluation under actual service conditions in a batch heat-treating furnace application. On the Metallamics CRADA, procedures were established for welding trunnions to transfer rolls for use in a steel processing plant. Also, welders at the foundry responsible for manufacturing the rolls were trained to weld Ni<sub>3</sub>Al alloys. Non-CRADA work continued to emphasize the development of filler metal compositions which can be processed by conventional metalworking techniques, have mechanical properties that are reasonable and compatible with Ni<sub>3</sub>Al alloy base metal compositions, and that do not compromise good weldability properties. Several promising compositions were identified. Also, an invention disclosure was filed for a technique for making Ni<sub>3</sub>Al alloy wire.

### 1.5 MECHANICAL PROPERTIES — C. R. Brinkman

The Mechanical Properties Group develops and analyzes data for metals, ceramics, and polymers; qualifies new materials; and provides materials engineering support for ongoing energy, CRADA, and work for others (WFO) programs in support of the DOE and U.S. industry. During the reporting period, we received support from the following programs: Fossil, 10%; Conservation, 25%, Nuclear Reactor Development, 20%; and miscellaneous, 45%. The overall effort on these programs was in characterizing the elastic, plastic, creep, fatigue, and creep-fatigue properties and studying the influence of environment on the mechanical behavior of base metals, weldments, and ceramics. Our laboratory contains a wide variety of uniaxial and multiaxial equipment for testing materials in air, high-vacuum, and gaseous environments. After statistical and parametric analyses of generated data, we store and/or present it in a form useful to engineers or code developers for design. We serve on several important American Society of Mechanical Engineers (ASME) and ASTM committees developing design rules and test method standards.

Data development and analysis for the materials technology to design and license MHTGR-NPR nuclear power systems emphasized collection of information on mechanical properties, thermal stability, and behavior of wrought materials and weldments. Structural and steam generator alloys under investigation included 2.25 Cr-1 Mo (including aged and decarburized material), A533 grade B steel, and Alloy 800. An extensive amount of time was spent evolving test plans and procedures during this period. This program was concluded on September 1, 1993.

Emphasis was placed on characterization of modified Alloy 800 and associated filler materials in the temperature range of 500 to 800°C for fossil plant applications. Evaluation was continued on nitrogen-bearing stainless steels that had seen up to 100,000 h of service in main steam line applications in support of fossil plant life-extension efforts. Efforts aimed at developing CRADAs with private industry for metallurgical examination of bellows and steam tubing were continued.

Creep and tensile testing of refractory metal alloys (Nb-1Zr and PWC-11) continued in support of the Space Reactor (SP-100) Program. Test temperatures ranged from 977 to 1427°C under high-vacuum conditions. The work involved tensile and creep testing as well as helping to coordinate work at other laboratories in support of program needs. Determining the effects of irradiation on the tensile properties of rhenium was continued.

Critical to the development of advanced automotive technology using ceramic components [e.g., gas turbines (GTs)] is the development of a mechanical properties data base for candidate structural ceramic materials. During the period, exploratory tensile, creep, and fatigue tests continued on a number of structural ceramics at both room and elevated temperatures. These tests were conducted with unique specimen grips developed at ORNL, which are now available from a commercial source. Ten creep frames were operational for the testing of ceramic materials for advanced engine development. These systems can test uniaxial specimens and control the load and specimen alignment. A high-temperature, laser-based, noncontacting extensometer developed in this laboratory was used to make highly precise creep-strain measurements. Considerable creep and fatigue data were generated on silicon nitride and models developed for predicting creep and rupture behavior. Papers were written covering work performed and submitted to various journals for publication.

We served as program monitors for several large programs in private industry aimed at developing the methodology for life prediction of monolithic ceramic components and continued our ceramic mechanical and physical properties computerized data storage program in support of advanced heat engine development. An international symposium was organized and held in Cocoa Beach, Florida, covering life prediction of ceramic components operating at high temperatures.

An autoclave facility was utilized for fatigue testing of various alloys in hydrogen at pressures up to 35 MPa. The work was in support of National Aeronautics and Space Administration (NASA) activities and involved fatigue testing of Alloy 718 at room temperature. We acted as a round-robin monitor coordinating the work of other laboratories doing similar work for NASA.

We continued work on determining the high-cycle fatigue properties of metal-matrix composite (MMC) alloys via a CRADA agreement with the automotive industry.

## 1.6 MATERIALS PROCESSING — V. K. Sikka

The Materials Processing Group deals primarily with the development of novel methods for melting, casting, powder making, MMCs and near-net shapes, cleaning of liquid metals, and materials processing. The development of these methods is supplemented with process models. The Materials Processing Group is also developing methods for successful processing of intermetallic alloys such as nickel aluminides, nickel aluminides containing chromium, nickel aluminides containing iron and chromium, iron aluminides, and titanium aluminides. The group also has a significant program for the fabrication of iridium sheet to provide iridium containment in support of space and terrestrial isotope power supply systems. It also has the responsibility for transferring to U.S. industry the

processing technology for nickel- and iron-aluminide alloys and other novel fabrication processes being developed. The group is also very active in developing CRADAs with industry.

The Materials Processing Group fulfills the metal processing needs of the other groups in the M&C Division and does work for other divisions of ORNL as well as other facilities operated by Energy Systems. Processing work is also carried out for other national laboratories, universities, and industries. Specific projects worked on and key accomplishments for FY 1993 are listed below:

1. Ductilization and Processing of Iron Aluminides. Iron aluminides are low-cost materials for highly oxidizing and sulfidizing environments. The use of these alloys has been limited because of their very poor room-temperature ductility ( $\leq 5\%$ ). Several researchers have tried to improve the ductility of these materials over the last 50 years. Only during 1982 through 1984 were the ductility values of these materials increased to 8 to 9%. During FY 1988 and 1989, the ductility values of  $\text{Fe}_3\text{Al}$ -based alloys have been increased in the range of 15 to 20%. The ductility improvement has been obtained through thermomechanical processing and heat-treatment control. The ductile  $\text{Fe}_3\text{Al}$  alloy won the R&D 100 Award for 1990.

It was recognized that the environmental effect was the primary cause of low ductility of iron aluminides. The environmental effect is related to the generation of hydrogen through the reaction of aluminum in the alloy with the moisture in the air. The surface-generated hydrogen diffuses into the alloy during straining and causes the hydrogen embrittlement. A systematic study was conducted on the effects of aluminum content on the extent of the environmental effect. The study revealed that the alloys containing  $\leq 16$  at. % Al are free from the environmental effect. This information has led to the development of new alloy compositions based on Fe-16 at. % Al. These compositions possess room-temperature ductility values of 25 to 28% in the wrought condition. A U.S. patent (No. 5,238,645) has been obtained on these compositions.

The iron aluminides including the new compositions are now licensed to four companies: Ametek Specialty Metals Division (Eighty Four, Pennsylvania); Hoskins Manufacturing Company (Hamburg, Michigan); Harrison Alloys (Harrison, New Jersey); and Cast Masters (Bowling Green, Ohio).

Many applications of Fe-16 at. % Al alloy FAPY have been identified. Among them, the heating element application seems to be the most exciting and ready for commercialization in the near term. The excitement of the heating element is based on the results of the ASTM thermal cycling test on the wire, which has lasted over 580 h as opposed to 300 h observed for the most commercially used materials. The commercial application of the FAPY alloy is awaiting more experience in melting and processing large heats. An R&D 100 package was prepared and submitted on the development of the FAPY alloy.

The  $\text{Fe}_3\text{Al}$ -based alloys have also been identified for a broad range of applications. The most near-term application of these alloys is in the walking-beam furnaces as furnace rails. The rails are expected to operate for 1000 h in the temperature range of 1250 to 1350°C. The simulated tests on beams at 1375°C have shown there is acceptable resistance to oxidation for times exceeding 300 h and sufficient strength to avoid any sagging or distortion under its own load. The walking-beam rails are

currently being produced by two routes: (1) casting, and (2) casting into slab, hot rolling into plate, and machining into the desired shape. The furnace rail application is awaiting delivery of the components by either of the methods described above.

2. Processing Technology and Mechanical Properties of Nickel-Aluminide Alloys. Castings were identified to be the most needed products for near-term applications of the nickel-aluminide alloys. During FY 1993, the effort continued in optimizing the casting process parameters. These included the pouring temperature, cooling rate, and the mold material. The effort was carried out jointly with PCC Airfoils, Inc. (Douglas, Georgia); Cummins Engine Company (Columbus, Indiana); and ORNL. The pouring-temperature and the cooling-rate requirements to minimize casting defects and maximize mechanical properties were established through the joint work between ORNL and PCC Airfoils. A detailed solidification study on the same alloy compositions was completed at the University of Cincinnati. This study provides correlations between the casting-defect characteristics and the solidification conditions. The solidification conditions include the cooling rates, vacuum on degassing, and the use of grain refiners. Good agreements were observed between the correlations developed in the solidification study and the actual castings poured at the commercial caster, PCC Airfoils.

The cast nickel-aluminide test bars show nearly two orders of magnitude improved fatigue life at 650°C over the commercial nickel-base alloy, IN-713C. However, a potential source for lower fatigue properties in nickel aluminide exists for conditions where it reacts with the casting shell material. The reaction depth is generally 1 to 2 mil deep and appears as oxide particles.

An alloy composition modification of IC-221M was developed during FY 1993 that completely eliminates the surface reaction of the alloy with the mold material. The final alloy composition has been procured as three 300-lb heats each from Cannon Muskegon Corporation (Muskegon, Michigan). These heats have been cast into test bars on which fatigue tests have been completed. At least three sets of turbochargers have also been completed from the 300-lb heats. The turbochargers are expected to go into engine test by the end of March 1994.

The rollers for heat-treating furnaces have been identified as another application for nickel aluminides, and very significant progress was made in this area. Bethlehem Steel Corporation - Burns Harbor Plant (Chesterton, Indiana) identified the roller application to replace the current HU material in one of their furnaces. The rollers were centrifugally cast at Sandusky International (Sandusky, Ohio), and the trunnions for roller ends were sand cast at Alloy Engineering & Casting Company (Champaign, Illinois). The machining and welding of the rollers were carried out at Sandusky under close cooperation with ORNL. In fact, the welder from ORNL completed the two circumferential welds and trained Sandusky's welder in order to complete other welds as needed. Metallamics, Inc. (Traverse City, Michigan), ORNL's licensee for the nickel-aluminide alloy, arranged the initial contact with Bethlehem Steel and participated in various manufacturing steps. The rollers were installed in Bethlehem's furnace and have accumulated over six weeks of service. Periodic inspection of the rollers and removal of the coupons from the furnace is continuing. The roller application has been a true collaboration between Bethlehem Steel, Alloy Engineering & Casting, Sandusky, Metallamics, and ORNL.

3. Iridium Processing. The production of defect-free ingots (63 mm diam) of iridium alloy continued during the last year. The extrusion temperature and ratios continued to work effectively for all ingots produced during the last year. The yield of blanks produced from 63-mm-diam ingots continued to exceed 85%.

To meet the production requirements for iridium, two major pieces of equipment were installed. These included a 150-kW EB furnace and a computer-controlled consumable-arc furnace. For use in iridium production, these furnaces required comprehensive operating procedures and qualification steps. During FY 1993, the operating procedures were completed for both furnaces. Tests were completed on the product made in these furnaces, and these furnaces are now considered qualified for iridium-production activities.

Efforts continued during FY 1993 to develop improved processing techniques. The major progress was made in the rolling of iridium sheet. The iridium sheet has been rolled for years by covering it in a molybdenum frame. However, the improved microstructure developed in the sheet bar from the extrusion of the vacuum-arc-remelted ingots provided the opportunity of bare rolling. Several trials have shown that the bare rolling of iridium sheet is possible and will result in significant savings in time and cost for the production of iridium blanks.

4. Sand-casting Facility and Solidification Modeling. A sand-casting facility was added to the Materials Processing Group. It is a complete facility including: blending of sands, mold making, melting, and pouring in the molds. A new 100-lb air-induction furnace was added to this facility to meet the molten-metal requirement for some of the sand molds. The facility is also equipped with temperature monitoring of the molten metal and the computerized data-acquisition system for monitoring the solidification behavior. The facility has already been used for sand castings of aluminum alloys and nickel and iron aluminides. This facility is closely coupled with the solidification-modeling capability. The modeling capability includes features such as mold filling, heat flow, and fluid flow. These models are also capable of helping in the development of quality criteria for castings.
5. Superconductor Fabrication. The effort on the fabrication of long lengths of multifilament, high-temperature superconductor material continued during 1993. The primary emphasis was on the development of deformation-processing requirements to yield high-current density. A new process has been identified to produce high-current density ribbons. A patent application for the process was filed. A systematic approach leading to the development of a commercial fabrication process is currently under way.
6. CRADAs. A 3-year CRADA was signed with GM-Saginaw Division (Saginaw, Michigan) for the use of nickel-aluminide parts in the heat-treating furnaces. As part of the CRADA, ORNL and GM are working jointly to optimize the casting process for the commercial manufacturing of parts required by GM. The CRADA work also includes the development of mechanical properties and microstructures on the cast parts. During FY 1992, two joint casting trials were carried out at Alloy Engineering & Casting, a supplier for GM. The parts cast during these trials have been shown to have the mechanical properties comparable to those developed on small experimental heats cast at ORNL.

A production run consisting of 7 heat-treating trays and over 50 test bars was completed. The trays were successfully repair-welded in the area with some casting defects. One of the trays and over 20 coupons have operated in one of the heat-treating furnaces at GM-Saginaw Division for over 6 months. Several progress meetings were held. The results to date are showing significantly better performance of nickel-aluminide coupons as opposed to the currently used HU alloy. The furnace testing is expected to continue during FY 1994 with new component casting trials scheduled for June 1994.

The use of nickel aluminide by GM is expected to result in substantial savings in heat-treating production costs.

7. **Technology Transfer.** Significant effort was spent in the transfer of various technologies to industries. This effort involved communication through telephone calls, technology transfer meetings, and personal visits. A large effort was also devoted toward supplying sample materials to various industries and universities.

Two CRADAs and one license for the use and production of nickel aluminide resulted from this effort. Many new applications were identified.

8. **WFO.** Work was carried out in the Materials Processing Group for Wright Patterson Air Force Base (Kettering, Ohio) [through Universal Energy Systems (Dayton, Ohio)]; Cummins Engine Company; Allison Gas Turbine Division; INEL (Idaho Falls, Idaho); and several universities.

### **1.7 NONDESTRUCTIVE TESTING — *D. J. McGuire***

The NDT Group develops new and improved methods and equipment for NDE and characterization of materials and components. Typical projects include theoretical studies and computer modeling, design and development of instrumentation and equipment, development of techniques and test procedures, and transfer of technology to users. The tasks require a broad base of multidisciplinary tools comprising expertise and equipment in ultrasonics, dye penetrants, eddy currents, and penetrating radiation. Applications of NDE methods are of interest to a number of sponsoring agencies, including the DOE and USNRC. Technical development and support services have also been provided in cooperation with other ORNL divisions and for outside agencies through the WFO Program.

We have worked for a number of years with the USNRC in developing improved inspection methods for nuclear power plant steam generators. A long-term goal in the nuclear industry has been an eddy-current inspection probe capable of both high-defect sensitivity and high-speed operation. ORNL, last year, developed and field tested two eddy-current probe designs that combine both speed and sensitivity for steam generator inspections. The new probe designs use multiple pancake and reflection coils arrayed around the circumference of the probe body. The new designs have much higher defect sensitivity than traditionally used bobbin coils and an inspection speed 75 times greater than the older rotating pancake probes.

We continue to develop improved software analysis tools for steam generator inspection as well as to provide technical consultation to the USNRC for commercial inspection activities and steam generator evaluation criteria.

Work continues for the DOE Ceramic Technology for Advanced Heat Engines (CTAHE) Program. A new ultrasonic acoustic microscope has been obtained to allow high-magnification acoustic images of ceramic surfaces with resolution of detail to  $<1 \mu\text{m}$ . The new instrument has been useful for studying microcracking and for imaging phenomena such as residual stress that are not amenable to optical and electron imaging systems. Our computerized tomography (CT) system has been upgraded with a SUN workstation to allow three-dimensional (3-D) reconstruction of X-ray images. We have been working toward process improvement in gelcast ceramics and silicon nitride turbine rotors. Additional studies have been done using both ultrasonics and X rays to identify machining damage in ceramic materials, as well as failure modes in industrial components, such as spark plug insulators and grinding wheels.

We continued our component inspection development and support for the DOE SNP through ultrasonic, dye penetrant, and visual testing of iridium disks. We also completed a study of ultrasonic Lamb wave propagation in the sheet iridium material for the development of weld inspection techniques.

The NDT Group has supported a number of Laboratory programs with measurement services. We provided ultrasonic and CT images to show internal detail of reinforcing fibers in continuous fiber-reinforced ceramic samples. We continued our studies for thermal shock-induced microcracking in irradiation-embrittled reactor vessel steel. We continued a service program for eddy-current thickness measurement of oxide layers on aluminum cladding for reactor applications.

### 1.8 MATERIALS PROCESS MODELING — *T. Zacharia*

The Materials Process Modeling Group was created in July 1993, within the Engineering Materials Science Section of the M&C Division, in response to the establishment of the High-Performance Computing Center at ORNL and industry's strong interest in modeling of materials processing activities. There is significant opportunity for increased involvement by the division in this rapidly growing area. The Materials Process Modeling Group has been successful in developing new projects under the DOE Technology Transfer Initiative. These include modeling of casting, forming, heat-treatment, and welding processes under CRADAs with U.S. industry. The group provides the technical lead for ORNL in the Supercomputing for Automotive Applications Partnership (SCAAP) under the United States Council on Automotive Research (USCAR). The objective of this partnership between the automobile industry and the DOE laboratories is to assist the U.S. automotive industry in becoming more competitive in the world marketplace, while meeting broad industrial and national goals in energy, environment, safety, and manufacturing by appropriate use of advanced computing and communications technologies.

The scope of materials and process modeling activities in a few selected areas are presented here.

High-Performance Computing: ORNL's leadership in materials process modeling, high-performance computing, and computational techniques has been utilized to expand

the quantitative understanding of advanced materials and processing technologies. Computational modeling techniques have been utilized to provide quantitative correlations between process parameters, materials properties, and performance. Current programs consist of basic research designed to further a fundamental understanding of materials synthesis and processing and applied modeling studies, which include extension of the fundamental principles and modeling techniques developed to manufacturing practice. The continued development of computational techniques and algorithms utilizing the massively parallel architecture will allow, for the first time, the simulation of conventional materials processes such as forming, casting and joining, and advanced processes such as microwave (MW) sintering and super-plastic forming in sufficient generality for real-time process characterization, optimization, and control.

Modeling of Welding Process: Understanding the development of the weld pool during welding is of considerable practical significance. A unique, state-of-the-art computational modeling capability was developed for investigating heat and fluid flow in a weld pool. There have been a number of attempts to predict the effect of processing conditions on weld pool development by mathematically modeling the transport phenomena that occur during welding. The predictive capability of earlier models of welding processes was limited by the large number of idealizations involved in their development, together with a lack of emphasis on experimental verification. The current model eliminates several of the earlier restrictive assumptions and includes the capability of nonlinear temperature-dependent thermophysical properties, vaporization flux, and free deformable surface effects. The analytical effort has been complemented by a systematic experimental study to verify the predictive capability of the model. A massively parallel version of the computational model has been developed that will allow for more accurate modeling of the welding phenomena using finer discretization and more detailed physical models.

Modeling of Casting and Solidification: The understanding and control of the microstructure and properties of cast materials is an important step in the development of materials and materials processes. The ability to analyze and predict features of casting and solidification processes is essential, as many features of the cast structure (such as microstructure, porosity, segregation, and the size and distribution of precipitates and second phases) can have a profound effect on the properties of the final product. Numerical modeling of solidification structures using novel techniques such as cellular automata allows us, for the first time, to quantitatively predict solidification microstructures. Models of casting solidification that predict mold filling and solidification are used to calculate solidification parameters such as thermal gradient, isotherm velocity, cooling rate, and fraction solid at particular locations.

Modeling of Grain Growth: Grain growth during continuous heating, in the presence of soluble second-phase particles, is being simulated using Monte Carlo techniques. Using this technique, the extent of grain growth in a temperature gradient and the distribution of the second-phase particles during grain growth can be modeled simultaneously. On-heating transformations such as the dissolution and liquation of second-phase particles are being modeled by solving the diffusion equation using finite-difference techniques. Microsegregation of solutes during solidification again involves the solution of the diffusion equation in the presence of moving solid-liquid interfaces. However, the chemical diffusivity in the liquid may be comparable to thermal diffusivity, so that the heat

and diffusion equations should be solved in a coupled fashion. Finite-difference codes for such an analysis have been developed and are being used to compute the extent of microsegregation. In order to predict the susceptibility to liquation cracking during thermal processing, the magnitude of the transient thermal stresses should be calculated using appropriate constitutive equations which adequately describe the mechanical behavior of the alloys at temperatures close to the melting point of the alloys. Such a macroscopic analysis should then be coupled with a microstructure model, which predicts the temperature range during which grain boundary (GB) film exists. The micro-model has already been developed for simple binary alloys and is being extended to multi-component alloys. Transient thermal stresses are being computed using finite element analysis of the process using elasto-visco-plastic constitutive equations for the material in the high-temperature zone.

Modeling of Deformation Processes: Modeling of large deformation forming of bulk or sheet processes has quite different natures. Furthermore, for a given forming process, objectives are often different. Depending on the information desired (forming limit, press capacity, die design, or material property sensitivity), different features are more desirable in a finite element method code. For determining forming limits in severely deformed areas, calculation speed may be the most critical factor. Speed is also most crucial when determining the press capacity needed, or the effect of an added drawbead or lubricant change. For a bulk-forming/thermal mechanical processing application, code capability (to model complex materials) is often most important. For die design and springback issues, maximum accuracy is important.

By and large, implicit techniques and computer codes such as NIKE3D are used for analysis of forming processes. These codes take advantage of the implicit integration algorithms in order to obtain computational efficiency. It would be desirable to modify the implicit algorithms to improve their performance. Therefore, improved equation solvers that take advantage of the massively parallel environment must be developed. Explicit techniques and codes such as DYNA3D have recently been used for sheet-forming processes. The results of these studies have been encouraging and have generated a lot of interest in developing algorithms to utilize DYNA3D in a massively parallel environment for sheet-forming modeling processes.

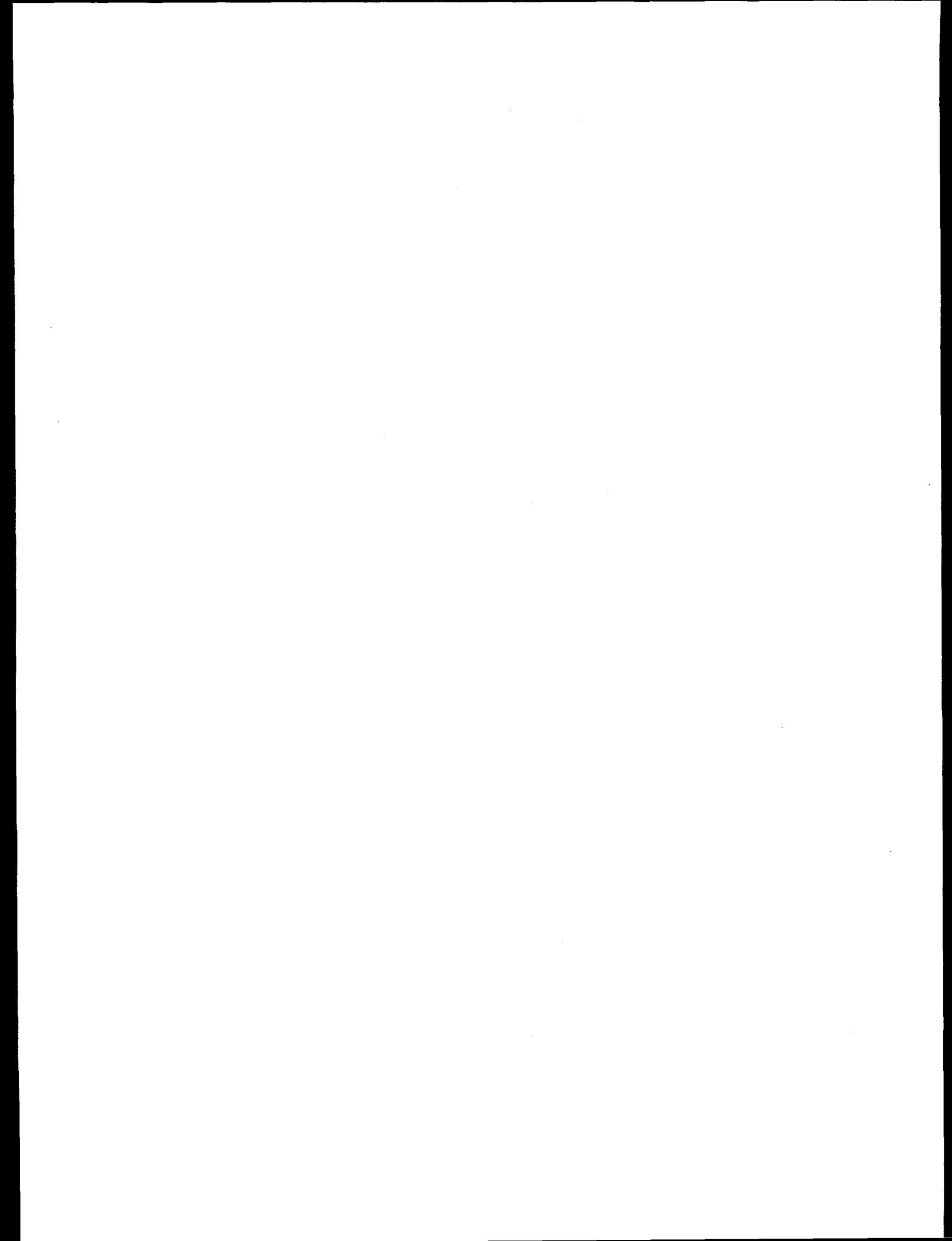
Recently, DYNA3D was ported to the Intel ipsc/168, distributed-memory, parallel computer. Initial experience on the Intel/I860 has been extremely encouraging. To date, the massively parallel processor version of DYNA3D has been applied to several process modeling applications. The model was applied to the 3-D finite element analysis of large deformation response during super-plastic forming of tin-lead plate. The analysis used 19,584 Belytschko shell elements. The model performed very well, exhibiting linear increase in computational speed for the 128 ipsc-nodes available for computation on the I860. From a practical sense, the results indicate a reduction in computational time from 31 d to 6 h. The model was used to determine fabrication parameters for super-plastic forming of a lead-tin pan at the Y-12 Plant. The experience from this project will enable significant developments in materials syntheses and processing research.

Massively Parallel DYNA3D and NIKE3D — The version of the finite element program DYNA3D developed for the massively parallel computers that use the message passing

processor communication has been ported on the ORNL Kendall Square Research (KSR)2 machine. The support for the message passing in the KSR2 shared-memory environment has been provided by the send and receive primitives from the TCGMSG and TSGTHREADS libraries. The two libraries utilize different approaches for implementing parallelism. The TCGMSG implementation follows the original idea of providing parallelism by generating separate Unix processes. This version requires minimal intervention in the original code. The main disadvantages are that a large memory has to be set aside for interprocessor communication. In addition, the KSR tools for the analysis, development, and debugging of a program cannot be used.

The second message passing model is based on the OSF-threads. The resulting program creates a single process with multiple threads performing work in parallel. The original code requires more modifications than in the previous case. This implementation requires no explicit message buffering; provides faster send, receive, and global operations; and allows the use of KSR tools (gist, udb, and pmon).

New massively parallel computer architecture has revolutionized the design of computer algorithms and promises to have significant influence on algorithms for engineering computations. Realistic engineering problems using finite element analysis typically imply excessively large computational requirements. Parallel computers that have the potential for significantly increasing calculation speeds can meet these computational requirements. The research explores the potential for the parallel Cholesky matrix decomposition algorithm on NIKE3D through actual computations. The examples of two- and three-dimensional nonlinear dynamic finite element problems are presented on the KSR1 multiprocessor system with 64 processors at the Center for Computational Sciences. The numerical results indicate that the parallel Cholesky matrix decomposition algorithm is attractive for NIKE3D under multiprocessor system environments.



## 2. HIGH TEMPERATURE MATERIALS

*A. E. Pasto and V. J. Tennery*

The High Temperature Materials Section performs two functions for the division: the three User Center groups perform materials characterization research for several programs, while the Metallography and Technical Photography groups serve as divisional resources, performing metallographic specimen preparation and examination and photographic services, respectively.

The User Center research groups include the Materials Analysis Group, the Mechanical Properties Group, and the Diffraction and Thermophysical Properties Group. Each of these groups is primarily supported by the High Temperature Materials Laboratory (HTML) User Program and acts as a User Center. (For a full description of the User Program, see Sect. 6.8). Each center has specific equipment and expertise related to the materials characterization area designated by its title. This expertise is also brought to bear on the materials characterization needs of other programs within the division. Several important changes occurred this year in the section, including the splitting of Section Manager and Program Manager functions, with A. E. Pasto being named the Section Manager and V. J. Tennery the Program Manager. Also, one User Center, Ceramic Specimen Preparation, was reconfigured to better address the needs of users as the Ceramic Machining User Center (CMUC), and new efforts started in the residual stress area, with inauguration of the Neutron Residual Stress User Facility within the Residual Stress User Center (RSUC).

### 2.1 MATERIALS ANALYSIS — *T. A. Nolan*

The Materials Analysis Group utilizes electron microscopy and surface chemical analysis techniques to characterize the structure and chemistry of advanced structural materials. The information obtained from these characterizations is used to elucidate the mechanisms that control material performance.

An important new instrument has been added to the suite of instruments in the past year. A Hitachi S-4500 scanning electron microscope (SEM) began beneficial operation in December 1993. This instrument combines very high spatial resolution at low accelerating voltage with the ability to observe a large portion of specimens with diameters of up to 15 cm. Thus, it is ideal for the study of fine surface detail on large ceramic components such as high-temperature tensile fatigue-tested  $\text{Si}_3\text{N}_4$  specimens. We were able to use the existing vacuum generator (VG) electron spectroscopy for chemical analysis (ESCA)/secondary ion mass spectrometry in trade for the new SEM. The VG instrument had many capabilities but was difficult to operate and had proven not suitable for a user environment.

The Hitachi HF-2000 200-kV field emission gun-transmission electron microscope (FEG-TEM) has completed nearly 2 years of beneficial operation. This instrument added three major new capabilities. On specimens having ideal geometry, it provides the highest lateral resolution presently attainable for X-ray elemental analysis; elemental composition of regions as small as 1 nm can be determined, thus greatly enhancing our abilities to analyze ceramic GB compositions. A second unique capability offered by the

HF-2000 is electron holography, made possible by the coherent beam from the FEG. Electron holograms preserve image phase information (lost in conventional TEM). The phase image is very sensitive to changes in specimen thickness, composition, or degree of strain, and so can be used to determine, to a high degree of accuracy, the morphology of small particles or the nature of interfaces. Also, utilizing the additional phase information, lens aberration corrections can be made that result in greatly improved resolution.

The third capability introduced on the HF-2000, and now being added to the JEOL 4000EX HRTEM, is digital imaging. A retractable 1024 by 1024 charge-coupled device (CCD) slow-scan camera system has been retrofit to run in conjunction with the standard television camera system on the instrument. This CCD camera effectively supplants film as the recording medium on the microscope and gives an enormous advantage to the throughput of data and the quality of the results available during an operating session. In fact, we have not had film in the microscope since April 1993, and all images from the HF-2000 are now acquired and stored directly on the local area network disc server. The Hitachi S-800 and the new Hitachi S-4500 are also being converted to all-digital format. Thus, by early spring of 1994, the Materials Analysis Group will be acquiring and processing all images digitally with the exception of those produced on the JEOL 2000FX TEM. An additional benefit is the reduction of photographic wastes.

### 2.1.1 Materials Analysis User Center (MAUC)

High-resolution electron microscopy (HREM), combined with SEM, high spatial resolution energy-dispersive spectroscopy (EDS) on the HF-2000, and low-glancing-angle X-ray diffraction (XRD) techniques, have been utilized to identify possible contaminants on the surfaces of alumina insulating rings used in chemical vapor deposition (CVD) furnaces (Coors Technical Ceramics). The electron microprobe has proved invaluable for the characterization and possible source identification of surface and subsurface defects in ceramic tile (Florida Tile Industries). The first HTML Faculty Fellow in this center from the HTML Fellowship Program, Professor A. K. Datye of the University of New Mexico in Albuquerque, performed research during the summer. His research centered on characterization of the microstructure of a number of model catalysts and commercial catalyst materials. Palladium nanocrystals on the order of 10 nm in diameter on microspheres of  $\text{SiO}_2$  were shown by electron holography on the HF-2000 to contain voids in the center of the nanocrystal. The results of this work have been submitted for publication. The analytical capabilities of the Hitachi HF-2000 are being used to determine the microstructures of AlliedSignal GS-44 and A-series  $\text{Si}_3\text{N}_4$ , as a part of another HTML User Project. The scanning auger microprobe (SAM) was upgraded this past year with new computer capability and operational software. An important industrial collaboration took place this past year with Dr. Suzanne Raebel from Cummins Engine Company spending 3 months at the HTML as the first HTML industrial fellow, using the instrument to analyze the surfaces of numerous diesel engine components and bearing materials. Other projects utilizing primarily the SAM included a study of the relationship of surface conditions of different electronic components to processing parameters by personnel from Coors Electronic Packaging. Work with universities includes the identification of  $\text{SiC}$  islands produced by CVD techniques (Stevens Institute of Technology), the identification of key elements present after the laser treatment of aluminum oxide wafers for selective plating (University of Tennessee), the in situ fracture and analysis of nicalon fibers [Rensselaer Polytechnic Institute (RPI)], and the analyses of ceramic films produced by a sol-gel process

(University of Arizona). The atomic force microscope (AFM) has contributed to a number of important projects over the past year. Analyses of the surface roughness of anodized aluminum components (Rockwell International) have shown why O-ring life is being shortened by contacting these surfaces. In work with universities, tiny SiC islands produced by CVD were imaged (Stevens Institute of Technology), a tin oxide/silicon oxide interface examined (Alfred University), and a thin molybdenum film analyzed (University of Denver).

### 2.1.2 Materials Analysis Research

The second year of a 3-year Director's Fund initiative to develop electron holography has been completed, and we are now routinely taking high-resolution electron holograms on the HF-2000. Dr. E. Volkl, an international leader in electron holography, is in his second year as a postdoctoral fellow on the holography project. He has developed numerous subprograms that run efficiently within the Macintosh software used to run our digital camera system. These programs allow us to perform the image reconstructions possible with holography, and they have the characteristic Macintosh ease of use that makes it possible for average users to obtain useful holography data.

The characterization of various interface coatings in CFCCs was accomplished using the high-resolution capabilities of the JEOL 4000EX. For example, it was shown that boron-doped carbon interface layers, which are beneficial for enhanced oxidation resistance of the composite, have a highly aligned, turbostratic carbon structure as compared to the partially graphitized carbon coating commonly used in CFCCs. The analytical capabilities of the Hitachi HF-2000 have been used to determine conclusively the presence of yttrium in GBs of the latest production  $\text{Si}_3\text{N}_4$  ceramic manufactured by Norton, NT-164. Several other  $\text{Si}_3\text{N}_4$ -based ceramics have been analyzed in a similar manner, including the intermediate-temperature Norton NT-451 and a Russian cutting tool material. Electron holography applications are rapidly developing on the HF-2000 as we are now fully operational on the digital image acquisition and hologram reconstruction software. We are using holography to characterize the structure of supergiant fullerenes and carbon nanotubes, the nature of charge effects at GBs in electroceramic materials such as bismuth-doped  $\text{MgO}$ , and the morphology and surface facetting in nanophase ceramic particles such as  $\text{ZrO}_2$ . Holography has been used in conjunction with heating stage experiments to quantify the nature of ferroelectric domain transformations in  $\text{BaTiO}_3$ . A newly developed operational technique has permitted us to record holograms over wide areas, which has allowed, for the first time, the ability to map electrostatic fields that surround a surface such as a non-conductive particle, or that surround dislocations in materials. In the latter case, for example, we expect this technique to become useful in characterization of the effects of fields on dislocation motion that governs the creep behavior of high-temperature materials.

At ORNL, the SAM has been used to successfully identify the contaminants present on the outer surfaces of aluminum tubes protecting the fuel elements in the HFIR (CTD). New methods of cleaning the tubes are now being investigated. In collaboration with the Structural Ceramics Group, an extensive investigation of the interface chemistry between SiC whiskers and the alumina matrix in whisker-reinforced composite materials has been completed. It was concluded that interfaces with lower oxygen contents produced composites with greater toughness. The AFM was used to characterize the ceramic coatings on advanced piston rings (Cummins Piston Ring Division) and has aided in the understanding of the relationship between coating parameters and

performance. The pore sizes of polycarbonate filters were determined (Department of the Navy), and the surface topography of the inside of glass capillary tubes was measured (Y-12 project). Within ORNL, surfaces of machined ceramics and wear surfaces of silicon nitride have been imaged and analyzed (Cost-Effective Ceramic Machining Project), surfaces of Nicalon fibers and the microstructure of SiC/SiC composites have been studied, and the depth of extremely small indents in materials from the Nanoindenter were measured. This latter effort led to the purchase of a second AFM for dedicated use on the Nanoindenter in the HTML.

## 2.2 MECHANICAL PROPERTIES — *M. K. Ferber*

The group leader of the Mechanical Properties User Center (MPUC) is Dr. M. K. Ferber. Other staff and their areas of expertise are as follows:

- Dr. A. A. Wereszczak** — characterization of the tensile creep/fatigue behavior of structural ceramics, fracture toughness testing, and evaluation of interfacial properties in fiber-reinforced ceramic composites;
- Dr. E. Lara-Curzio** — characterization of macro- and micro-mechanical behavior of ceramic composites, fiber testing, and modeling of the constitutive equations for mechanical performance in ceramic composites;
- Ms. L. Riester** — indentation and flexure testing;
- Mr. T. P. Kirkland** — measurement of mechanical properties of structural materials; and
- Ms. D. Green** — secretary.

### 2.2.1 Mechanical Properties User Center

The MPUC is dedicated to the study of the mechanical performance of high-temperature materials. A major thrust of the MPUC is to examine the influence of temperature, time, and applied stress level upon properties such as strength, toughness, fatigue, and creep resistance of advanced materials. The major research facilities include: (1) a Flexure Test Facility comprising six high-temperature flexure load frames; (2) a Tensile Test Facility consisting of ten high-temperature tensile testing load frames (one with environmental capability), a fiber test machine, a composites test machine, and servo-hydraulic universal test machine (UTM) equipped with tension/compression grips; (3) a general-purpose testing lab comprising two UTMs; (4) a mechanical properties microprobe (MPM) [Nanoindenter]; and (5) an Interfacial Test Facility. Currently, one of the UTMs is equipped with a ceramic retort so that the high-temperature mechanical properties can be evaluated in inert environments or in vacuum.

A major accomplishment during FY 1993 involved the completion of an AlliedSignal user project on the mechanical characterization of the long-term mechanical reliability of a silicon nitride ceramic. Specifically, the creep/fatigue behavior of a hot isostatically pressed silicon nitride (Norton's NT154) was evaluated at temperatures of 982, 1149, 1204, 1260, 1315, 1371, and 1400°C. In the range 1204 to 1400°C, creep deformation was reliably measured using high-temperature contact probe extensometry. The stress and temperature sensitivities of the secondary (or minimum) creep rate were estimated using a global regression procedure. This approach provided an assessment of the scatter typical of this class of materials as well as the identification

of critical transition temperatures associated with a change in the failure mechanism (as reflected by a change in the stress dependency).

The resulting data were used to verify models for predicting both primary and secondary creep rates and the stress-rupture life. The parameters used for relating failure time to stress at a given temperature were based on those developed previously for metals. In this work, the Dorn parameter was used for the modeling of stress-rupture because of its closeness in formulation to the creep rate model. However, it was further modified to accommodate an apparent change in mechanism observed both in the creep rate and stress-rupture behavior of NT154. The applicability of a modified Monkman-Grant expression for describing the stress-rupture life was also examined and found to be inappropriate since the rupture life was not uniquely dependent upon creep rate. A modification to the Monkman-Grant expression was necessary to accommodate an additional temperature or stress dependency. In effect, the stratification of the Monkman-Grant curves could be explained on the basis of a crack growth model. These models, being phenomenological, are expected to be applicable to other silicon nitrides of similar class or chemical composition.

### **2.2.2 Mechanical Properties Research**

Research activities in the MPUC have focused on the mechanical characterization of both monolithic ceramics and ceramic matrix composites (CMCs). A major objective of these studies was to measure the temperature and stress sensitivities of the dominant failure mechanisms and then compare the resulting experimental data to model predictions. A major finding from these tests was that when failure was controlled by creep damage generation and accumulation, the fatigue life was uniquely determined by the steady state creep rate (i.e., Monkman-Grant behavior). A second objective was to examine the effect of loading mode (cyclic versus static) upon the high-temperature failure behavior.

Research activities on CMCs have been directed toward the development of standardized test methods for flat and cylindrical specimen configurations. The unique stress-strain response and fracture behavior of CMC materials require specialized test techniques, systems, and sensors to accurately assess their mechanical properties and define the salient events associated with fracture processes. Additional efforts have focused on the development of test methods for the measurement of interfacial properties.

### **2.3 DIFFRACTION AND THERMOPHYSICAL PROPERTIES GROUP—*C. R. Hubbard***

The principal activity of the Diffraction and Thermophysical Properties Group is operation of the Physical Properties User Center (PPUC), the X-Ray Diffraction User Center (XRDUC), and the RSUC. The group also contributes to a number of other DOE and agency-sponsored programs.

The group maintains a computer network consisting of Macintosh personal computers (PCs) for the staff connected by ethernet to a group server. Also connected is the VAX cluster which supports data collection and analysis for the XRD systems. Finally, the IBM-compatible computers on the PPUC instruments are connected by ethernet to the servers and the VAX cluster. This provides for data logging and remote data processing.

Users' needs for data analysis and data transfer are provided by the VAX cluster (telenet, FTP); a Macintosh computer; and an IBM-compatible 486 computer. Printing options include several postscript printers, which include the Tektronix Phaser 200i for color images.

### 2.3.1 Physical Properties User Center

The PPUC is dedicated to measurement of physical properties as a function of temperature and correlation of the thermophysical properties with processing, microstructure, and performance. Current facilities include the instruments listed below.

Stanton Redcroft STA1500 simultaneous thermal analyzer (STA) with  
differential thermal analysis (DTA)  
thermogravimetry  
evolved gas analysis (EGA)  
Stanton Redcroft DSC1500 differential scanning calorimeter (DSC)  
Theta dual push rod dilatometer  
Holometrix laser flash thermal diffusivity system  
autopycnometer  
xenon flash thermal diffusivity system  
longitudinal bar thermal conductivity system

The last two instruments were built by the staff to meet specific measurement needs where no commercial instruments exist. The room-temperature xenon flash thermal diffusivity system complements the high-temperature laser flash thermal diffusivity system. The longitudinal bar thermal conductivity system is now in the testing and debugging stage. It will provide direct measurement of thermal conductivity from liquid nitrogen temperatures up to 200°C, thus covering a temperature range not available with the two thermal diffusivity systems. Data in this range are needed to extend the group's ability to model thermal conductivity with microstructural models containing terms for grain size and defects.

Two new instruments were ordered to meet the needs of the users in the PPUC. These instruments will be delivered and made operational in FY 1994:

Cahn thermogravimetric analysis (TGA) and companion DTA  
Topometrix scanning thermal conductivity microprobe

The high-temperature, high-capacity Cahn TGA/DTA will extend the thermal analysis measurement range from 1500 to 1700°C. This temperature range is needed for a variety of users and DOE projects. With capacity for 100-g samples and for sensitivity of 1 part in  $10^6$  weight changes, data on long-term oxidation/corrosion can be obtained. The Cahn unit also permits sample exposure to corrosive atmospheres, a capability not currently available. The scanning thermal conductivity microprobe was built to ORNL specifications for a means to measure contrast in thermal conductivity on a micron scale. The unit is an extension of the well-established AFM. This instrument will be the first of its kind in the world.

The PPUC Program supported a wide variety of users from both industry and university. In 1993, the user activity level increased nearly 25% over that of the preceding year. The *HTML Sixth Annual Report* describes the user research projects. Two highlights of this research are summarized below.

*"Investigation of the Thermal Expansion and Heat Capacity of (Mo,Cr)2C,"* C. Kneipfle and G. Olson, Northwestern University

Recent interest in improving the strength and toughness of ultra-high-strength steels has led to an interest in the properties of molybdenum-chromium carbides and molybdenum-chromium-vanadium carbides of the M<sub>2</sub>C type. There is currently little information on the thermodynamic properties of the M<sub>2</sub>C carbide phase. Thermodynamic information of the steel phases can be used to predict the compositional dependence of the precipitation-driving forces and coarsening-rate constants. Measurements of the physical properties of the molybdenum-chromium and molybdenum-chromium-vanadium carbides were undertaken at the HTML in order to expand the thermodynamic data base of these M<sub>2</sub>C carbides, which will allow the effects of the M<sub>2</sub>C carbides to be more completely modeled.

Bulk samples of M<sub>2</sub>C (M = Mo,Cr,Fe,V) carbide were synthesized, and the specific heat capacity was determined using the Stanton Redcroft DSC 1500. The specific heat of the carbides is used to provide information on the free energy of the carbide system. The lattice thermal expansion behavior of the M<sub>2</sub>C carbides was investigated using Scintag high-temperature X-ray diffraction (HTXRD). The expansion behavior of the M<sub>2</sub>C carbides is important for the understanding of the nucleation and strengthening behavior of these carbides within secondary hardening steels. This information enables the investigation of the coherent precipitation behavior of the carbides within the parent steel matrix, as well as the consideration of the effect of residual stresses on the observed strengthening.

*"Thermal Conductivity of Physical Vapor Deposited Thermal Barrier Coatings,"* A. Nagaraj, General Electric Aircraft Engines

The xenon flash thermal diffusivity system was used to study the effects of thermal aging on the thermal conductivity of partially stabilized zirconia (PSZ) thermal barrier coatings (TBCs). TBCs are currently used to increase component reliability in gas turbine engines. Two types of TBCs were investigated. The first type was fabricated by physical vapor deposition (PVD) and the second type by plasma spraying. The specimens were heat treated at various temperatures, up to 2200°F, for times ranging to 100 h. At room temperature, the PVD TBCs have a thermal conductivity 35% higher than the plasma-sprayed TBCs. However, the PVD TBCs were found to be less susceptible to increases in thermal conductivity due to thermal aging than the plasma-sprayed TBCs.

### 2.3.2 Physical Properties Research

The Physical Properties Group is responsible for subtask 2.1.4, "Thermal Models," of the DOE's CFCC Program. The objective of this subtask is to develop and evaluate modeling algorithms and techniques that describe and predict the thermal conductivity of CFCCs as a function of constituent properties. During FY 1993, the Physical Properties Group investigated the effect of porosity in Al<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> unidirectional composites and the effect of fiber interface coating thickness in SiC/SiC composites. In addition to the measurement and modeling of CFCC material systems, the Physical Properties Group is

also developing techniques for the measurement of composites and their constituent thermal properties. These techniques include the step-heat flux system and the scanning thermal conductivity microscope.

In the past reporting period, the Physical Properties Group continued its investigation into the high-temperature (up to 2000°C) thermal diffusivity and conductivity of carbon-bonded carbon fiber (CBCF) insulation for RTGs. This material possesses an extremely low thermal conductivity which makes it a very difficult material to measure at high temperatures. Thus, in FY 1993, we have initiated a study into the validity of high-temperature thermal diffusivity measurements of CBCF insulation. This research includes modeling the temperature dependence of the calculated thermal conductivity, a critical review of the measurement technique, and development of an alternate measurement technique. This work will continue into FY 1994.

In support of the DOE's Space Power Program, the Physical Properties Group began an investigation into the effect of gamma irradiation on the thermal properties of LiH + 2% Li. The properties measured included thermal expansion, specific heat, and thermal diffusivity. All properties were measured up to the irradiation temperature. Temporary glove bags were used in order to transfer the moisture-sensitive specimens in and out of the measurement equipment. This work is scheduled for completion in the second quarter of FY 1994.

Batch processes such as iron and steel, glass, and specialty brick manufacturing could benefit from capture/reuse of waste thermal energy. The industrial sector consumes about 40% of the total U.S. energy requirements with most of this energy being exhausted as heat. Existing regeneration systems are based on sensible heat (heat capacity) only. Use of latent heat (heat of melting) in addition to sensible heat could result in reductions in storage media volume requirements, high heat transfer capabilities, and lower costs. Composite salt/ceramic materials, fabricated such that the ceramic matrix retains its integrity through the salt melting temperature, are under investigation for use in such thermal energy storage (TES) systems. During FY 1993, the Physical Properties Group conducted the characterization of a SiO<sub>2</sub>-40 wt % Na<sub>2</sub>SO<sub>4</sub> (nominal) composite TES material including the determination of the phase composition, phase stability, specific heat capacity, thermal diffusivity, and thermal conductivity of the material. These values are required in support of efforts to evaluate the efficiency of such TES systems through computer modeling of an operating test facility.

Also during FY 1993, the Physical Properties Group contributed to the efforts to develop and commercialize aluminide intermetallic alloys. Properties including thermal expansion, specific heat capacity, thermal diffusivity, thermal conductivity, solidus and liquidus temperatures, and heat of fusion were determined for representative compositions of Fe<sub>3</sub>Al, FeAl, and Ni<sub>3</sub>Al-type alloys. The data developed have been included in a handbook of mechanical and physical properties for use by potential licensees. The data are also being used in modeling of casting processes using the alloys.

### **2.3.3 X-ray Diffraction User Center**

The XRDUC utilizes room- and high-temperature diffraction methods to characterize the phase(s) and stability of advanced structural materials. The data obtained individually,

as a function of temperature, and/or in conjunction with data from thermal analysis or electron microscopy, are used to relate phase composition and stability with materials performance.

There are two major instruments in the XRDUC:

Scintag Q-2Q PAD V goniometer with I-N<sub>2</sub>-cooled Ge detector  
Scintag Q-Q PAD X goniometer with Buehler high-temperature stage

Orders for upgrades and options placed in 1993 include a scanning position-sensitive detector (PSD) for the high-temperature diffraction system. This optional detector will permit more rapid data collection for such simulations as rapid heating and for following kinetics of thermal transformations. Optional computer-controlled incident beam slits will permit precise control of the irradiated sample length and enable "on-the-fly" changes in the incident slit width. This capability provides for greater diffracted intensity at higher diffraction angles, a need for application of the Rietveld method.

The capability for Rietveld method, or whole pattern fitting, was developed to a useful level by Mr. N. McAdams and Dr. X-L. Wang. This technique utilizes the intensity versus diffraction angle data and refines the atomic structural parameters, lattice parameters, and instrumental parameters to obtain the best fit. Through a series of tests with standards, the technique was shown to be valuable for thermal expansion measurements in addition to the well accepted atomic structure refinement. This technique, while long applied to neutron diffraction data, is equally applied to X-ray powder diffraction data. However, the effects of preferred orientation can be much more severe in XRD.

The facilities of the XRDUC are extensively used in support of user program projects and are also used by the group and other staff within the division for routine phase identification. The latter amounts to approximately 40% of the time on the room-temperature instrument. The room-temperature instrument is extensively used in support of research on two more specialized systems: the high-temperature diffraction system and the residual stress system. The XRDUC often is utilized in combination with research involving other user centers such as Materials Analysis and Physical Properties. Several examples of user program research are given below. A more complete listing is available in the *HTML Sixth Annual Report*.

*"The Mechanical and Thermal Expansion Behavior of Sodium Zirconium Phosphate Structure-type Materials,"* J. E. Spruill and S. Shanmugham, University of Tennessee.

Room- and high-temperature XRD data were collected on some of the compositions in the barium zirconium phosphate silicate system, which belongs to the sodium zirconium phosphate structure type. These materials are being studied as candidate low (near-zero)-thermal-expansion inserts for low-heat-rejection engine applications. Room-temperature X-ray diffraction (RTXRD) data were used to identify the phases present in the compositions. Unit-cell parameters, *a* and *c*, were determined using internal standards and least-squares cell refinement methods. HTXRD data were collected, and the axial thermal expansions were determined. The HTXRD data show conclusively that a phase transformation occurs near 900°C. Use of Rietveld analysis clearly determined that the transition corresponded to an order-disorder of the barium cation with a corresponding change of symmetry. Subsequently, DSC studies were performed in the HTML to observe/confirm phase transitions in some of the compositions.

*"A Phase-Transformation Study in Barium Alumino Silicate (BAS)-Silicon Nitride System Using High-Temperature X-Ray, Thermal Gravimetric Analysis, and Differential Thermal Analysis,"* P. B. Aswath and A. Bandyopadhyay, University of Texas/Arlington

Composites of 70%  $\text{Si}_3\text{N}_4$  with 30% BAS and 40%  $\text{Si}_3\text{N}_4$  with 60% BAS compositions were studied using DSC, STA, dilatometry, RTXRD, and HTXRD. Presintered powders of both the compositions, containing  $\text{BaCO}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ , and a mixture of alpha and beta- $\text{Si}_3\text{N}_4$  with predominantly alpha- $\text{Si}_3\text{N}_4$ , were mixed in an appropriate proportion and sintered for different sintering times. Studies were conducted at the HTML on both presintered powders and on sintered composites that were sintered for different sintering times. Thermal analysis was first conducted using STA, DSC, and dilatometry. For both DSC and STA, experiments were conducted from room temperature to 1450°C using alumina and platinum pans. It was observed that at high temperature, samples were reacting with Pt pans and resulting in some error in the experimental data. Dilatometry tests were conducted from room temperature to 1000°C at a heating rate of 3°C/min for ten samples with different compositions and different sintering times. XRD studies were conducted after thermal analysis. RTXRD experiments were conducted on both presintered and postsintered powders. HTXRD tests were done on both compositions with presintered and sintered powders. Tests were conducted at several temperatures between room temperature and 1600°C in an He environment and with a  $2\theta$  range of 10 to 45°.

*"Characterization of Doped Titania,"* S. E. Pratsinis, University of Cincinnati

The aim of the HTXRD work was to study the phase evolution of alumina- and silica-doped titanium dioxide powders made via the gas-phase oxidation of the respective metal chlorides. The qualitative results obtained from the high-temperature diffraction study confirmed the room-temperature observations that the presence of alumina enhanced the formation of rutile titania, while the presence of silica retarded rutile titania formation.

High-temperature scans between 600 and 1000°C under flowing helium shed some light on the mechanism of phase transformation. In the case of silica-doped titania, the high-temperature scans revealed a progressive increase in rutile content and a corresponding decrease in anatase content as a function of time and temperature. This clearly confirms that the silica interferes with and hinders the phase transformation mechanism in titania. Only on increasing the temperature are the titania crystallites able to transform to the rutile form in the presence of silica, suggesting an increasing activation energy for the transformation which, in the absence of silica, could occur at lower energy levels.

In the case of alumina-doped titania, the powders were primarily rutile initially, while high-temperature measurements investigated all rutile. At room temperature, the alumina dopant was amorphous, but heating during the high-temperature scans resulted in transformation into alpha-alumina. This was clearly observed in samples doped with greater than 20% alumina.

Simultaneously with the high-temperature work, room-temperature analyses were obtained for silica-doped titania and for titania made in the presence of sodium, potassium, and cesium chloride. Profile fitting and silicon internal standard were used

for accurate determination of lattice parameters. For silica contents up to 5%, and for all the titania powders made in the presence of ionic additives, there was no increase in the anatase titania lattice parameters.

Within the MAUC, TEM in conjunction with EDS was used to study particle morphology and chemical composition of titania doped with alumina. The use of the HF-2000 enabled the mapping of chemical composition variation across the cross section of particles at resolutions as high as 10 nm. At high alumina content (40%), two distinct morphologies were observed: large, round particles and small, polyhedral ones. The larger particles were between 100 and 200 nm in diameter, and the chemical composition varied from mainly Ti on the surface to predominantly Al in the interior. The smaller particles were more uniform in chemical composition, and the surface was richer in Al. These particles were better crystallized as evidenced from the distinct lattice fringes observed.

At lower alumina content (7%), the particles were polyhedral with round edges. A few of the larger particles were also observed. In all cases, the particle surface was richer in Al than the particle interior. For the small particles, a large variation in the Ti/Al ratio was observed; in some, the Al content was as low as 2%, while in others, it was as high as 11%. The results discussed are from three samples, and more samples need to be studied before drawing any conclusions. These results are very interesting, and the dopant-crystallinity-chemical composition relation will provide some insight into multicomponent particle formation once the analytical TEM results are combined with ESCA and Auger data.

#### 2.3.4 Residual Stress User Center

The RSUC has completed 20 months of beneficial use of a unique, state-of-the-art instrument providing high-intensity X rays along with very high-precision measurements. The instrument consists of two components:

Scintag powder texture stress (PTS) 4-axis goniometer with chi tilt capability  
MAC Science 18-kW rotating-anode generator

The system has been shown to have high sensitivity (~10 ppm) to strain and is very stable over a period of several days. These attributes permit measurement of strain in stiff materials such as structural ceramics as well as materials with more typical elastic properties. To date, over 20 user proposals for use of the instrument have been approved by the HTML user advisory committee. This extensive demand, which could not be fully met, has led to initiating procurement of accessories to speed up data collection. The major accessories on order are (1) a PSD and (2) a laser-positioning sensor.

The laser-positioning sensor will permit locating the specimen at the correct height quickly and reproducibly. This will be particularly valuable for studies involving multiple electropolishing steps to determine stress gradient as a function of depth. The PSD will speed data collection by a factor of five to ten, enabling user projects to collect more accurate data, faster data, or study more samples. This speed does come at the expense of a trade-off in resolution and need for a beta filter in the incident beam. Both of these items will be delivered in 1994.

A number of industrial and university requests for texture and orientation distribution function measurements, along with more availability for residual stress measurement,

have also led to the initiation of procurement of a Scintag PTS goniometer. This instrument, using a sealed-beam X-ray tube, will be operational by early 1994.

Selected User Program research projects performed in this center are summarized below:

*"Residual Stress Analysis of Si<sub>3</sub>N<sub>4</sub> Cylindrical Tensile Specimens,"* M. Foley, Saint Gobain-Norton Industrial Ceramics

The objective of this study was to determine the effect of machining-induced residual stresses (damage) on strength of tensile specimens made of 4% Y<sub>2</sub>O<sub>3</sub>-doped Si<sub>3</sub>N<sub>4</sub> and thereby increase Norton's understanding and control of their ceramic grinding procedures. As-machined and machined-heat-treated samples were examined. The results from the first visit indicated hoop compression and no axial residual stress in the samples. This conflicted with earlier work done independently upon similar samples. Major equipment problems and scheduling conflicts prohibited any further work from being done until recently. Consequently, this study has been reduced to trying to confirm some of the results from the previous study, and is still ongoing.

*"Measurement of Residual Stress Distribution of Stainless Steel Workpiece Machined by CNC Turning Center,"* D. Y. Jang, University of Missouri-Columbia Center

The objective of this study was to determine the effect that various machining parameters have on residual stress in 304 stainless steel and then to test the validity of a finite element model of machining. The full-stress tensor was determined for eight stainless steel samples representing different machining conditions. The residual stresses were measured using a back-reflection XRD technique and calculated using the multiple sin<sup>2</sup> psi method. The residual strains were measured using CrK<sub>β</sub> radiation and (311) crystallographic reflection ( $2\theta = 147.68^\circ$ ). The residual stresses were calculated from the measured strains using XRD-effective elastic moduli derived from single-crystal elastic constants for 304 stainless steel using generally accepted averaging schemes. The measured strains were normalized against a stress-free standard (SS304 polycrystalline sample annealed at 2000°F for 2 h).

*"Residual Stress Distribution in Graded Ceramic-to-Metal Joints,"* B. Rabin, INEL

The goal of this collaborative research effort is to understand the effects of microstructure and specimen geometry on the distribution of residual stresses in ceramic-metal gradient materials using a combination of experimental and modeling techniques. XRD residual stress-mapping measurements were made at the HTML RSUC at the ORNL and at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory (BNL) on a graded Ni-Al<sub>2</sub>O<sub>3</sub> specimen that was fabricated at the INEL by powder-processing techniques. Surface residual strains were measured at several locations along the length of the specimen using X-ray spot sizes of 0.5 to 1.0 mm. The experimental data are currently being analyzed, and the results will be compared with finite element modeling predictions. One or more peer-reviewed publications will be generated from this work. Additional experiments are planned for the future, including X-ray analysis of Ni-Al<sub>2</sub>O<sub>3</sub> specimens having other geometries and microstructures and neutron diffraction mapping of interior residual strain distributions.

*"X-Ray Analysis of Residual Stress in Metalized Ceramics,"* F. Baldwin and P. H. Holloway, University of Florida Center

The objective of this study was to determine the residual stresses and stress relaxation rates in thin films of an Al-Si-Cu alloy on Si substrates in order to predict the mechanisms and times of failure. These films were found to possess an extreme (111) texture that limited the number of tilt angles (psi) to three. The "unpatterned" samples consisted of a uniform thin film of alloy on an Si substrate. The "patterned" samples consisted of 2.5- or 1.5- $\mu$ m-wide conductor lines of alloy on an Si substrate. The stress states were found to be biaxial. The unpatterned samples possessed less residual stress than the patterned. As the line width (cross section) of the patterned samples decreased, the tensile residual stress in the alloy increased. The residual stresses in the samples containing voids in the lines were less than those without voids. These results will now be correlated with residual stresses from optical measurements of the radius of curvature.

*"Residual Stress and Indentation as Machining Damage Discriminators,"* R. D. Silvers, R. G. Rateick, Jr., P. J. Whalen, and F. Reidinger, AlliedSignal, Inc.

The objective of this study is to determine if the machining-induced residual stresses (damage) scale with the flexural strength distribution of  $\text{Si}_3\text{N}_4$  flexure specimens. If so, then residual stress measurements may be appropriate for part inspection purposes. To date, the results have shown that machining aggressiveness, which affects the strength of silicon nitride (AS-44), also affects the XRD peak positions. However, significant peak anisotropy precludes quantitation of the results using standard profile-fitting methods. The use of peak centroids will be attempted pending establishment of a working internet link. Once an approach for quantitative analysis of the XRD data is identified, a follow-on project may be proposed to develop the technique.

*"Residual Stress Determination of Thick Thermal Barrier Coating Systems,"* J. L. Bjerke and C. J. Anderson, Caterpillar, Inc.

The objective of this study is to determine elastic constants of thick TBCs for use in current finite element models and experimental stress determinations by Caterpillar. Cylinders of plasma-sprayed ceria- and yttria-stabilized zirconia were loaded in compression and examined with X rays to determine the Young's moduli of the (213) and the (331) reflections in tetragonal and cubic polymorphs, respectively. The loading fixture, developed by Caterpillar, contained a calibrated load cell with an applied load display. This fixture was mounted on a goniometer in the "psi-geometry." The Young's moduli were determined using a  $\sin^2\psi$  analysis at various loads. The average Young's moduli for the (213) and (331) reflections were found to be 200 and 160 GPa, respectively, which are in reasonable agreement with literature values for dense zirconia. The stabilization phase appears to have only a small effect on Young's modulus. This study will continue by examining the stress-strain behavior of a  $\text{Y}_2\text{O}_3$ -stabilized  $\text{ZrO}_2$ -NiCrAlY cylinder.

### 2.3.5 XRD Residual Stress Research

In addition to supporting industrial and academic research via the User Program, the RSUC instrument has been employed on two major and several small internal projects:

#### *"Residual Stresses Due to Grinding Parameters in Al<sub>2</sub>O<sub>3</sub>," Navy*

The role of grinding parameters with respect to residual stress was investigated under Navy sponsorship. This "Navy project" was a team research effort involving several groups within the division with the goal to recommend methods to eliminate/reduce a persistent cracking phenomenon that occurs in ceramics utilized in the development of deep-submergence vessels. Small cracks are thought to originate from the machining processes applied to the ceramic. These cracks then grow under the cyclical loading of emergence-submergence cycles. Toward this end, the residual stresses in the ceramic were measured and related to grinding damage and the various grinding conditions. The results of the research team were pooled, and an understanding of the fracture behavior of the selected ceramic (Al<sub>2</sub>O<sub>3</sub>) was presented.

#### *"Residual Stress Mapping of a Metal/Ceramic Joint," B. Rabin, INEL*

This project is part of a large Laboratory-Directed R&D project on mapping residual stresses by neutron and XRD methods (see next section). In this portion, X-ray measurements provide surface residual stress data to complement neutron residual stress-mapping data. Modeling of the joints by finite-element analysis (FEA) methods had been completed by B. Rabin of INEL, and assessments of the experimental opportunities for the use of synchrotron radiation have been completed at the NSLS. High-strain gradients are predicted by the FEA codes and indicate the need for small spot sizes (<0.3 mm diam). The preliminary results obtained at NSLS indicate compressive residual stresses in the surface of the metallic part of the brazed ceramic-to-metal cylinder but do not agree quantitatively with the FEA results. Work is continuing to resolve this discrepancy. Methods to accurately spin the specimen in order to average over a large number of grains were developed.

### 2.3.6 Neutron Diffraction Residual Stress Research

The Laboratory-Directed R&D project, entitled "Development and Demonstration of Neutron and X-ray for Residual Stress Mapping," was a major component of the group's efforts in residual stress.

The long-range goals of the project were to develop and demonstrate world-class expertise in the new field of residual stress measurement and mapping and to provide the foundation for establishing unique DOE-supported Neutron and XRD Residual Stress Facilities for use by ORNL, industry, and universities. The goals of the project were sixfold: First, to successfully develop neutron diffraction macro-residual stress mapping capabilities at the HFIR's HB-3 and -2 beam lines; second, to demonstrate the power of the method by completing a series of measurements on technically important materials; third, to demonstrate the capability to measure micro-residual stresses in composites; fourth, to develop a load frame and use it to conduct experiments to determine diffraction elastic constants; fifth, to couple complimentary X-ray residual stress mapping capabilities to the neutron stress mapping results; and finally, to present to ORNL program managers, DOE, and U.S. industry the potential for application

of this unique ORNL facility to DOE, and industrial problems of importance, and, if successful, to secure programmatic support to further develop this facility and to make it available to industry and DOE.

Strain mapping attachments for the spectrometers at the HFIR were developed and successfully demonstrated. With the use of Be monochromator crystals installed at HB-2 and -3, we demonstrated that mapping could be accomplished at either beam line with gauge volumes as small as 1 mm<sup>3</sup> in iron. This permits flexibility in scheduling the neutron diffraction residual stress experiments and yet achieves useful results in a reasonable time. The automation system provides complete control of all spectrometer axes, an X-Y-Z-chi translation/rotation stage, a tensile load frame, a PSD-multichannel analyzer, and an incident-beam flux monitor. Data analysis packages were developed that automatically apply corrections for nonlinearity in detector intensity and position response and accurately determine the strain. The hardware and software system has been thoroughly tested and refined through conducting a series of demonstration residual stress mappings. These demonstration experiments were selected to be of interest to a variety of industrial segments and to selected DOE programmatic areas.

With the developed ORNL capability, the first-ever mapping of residual stresses in a multipass weld in a ferritic plate was conducted at the HFIR. To develop a more complete understanding of the multipass weld results, weld simulation specimens, known as Gleebles and widely used to test the effects of welding conditions such as heating time, temperature profile, and cooling rates, were characterized. The multipass weld results have already been widely reported—in a number of technical news articles, a prize-winning poster, invited presentations, and two papers. Another major demonstration project involved a detailed mapping of two ceramic-to-metal brazed joints. One joint had a metal strain relief layer between the bulk Fe and ZrO<sub>2</sub> disks. Surface residual stresses near the joint were measured with laboratory and synchrotron X-ray sources, and the data are being compared with finite element model results. Other residual stress mapping studies included welded plates subjected to several proposed stress-relief methods, TBC samples, a rolled aluminum plate, silicon nitride ceramic cam roller follower, fiber-reinforced metal matrix composites, an Fe-Al weld overlay, and an austenitic stainless steel multipass welded plate. Micro-residual stresses in a series of ZrO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> composites were studied at HB-4, and comparative measurements were made at ANL's Intense Pulsed Neutron Source. A neutron diffraction tensile load frame was built, automated, and used for the determination of diffraction elastic constants and the microstructural response of steel and ceramic test specimens.

Extensive industrial demand for further development of the capability has developed based on the demonstration experiments and the need for nondestructive, through-the-volume residual stress measurements. Formal and informal proposals for utilizing ORNL's neutron residual stress facilities have been received from Caterpillar, General Electric (GE)-Aircraft, Penn State, RMI Titanium, ALCOA, United Technologies, Cummins Engine, Pratt and Whitney (Florida), Concurrent Technologies, Coors Electronic Packaging, and others. The National Center for Manufacturing Sciences included efforts for neutron and X-ray residual stress mapping in the CRADA that began in FY 1994. Caterpillar requested our contribution to an Advanced Research Projects Agency proposal, and two proposals were recently submitted with industrial partners from the pulp and paper industry to DOE-Energy Efficiency and Renewable Energy (EE). The Edison Welding Institute, an association of 292 industries, is evaluating the formation of a consortia group to conduct collaborative research with ORNL on stress

prediction and verification in order to improve welding practices and codes. A major proposal to establish a dedicated facility at the HFIR under a cooperative Energy Research (ER) and EE Program has been approved, and financing is expected by early 1994. This effort will establish a Neutron Diffraction Residual Stress Facility as part of the HTML User Program's RSUC. This proposal calls for continued use of HB-2 or -3 while constructing a new, dedicated beam line and instrument at the HFIR. The design goal is to obtain a factor of five greater effectiveness in measurement. Initiation of the User Program efforts in applied neutron diffraction will enlist a new group of industrial users at the HFIR, and the experience gained will lay the foundation for construction of residual stress and materials science instruments at the proposed ANS.

#### **2.4 CERAMIC MACHINING USER CENTER — V. J. Tennery**

The CMUC includes specialized instruments for the study of the machining process in brittle materials. During this year, instrumentation was added to the surface grinder obtained during FY 1992 that allows direct measurement of both the normal and tangential forces in real time during the grinding process. In addition, this system includes a dynamometer which provides the capability of detecting and measuring acoustic events in the workpiece during the grinding process. This is the first such equipped grinding machine in the Oak Ridge complex. Plans were made and implemented to replace the Junger grinder with a world-class stiff cylindrical grinding instrument, and this machine was ordered. It is scheduled for delivery during the spring of 1994. This machine will also be instrumented. A new NICCO grinding machine having the COMMEC capability was obtained and made operational. This machine is used to study the electrochemical grinding enhancement process developed in Japan. It allows major enhancement of the material removal rates for grinding of hard metals, and work will be conducted in the CMUC to determine if this accelerated material removal effect is present for electrical insulators, and if so, to determine how this mechanism works with insulators. This very stiff grinding machine will be instrumented during FY 1994. In addition, plans have been finalized for putting a special measurement room in place as part of this User Center. During this year, several key instruments were obtained for use in this special facility. These include a laser profilometer; a high- frequency spectroscopy unit; and a form tool for precision measurement of the dimensions, cylindricity, and concentricity of axially symmetric objects, such as engine valves and cylinder liners. The laser profilometer allows the non-contact measurement of surface features, using a laser head providing the instrument a vertical resolution of  $\pm 0.05 \mu\text{m}$ , and collects up to 350,000 data points over a field of  $4 \text{ mm}^2$  in less than 2 min. Special techniques are being developed that will allow the quantitative description of the machined surfaces from these profilometer data. The acoustic spectroscopy unit is able to measure the various resonant frequencies of an object with high resolution while sweeping the excitation frequency using a variety of transducers. The form tool is able to align the vertical axis of an axially symmetric object to within better than  $\pm 0.1 \mu\text{m}$  and then to measure its cylindricity and circularity to this accuracy. These instruments are of great importance to the grinding and dimensional measurement instruments in both the Ceramic Grinding User Center and the Ceramic Manufacturability Center (CMC).

#### 2.4.1 Ceramic Machining Research

During this year, several interesting investigations were made using the instrumentation in this user center. Studies were conducted of the relation between the mechanical properties of two types of silicon nitrides as a function of the grinding parameters, specifically the normal and tangential forces on the resin-bonded grinding wheel during the actual grinding process on the instrumented Harig grinder. It was clearly shown that higher wheel rim speeds result in higher Weibull modulus and characteristic strength. It was also shown that for selected ranges of these variables, the depth of cut (and hence material removal rate for a fixed table traverse speed) could be increased without significant negative effects upon the mechanical properties if the wheel rim speed could be high enough, i.e., greater than 60 m/s for this machine. In another study, it was shown that cross-ground flexure specimens could be used to very selectively compare a set of six silicon nitride materials being considered for use in a large cooperative investigation with ten U.S. industrial companies. Considerable work was also done in studying the grinding properties of continuous fiber-reinforced CMCs. Studies were also done on a new CMC including alumina and aluminum. This new composite, which shows considerable promise as a new brake system friction material, is extremely difficult to machine, even with diamond grinding media.

### 2.5 METALLOGRAPHY AND TECHNICAL PHOTOGRAPHY—*J. R. Mayotte and J. W. Nave*

#### 2.5.1 Metallography — *J. R. Mayotte*

The Metallography Group provides research support in optical, general metallography, and photography of both alloy and ceramic materials. Metallographic examination is performed in collaboration with researchers within the M&C Division, ORNL, other federal laboratories, and industrial firms. This past reporting period, the Metallography team prepared 2318 metallographic specimens. Microanalysis was performed on 209 specimens; an additional 84 specimens were analyzed in the user facility.

This year, our work capabilities expanded to include specimen-preparation techniques and a procedure for radiation metallography located in Building 3500. In addition, we assisted the Research Support Group in preparing alloy and ceramic specimens for TEM and related analyses. The group participated in the National Educator's Workshop. Personnel attended ceramic specimen-preparation and electronic-imaging courses as well as the International Metallographic Society Convention.

Team members continue to improve specimen-preparation techniques for both alloy and ceramic materials, utilizing the Rotopol/Pedemat automatic grinding/polishing machine. Metallographic preparation of cross-sectioned materials for materials characterization continued as a major effort. We are exploring new capabilities such as image analysis, digital imaging from the metallograph, and transporting and storing images.

Environmental, safety, and health improvements continued during this period.

### **2.5.2 Technical Photography — J. W. Nave**

The Technical Photography Group continues to develop new ideas of service that are beneficial not only to the M&C Division, but to all of the Energy Systems facilities.

Our video capabilities are continuing to expand to include recording of training sessions, group meetings, special projects, and all documentations that require video presentations.

In addition to research photography, the group is involved in public relations photography for ORNL and M&C.

We are investigating the latest digital-imaging technology, all the capabilities of storing images instead of negatives, and the ability to produce higher quality and increased production of various type images that include computerized photographic images, still photographs, slides, and viewgraphs. We expect to have this on line by the end of this year.

We will be going to silver-recovery units this year in order to reduce our photographic chemical waste.

After a prolonged process, our lab renovation has been completed.

This past reporting period, the Technical Photography staff members completed 965 photographic work orders that included 24,336 units of photographs, photomicrographs, slides, and viewgraphs in color and black and white.

### 3. MATERIALS SCIENCE

*E. E. Bloom*

Research in the Materials Science Section has two primary objectives: (1) to contribute to the fundamental understanding of the behavior of materials and (2) to apply this understanding in the development of improved and new materials for advanced technologies. We accomplish these objectives through close coordination of our capabilities and expertise in theory, modeling, structural characterization, material synthesis, and physical and mechanical metallurgy.

The largest single effort is the research supported by the DOE BES, Division of Materials Sciences. Basic research on the electronic theory of materials (Theory Group), radiation effects (Radiation Effects in Materials Science Group), and alloy design (Alloying Behavior and Design Group) provides the foundation of understanding required for the development of high-temperature alloys and neutron-radiation-resistant structural materials. Microscopy and microanalysis research (Microscopy and Microanalytical Sciences Group) and X-ray research (X-ray Research and Applications Group) continually advance the state of the art in tools for structural characterization [TEM and analytical electron microscopy (AEM), imaging atom probe, and synchrotron X-ray sources].

We have materials development activities in the following areas: (1) alloys for high-temperature applications (centered in the Alloying Behavior and Design Group), (2) alloys for fusion reactor first wall and blanket structure applications (Structural Materials Group), (3) alloys for advanced fossil systems (Structural Materials Group), and (4) modification of the properties of ceramics and polymers by ion implantation and the synthesis of multilayered metal and ceramic structures using molecular beam epitaxy (MBE) [Radiation Effects in Materials Science and X-ray Research and Applications Groups]. Each of these efforts draws heavily on the total experience and capabilities of the M&C Division and ORNL.

#### 3.1 THEORY — *W. H. Butler*

The majority of the research in the Theory Group is aimed at providing an understanding of the properties of materials at the microscopic electronic level. This understanding is used to solve fundamental materials-related problems and to guide the design and development of new materials. To these ends, we develop new theoretical methods and computer algorithms for performing first-principles calculations of the properties of materials that continually extend the domain of applicability of *ab initio* techniques. The electronic structure and energetics of periodic solids are treated using the first-principles mixed-basis pseudo-potential method, the full-potential augmented plane wave method, and the Korringa-Kohn-Rostoker (KKR) approximation method. Random substitutional alloys are treated with the KKR-coherent-potential-approximation method. The Augmented Gaussian Basis atomic cluster technique is used to model microchemical interactions. These methods are used to solve outstanding basic research and textbook problems, to interpret experimental observations, and to aid materials development efforts, particularly in the Alloying Behavior and Design Group in this division.

For this reporting period, we highlight the following noteworthy accomplishments:

- **Basic theory and technique.** Development of a new partial-wave self-consistent field (PWSCF) method for calculating the electronic structure and total energy of clusters of atoms has been completed. The PWSCF method imposes no constraints on symmetry or composition. The energy algorithm is designed for efficient calculation of energetics with reference charge densities that may be only approximate, and offers wide flexibility in the choice of the level of completeness, permitting some sites to be treated more rigorously than others in order to balance accuracy with speed of computation.
- **Large System Multiple Scattering (LSMS) algorithm.** An LSMS algorithm for calculating the electronic structure and energetics of systems comprising large numbers of atoms has been implemented on the 512-node Intel Paragon XP/S-35 massively parallel supercomputer (MPS). The algorithm makes an equivalence between atoms in the simulation region and nodes on the MPS. The LSMS algorithm has been shown to be scalable up to systems containing 512 atoms/unit cell. Here, scalable means that the computational time required to solve a given problem increases linearly with the system size,  $N$ , rather than  $N^3$  as is typical of conventional electronic structure methods.
- A new technique for solving the Poisson equation for application to full potential multiple scattering methods has been developed. The method is superior to previous techniques used in this context.
- **Mechanical properties and support of alloy design.** A systematic study of cleavage fracture has been carried out for Ni, Fe, and Ti aluminides and trialuminides. Significantly, it is found that measured fracture toughness values are closely related to the Griffith strength for a Mode-I crack obtained using calculated elastic constants and cleavage energies. The important implication of this result is that a twofold increase in the cleavage energy can lead to a hundredfold increase in the fracture toughness.
- The point defect structure of  $\text{Ni}_3\text{Al}$  has been investigated from first principles in order to understand the compositional dependence of mechanical behavior of  $\text{L1}_2$  structure transition-metal aluminides. The absence of structural vacancies is predicted, in sharp contrast to  $\text{NiAl}$  and  $\text{FeAl}$   $\text{B2}$  aluminides.
- We have calculated the shear fault energies of the  $\text{Ni}_3(\text{Si},\text{Ti})$  system in order to understand the beneficial effect of Ti additions on the strength and ductility of  $\text{Ni}_3\text{Si}$ . The calculations indicate that, in  $\text{Ni}_3(\text{Si},\text{Ti})$ , the increased driving force for cross-slip-pinning and the corresponding strength anomaly stems from both increasing shear elastic anisotropy and decreasing (010) antiphase boundary (APB) energy.
- A cluster investigation of the contrasting segregation behaviors of boron and carbon in  $\text{NiAl}$  was completed. It was concluded that the stronger bonding of Ni to carbon, compared with boron, leads to carbon being lattice bound. Thus, carbon does not segregate to GB, as less strongly bound boron does. These results confirm experimental findings of the Alloying Behavior and Design Group.

- **Transport properties of alloys and magnetic multilayers.**  $\text{Ni}_{1-c}\text{Fe}_c$  alloys have a potentially important role to play in multilayer systems displaying giant magnetoresistance. The electrical resistivity of  $\text{Ni}_{1-c}\text{Fe}_c$  alloys including spin-orbit coupling has been calculated. Spin-orbit coupling plays a dominant role in determining the transport properties of these alloys. Thus, previous work on transport in these materials will require reevaluation.
- **Alloy phase stability.** Fermi surface nesting has identified as the driving mechanism for the long-period ordering observed in  $\text{Ag}_c\text{Mg}_{1-c}$  alloys. Calculations of the short-range-order diffuse scattering are in excellent agreement with electron diffraction diffuse scattering measures. At 75 at. % Ag, the calculated transition temperature for ordering into  $\text{L1}_2$  structure is  $\sim 670$  K compared with the experimental value of 665 K.
- **Small metallic clusters.** A study of the nature of metal-metal bonding in the first-transition metal series was extended to include all metals from H through Cu. Bulk-like trends of cohesive energies and lattice constants are found even in the few-atom regime. In addition, three families can be distinguished: alkali, sp-bonded, and transition series. The important implication of these findings is that coupling of the cluster to the bulk simply provides a rather uniform shift of bond lengths and cohesive energies to reach the bulk limit.
- **Point defects in graphite.** Point defect energetics and diffusion mechanisms in graphite have been investigated using a semi-empirical tight-binding force model. It was found that self-diffusion in the direction parallel to the basal plane can be mediated by vacancies. However, it is argued that Frenkel pairs could exist as equilibrium defects because the calculated vacancy and interstitial formation energies are nearly equal. Thus, at high temperatures, self-diffusion parallel to the basal plane should occur by an interstitial mechanism. Our calculations rule out a non-defect mechanism.

### 3.2 X-RAY RESEARCH AND APPLICATION — C. J. Sparks

As materials properties depend in a major way on the atomic arrangements, we maintain a significant capability in MBE and chemical vapor deposition (CVD) synthesis and in advanced XRD characterization. This allows us to produce new materials and to relate changes in materials properties to differences in their atomic arrangements. We are pursuing studies on optical wave guides; CVD diamond; superconductors; interfaces; binary alloys of Fe, Ni, and Cr; and X-ray optics.

Developments in the growth of  $\text{BaTiO}_3$  and  $\text{SrTiO}_3$  ferroelectric oxide thin films on  $\text{MgO}$  for optical guided wave device applications have demonstrated that optical clarity, comparable to that achieved for bulk- $\text{LiNbO}_3$ -based waveguides, can be obtained. Optical loss coefficients of 1.0 dB/cm at 705 nm are measured for our thin-film perovskite waveguides on  $\text{MgO}$ . A whole new class of waveguide structures, perovskite thin films on alkaline earth oxides, has been created. Our research is motivated by the need for epitaxial oxide materials that can be integrated as optical waveguide structures into silicon-based technology.

Models of current transport have been unable to account for spatial fluctuations in the critical current density of thin films of the high-temperature superconductor  $TlBa_2Cu_3O_x$ . It has been assumed that grain alignment develops only along the surface normal of films grown on polycrystalline substrates. With X-ray microdiffraction, we have shown that grains are locally aligned in-plane, and that regions of low-critical-current density correspond to abrupt shifts in in-plane alignment. These results led to a new current transport model in which current percolates through low-angle GBs.

X-ray measurements of the atomic pair distances in  $Fe_{22.5}Ni_{77.5}$  and  $Cr_{47}Fe_{53}$  crystalline solid-solution alloys have shown that the distances of closest approach are between atom pairs that have a chemical preference for each other. The hard sphere model is shown to be invalid as it predicts that Cr-Cr pairs would be the furthest apart since Cr additions expand the lattice.

Research into the relationships between the activated CVD growth process, structure, and properties of new diamond materials shows the effects of temperature and certain growth parameters. Impurities and minor additions to the growth environment, such as boron, can have dramatic effects on morphology and defect structure. State-of-the-art materials from commercial vendors are being characterized for infrared optical applications. Homoepitaxial and heteroepitaxial growth are being studied.

### **3.3 MICROSCOPY AND MICROANALYTICAL SCIENCES — *K. B. Alexander***

The primary focus of the group is on the characterization of materials with advanced AEM, APFIM, and MPM techniques. Research efforts concentrate on the development of microanalytical techniques to characterize the structure, chemistry, and mechanical response of materials, and on the application of these techniques to scientifically and technologically relevant materials science problems. In addition, the group maintains and develops the equipment required to support the characterization technologies. Primary research funding comes from the Division of Materials Sciences, BES. Research is also supported by other programs including Fusion Energy, Conservation, and the USNRC. Group members are directly involved with the research efforts of other groups within the M&C Division including Structural Materials, Materials Joining, Superconducting Materials, Radiation Effects, and Carbon Materials Technology (CMT).

#### **3.3.1 Electron Microscopy Research — *J. Bentley and E. A. Kenik***

Evaluation tests were made, specifications were developed, and purchase orders were placed for three major pieces of AEM instrumentation: (1) a Philips CM200ST- $\alpha$ /STEM/FEG 200-kV AEM equipped with a Schottky FEG; (2) a Philips XL30-FEG computer-controlled Schottky-FEG SEM equipped with an EDS; and (3) a Gatan imaging filter (GIF) to be installed on our Philips CM30 300-kV AEM. As part of the GIF evaluation process, nanometer-resolution elemental maps were obtained (in collaboration with O. Krivanek, Gatan R&D, Pleasanton, California, and with M. Otten, Philips Electron Optics, Eindhoven, The Netherlands) of chromium depletion at GB in a sensitized type 304 stainless steel and of radiation-induced segregation (RIS) at dislocation loops in an ion-irradiated, modified type 316 stainless steel. In preliminary measurements on Fe-doped NiAl and on  $Ni_3Al$ -based alloys, a new statistical analysis method for the atom location by channeling enhanced microanalysis method appeared to correctly account for ionization delocalization.

Applications of AEM to materials science supported by the BES Microscopy and Microanalysis Task were increasingly undertaken in collaboration with Shared Research Equipment (SHaRE) Program participants. Microanalysis of phases in Ni-Ti-O materials, procedures for accurate composition determination by EDS, and further work on a comprehensive treatment of secondary fluorescence in EDS of nonhomogeneous specimens of complex geometry were studied in a SHaRE project with C. B. Carter (University of Minnesota) and I. M. Anderson [Cornell University and now an ORNL/Oak Ridge Associated Universities (ORAU) postdoc]. In additional SHaRE research with C. B. Carter and colleagues, the crystallization of anorthite glass deposited by laser ablation onto alumina substrates was studied by *in situ* annealing, and further measurements by parallel-detection electron energy-loss spectrometry (PEELS) were made of an oxidized CoO-ZrO<sub>2</sub> eutectic. In the continuation of a SHaRE project with R. Sauerbrey and H. M. Phillips (Rice University), second-difference PEELS data indicated that pyrolysis effects, which result in oxygen and nitrogen loss, are responsible for the electrical conductivity that is induced by excimer laser irradiation of Kapton™ polyimide. In a SHaRE project with R. F. Davis and S. Tanaka (North Carolina State University), no interdiffusion of AlN and SiC was detected at low temperatures, but several complex sialon phases were identified by AEM in AlN/SiC couples contaminated with oxygen (from the use of commercial-grade AlN powder). RIS at GBs in proton-irradiated type 304 stainless steel was studied by high-spatial-resolution AEM in a SHaRE project with G. Was (University of Michigan) and in collaboration with J.-J. Kai (National Tsung Hua University, Taiwan) while on assignment at ORNL. An AEM study (SHaRE research with D. Joslin and C. J. McHargue, University of Tennessee) on ion mixing of thin ZrO<sub>2</sub> films on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> by Cr<sup>+</sup> and Kr<sup>+</sup> revealed that solid solubility was not enhanced by mixing, and that displaced Zr amorphized the Al<sub>2</sub>O<sub>3</sub> at room temperature but not at 900°C. In SHaRE research with M. Kaufman et al. (University of Florida), rapidly solidified Al-Fe alloys were characterized by AEM and their decomposition investigated by *in situ* annealing at ~400°C. Structural disorder in graphite was characterized by AEM following *in situ* 300-kV electron irradiation (with J. Koike, Oregon State University) and following ion implantation (with D. F. Pedraza). Several other SHaRE collaborations featuring AEM were active, including: dopant (B, C, and P) distribution and degree of crystallinity in microcrystalline/amorphous silicon (D. M. Maher and Y. L. Chen, North Carolina State University); GB segregation in irradiated ferritic steels (M. G. Burke, Westinghouse Science and Technology Center); *in situ* studies of nucleation and growth in Al-Zn alloys (J. J. Hoyt, Washington State University); AEM characterization of precipitation in Alloys 800 and 800H (M. Marek, Georgia Institute of Technology); precipitation in aged Hastelloys B and B2 (C. Simon and C. Brooks, University of Tennessee); thin ceramic films deposited by pulsed-laser ablation (G. Norton, Washington State University); and AEM of iron aluminides (A. Castagna and N. Stoloff, RPI).

The improved catalytic performance and lifetime of automotive catalyst systems is the goal of a CRADA with GM-AC Rochester Division (CRADA funded by the AIC Materials Program). Characterization of the morphological, structural, elemental, and chemical state of the catalysts is being studied by a variety of analytical techniques, including analytical SEM and TEM, X-ray fluorescence microanalysis, and Auger electron spectroscopy (AES).

High-spatial-resolution AEM has been performed in collaboration with the Fusion Energy Materials Program on both austenitic and ferritic materials. The work on austenitic alloys has concentrated on RIS to GBs and other point defect sinks, which is

of concern in irradiation-assisted stress-corrosion cracking. The work on ferritic alloys has dealt with radiation hardening/embrittlement.

### **3.3.2 Atom Probe Research — *M. K. Miller***

The main instrument development effort has been the design and construction of the mapping atom probe. Further enhancements of the atom probe control and analysis software package and the software for the MAP were made. Work continued on the 3-D characterization of the scale and composition of  $\alpha$  and  $\alpha'$  phases that form during the early stages of phase separation within the low-temperature miscibility gap in the FeCr system. The atom probe data were fitted with the use of moment methods to some new models of the decomposition process. New facilities for preparing APFIM needles of irradiated pressure-vessel steels and model alloys were completed, and research into these materials was resumed. A project to characterize the decomposition in irradiated austenitic stainless steels was initiated. Characterization of unirradiated, commercial, Russian-type 15Kh2MFA (VVER 440) Cr-Mo-V and 15Kh2NMFA (VVER 1000) Ni-Cr-Mo-V pressure-vessel steels (with G.-Brauer, Research Center Rossendorf, Dresden, and P.-Othen, Oxford University) revealed extremely high phosphorus segregation to, and ultrafine precipitation of, molybdenum carbo-nitride precipitates at the lath boundaries. Solute partitioning and  $\gamma$  precipitation was characterized as a function of heat treatment in commercial, single-crystal, nickel-based superalloy, Alloy PWA 1480, turbine blades (with L. S. Lin, Pratt and Whitney). Ultrafine-scale precipitation was characterized and related to the changes in the mechanical properties in boron-doped NiAl single crystals. The site occupancy of iron in the B2-ordered NiAl lattice was determined.

### **3.3.3 MPM Research — *W. C. Oliver***

The MPM research at ORNL has been enhanced by the completion of a specially designed laboratory space with precise environmental control to allow the highest quality data to be obtained from these sensitive instruments. The facility includes five research instruments. These are a Nanoindenter II designed for very high-resolution ambient temperature indentation experiments, a Nanoindenter I modified to perform ultralow load scratch experiments, the high-temperature mechanical properties microprobe (HTMPM), an AFM, and a Lietz microhardness tester. Research efforts to examine the measurement of fracture toughness using indentation experiments, and the effects of residual stresses on the measurement of hardness and modulus measurements, are nearing completion. Finite element modeling of the residual stress effects is proving to be very useful. The finite element modeling efforts are now being extended to investigate improved techniques for hardness and modulus measurements that account for material pile-up near the indentation and to examine the problem of films on substrates. The high-speed data acquisition available on the HTMPM (150 kHz) is proving most useful. Initial experiments indicate that it will be possible to measure internal damping with indentation experiments. The modeling of the indentation process is being extended to more sophisticated, time-dependent material constitutive equations. A study has been initiated to examine the effects of crystalline anisotropy on the measurements made with the MPM.

### 3.4 ALLOYING BEHAVIOR AND DESIGN — C. T. Liu

The primary goal of the Alloying Behavior and Design Group is to generate understanding of alloying behavior and structure/property relationships in metallic and intermetallic alloys so that the design principles for new alloys to meet specific energy technology needs can be developed. Group activities have focused on four major tasks: (1) understanding of physical metallurgy and mechanical behavior of ordered intermetallics, (2) design and characterization of intermetallic alloys for industrial and energy-related use, (3) development and qualification of iridium alloys for space power systems, and (4) processing of metallic and intermetallic materials by innovative methods (e.g., rapid solidification). The first two tasks are closely related, with emphasis on design of new high-temperature structural materials through control of alloy composition, crystal structure, atomic bonding, microstructure, and processing techniques.

Our earlier experimental work had demonstrated that the ductility of carefully prepared specimens of  $\text{Ni}_3\text{Al}$  increases significantly when tested in relatively moisture-free environments (like dry oxygen). We recently extended that work by measuring the ductility of  $\text{Ni}_3\text{Al}$  as a function of vacuum level from  $\sim 105$  Pa (in air) to  $\sim 10^{-8}$  Pa (in ultrahigh vacuum). One of our objectives was to determine by how much the ductility increases when the test is performed in ultrahigh vacuum, i.e., when the moisture content is reduced to extremely low levels. Put another way, conventional vacuums may not be good enough to entirely eliminate environmental embrittlement, and testing in ultrahigh vacuum may be necessary to determine the intrinsic properties of GBs in  $\text{Ni}_3\text{Al}$ . Our major finding is that polycrystalline, B-free  $\text{Ni}_3\text{Al}$  (23.4 at. % Al), produced by cold-working and recrystallizing a  $<100>$  single crystal, exhibits room-temperature tensile ductility of 23.4% in ultrahigh vacuum. This is the highest ever ductility reported for equiaxed, polycrystalline, B-free  $\text{Ni}_3\text{Al}$ . Ductility decreases systematically with increasing amounts of air in the test environment: 23% at  $10^{-8}$  Pa, 13% at  $10^{-4}$  Pa, 8% at  $10^{-1}$  Pa, and 3% at  $10^5$  Pa. Moisture in air was identified as a major cause of this environmental embrittlement. We speculate that oxygen competes with moisture for the oxidation of aluminum, thereby lowering the amount of atomic hydrogen generated for a given amount of moisture in the environment.

Near-stoichiometric  $\text{NiAl}$  alloys exhibit mainly intergranular fracture at ambient temperatures.  $\text{NiAl}$  doped with 300 wt ppm C also shows GB fracture at room temperature, whereas  $\text{NiAl}$  doped with 300 wt ppm B exhibits mainly transgranular fracture. In addition to suppressing intergranular fracture, the boron addition was found to dramatically increase the yield strength and reduce plastic deformation in  $\text{NiAl}$ . These results suggest two possibilities: (1) boron segregation enhances GB cohesion and suppresses GBs fracture, and (2) boron suppresses plastic flow and reduces strain incompatibility and intergranular fracture. To separate these two factors, we are studying the GB fracture behavior of  $\text{NiAl}$  doped separately with boron and carbon as a function of compressive strain at room temperature. Our results clearly indicate that the GBs in  $\text{NiAl}$  are relatively weak (as compared with bulk material) and that boron segregation increases GB cohesion in  $\text{NiAl}$ .

The effect of boron doping and deviations from stoichiometry were investigated in two B2 alloys,  $\text{NiAl}$  and  $\text{FeAl}$ . Boron was found to segregate to the GBs of both these alloys. However, in the case of  $\text{NiAl}$ , only in the stoichiometric and Ni-rich alloys was boron able to suppress intergranular fracture. In the case of  $\text{FeAl}$ , only in the very Fe-rich alloys ( $\text{Al} \leq 43\%$ ) was boron able to suppress intergranular fracture. In both alloy systems, with increasing Al concentration, the GBs became progressively more brittle until, at

some Al level, they were so brittle that even though boron segregated to the GBs, it was unable to improve cohesion and suppress intergranular fracture. These observations are reminiscent of those reported earlier for Ni<sub>3</sub>Al alloys and indicate a possibly systematic variation in GB structure with Al concentration in many different aluminide systems.

As evidenced by slow crack-growth studies, micro- and macro-alloying additions have a distinct influence on moisture-induced hydrogen embrittlement. Since NiAl does not appear to be sensitive to environmental embrittlement, and since FeAl and NiAl form a pseudobinary solid solution, it was decided to replace some of the Fe in FeAl by Ni. Within our experimental window, the Fe-10Ni-40Al (at. %) intermetallic was not as sensitive to the crack growth velocity as Fe-40Al. However, at the same time, the Ni addition increased the yield strength and reduced the fracture toughness significantly. A small addition of B (0.1 at. %) enhanced the mechanical properties only slightly, even though it changed the fracture mode from partly intergranular to fully transgranular. Experimental work on the effect of other soluble ternary alloying additions has been started. Whereas Cr additions to Fe-45 Al show results similar to those found for Ni, Mn additions result in a pronounced dependence of the crack growth resistance on the crack propagation velocity.

Previous work showed either a small or no anomalous yield-strength peak in FeAl (i.e., yield strength increasing instead of decreasing with temperature). Recently, with the realization that vacancies can cause significant low-temperature hardening and thus mask the anomalous yield strength peak, we have carefully annealed out the vacancies in FeAl and reproducibly shown anomalous yielding in several different FeAl alloys. At low temperatures (below room temperature), the yield strength of FeAl behaves like that of typical body-centered cubic metals (decreasing sharply with increasing temperature). Above room temperature, however, the yield strength increases, rather than decreases, with increasing temperature. It peaks around 600 to 900 K and then decreases sharply with further increases in temperature. Boron doping not only shifts all the yield strength-temperature curves upward, but also shifts the peak temperatures higher. There is, as yet, no clear mechanism to explain this anomalous yielding behavior in FeAl, although there are several possibilities including cross-slip pinning, APB dragging, and change in slip system.

Although MoSi<sub>2</sub> has a high melting point (2020°C), moderately low density (6.3 g/cc), and excellent high-temperature oxidation resistance, its high-temperature mechanical properties, its susceptibility to disintegration during oxidation at temperatures near 500°C, and difficulties in fabrication continue to draw research efforts across the country. We have devoted considerable effort to understanding the so-called pest reaction in MoSi<sub>2</sub> oxidized at 400 to 600°C. We have found that the disintegration of MoSi<sub>2</sub> into powder (pest reaction) is caused by formation of MoO<sub>3</sub> on the walls of cracks and cavities (and eventually along GBs) when exposed to oxygen in this temperature range. At higher temperatures, two events prevent pesting: (1) SiO<sub>2</sub> tends to form quickly on surfaces, protecting against MoO<sub>3</sub> formation, and (2) the MoO<sub>3</sub> that does form is volatilized, so that no expansion occurs and the material remains intact. We have found that the susceptibility to pesting depends not only on the temperature but also on the amount of silicon present, the presence of other elements, and on the density of the material. The addition of extra silicon or small amounts of elements such as Al or Cr tends to delay the initiation of pesting, as does increased density.

The comparative studies of Ni<sub>3</sub>Al and Ni<sub>3</sub>Si have been extended to include the cleavage fracture behavior and the effect of alloying additions on strength and ductility. The ideal

Griffith energies and the critical stress-intensity factors for three cleavage modes were determined from the calculated elastic constants and surface energies. The propensity for crack-tip deformation was estimated on the basis of the calculated APB and superlattice intrinsic stacking fault (SISF) energies and the anisotropic coupling effect on the dislocation mobility, i.e., non-Schmid effects. While the calculated Griffith energy of  $\text{Ni}_3\text{Si}$  is larger than that of  $\text{Ni}_3\text{Al}$ , dislocation emission from a crack tip is easier, and dislocation mobility is higher in  $\text{Ni}_3\text{Al}$  than in  $\text{Ni}_3\text{Si}$ . When a crack is loaded in a mixed mode ( $K_I+K_{I\parallel}$ ), emission of super-Shockley partials from the crack, trailing extended SISFs, is predicted and confirmed by in situ straining TEM observations of crack tips in  $\text{Ni}_3\text{Al}$ . The activation energy of dislocation emission from a crack tip is estimated to be higher in  $\text{Ni}_3\text{Si}$  than in  $\text{Ni}_3\text{Al}$  by more than a factor of two. This estimate is based on the larger (by about four times) APB energy in  $\text{Ni}_3\text{Si}$  as compared with  $\text{Ni}_3\text{Al}$ , which is attributed to the polarization of an extra p-electron in  $\text{Ni}_3\text{Si}$ . This suggests an alloy design strategy of searching for a ternary element that reduces the APB energy, such as Ti in  $\text{Ni}_3(\text{Si},\text{Ti})$ .

In order to better understand the composition dependencies of mechanical properties of transition-metal aluminides, the bonding mechanisms and point defect structures in  $\text{Ni}_3\text{Al}$  are investigated using first-principles quantum mechanical calculations of defect self-energies and thermodynamic analyses of defect concentrations. In a sharp contrast to the case of the B2-type aluminides (e.g.,  $\text{NiAl}$  and  $\text{FeAl}$ ), the absence of structural vacancies is predicted in  $\text{Ni}_3\text{Al}$  of the  $\text{L}_1_2$  structure. The enthalpy of vacancy formation at the Ni sites is 1.85 eV. This result is consistent with the earlier results on  $\text{TiAl}$  in that the dominant defects in transition-metal aluminides of the close-packed structures are substitutional antisite defects. Also consistent with this result is the defect hardening of  $\text{Ni}_3\text{Al}$ , observed by means of hardness and yield strength measurements, at both hyper- and hypo-stoichiometric compositions.

A new reaction synthesis system has been designed for the study of processing of aluminides in controlled environments and with the application of load during the reaction. The system has the capability of applying a very rapid load stroke controlled up to 1 in./s. The load capacity is 110 kips and can accommodate samples up to 8 cm diam and 2.54 cm thick. Temperature-controlled compression platens will provide variations in heat flow to establish certain temperature gradients across the samples. The high-speed (1000 frames/s) color video camera will be used to record the reaction in detail through a cooled borescope focused on the reaction region.

The effects of aluminum concentration and green compact thickness on the reaction synthesis of powder compacts containing 25 to 40 at. % Al are studied under a uniaxial compressive stress of 10 MPa. The results indicate that both reaction product and material density are sensitive to these two parameters. For all powder compacts with a thickness of 5 mm, no temperature increase is detected during reaction synthesis, indicating no formation of transient liquid phases and thus no self-sustaining combustion reaction. For 6- and 7-mm-thick compacts containing 40% Al, a full synthesis reaction took place, resulting in production of the  $\text{NiAl}$  alloy with a high material density (>98%) and a fine grain size (14  $\mu\text{m}$ ). Other compacts containing 25 and 30% Al showed a coexistence of partially and fully reacted zones, with the reaction zone size strongly dependent on aluminum concentration and compact thickness. The reaction kinetics are explained in terms of local heat accumulation and heat transfer as well as thermodynamic analyses of exothermic reaction during each reaction step.

Microstructure and mechanical properties of Cr-Nb alloys containing 5.6 to 17% Nb were studied for high-temperature structural applications in corrosive environments. The alloys basically contain the two phases of Cr<sub>2</sub>Nb- and Cr-rich solid solution. Re, Al, Ni, Fe, and Co were added to the alloys for control of microstructure and properties. The alloys show excellent strength at temperatures to 1250°C. Among the alloying elements, rhenium is found to be most effective in improving the mechanical properties at room and elevated temperatures. Alloying with  $\geq 8\%$  Al, Ni, and Co, on the other hand, lowers the strength and ductility at elevated temperatures. Further development is required to improve the fracture resistance of the alloys at ambient temperatures.

Efforts on alloy development of Fe<sub>3</sub>Al, sponsored by the Fossil Energy Advanced Research and Technology Development (AR&TD) Program, have centered on understanding the mechanism of room-temperature embrittlement in this system and developing Fe<sub>3</sub>Al-based alloys with improved ambient-temperature ductilities and increased strengths at temperatures of 600 to 700°C. It has been determined that ambient temperature brittleness in this system is not "inherent" but is caused by atomic hydrogen, which is produced by an environmental reaction between aluminum in the alloy and water vapor in the atmosphere. During the past 5 years, great strides have been made in understanding this embrittlement phenomenon, and the production of alloys with room-temperature ductilities of over 10% and tensile yield strengths at 600°C of as high as 500 MPa is now possible through modifications in alloy composition and control of thermomechanical processing techniques. Recent studies of Fe<sub>3</sub>Al alloys have also indicated that creep-rupture lives of over 1000 h at 593°C (1100°F) and 207 MPa (30 ksi) can be produced through control of alloy composition and microstructure. These improved properties compare well with many of the iron- and nickel-based alloys currently used for structural applications.

Efforts on FeAl alloy development have included: (a) alloying for improved weldability and (b) microalloying and microstructural control through processing/heat-treatment variations for improved high-temperature strength and creep-rupture resistance, combined with better room-temperature ductility. Scientific understanding of the mechanisms underlying alloying or microstructural effects on properties behavior has been crucial to obtaining substantial improvements in an efficient and effective manner. The FeAl alloys with improved weldability are currently being patented. These alloys also have been applied directly as corrosion/oxidation-resistant weld-overlay cladding on commercial structural materials, like 2.25 Cr-1 Mo and 304L-type steels. Iron-aluminide weld consumables have been produced commercially that allow crack-free FeAl weld-overlay deposits to be made on 2.25 Cr-1 Mo steel substrates with reasonable preheat and PWHTs. These efforts aimed at developing materials technology for industrial applications have also been supported by specialized scientific studies, like neutron diffraction mapping of residual stresses in the FeAl weld-overlay cladding and the steel substrate.

In our research on ductile-phase-toughened ceramics, as well as ceramic particulate MMCs, Ni<sub>3</sub>Al alloys have been optimized for wetting on, and bonding to, alumina. Wetting angles between 75 and 80° have been achieved by controlled carbon additions to the Ni<sub>3</sub>Al. Screening tests with Ni<sub>3</sub>Al/Al<sub>2</sub>O<sub>3</sub> laminate composites were performed to optimize the bonding and energy absorption of the Ni<sub>3</sub>Al. While vacuum infiltration of porous Al<sub>2</sub>O<sub>3</sub> preforms has not been successful to date, preforms made from TiC, which exhibits good wetting by Ni<sub>3</sub>Al, can be infiltrated to make ductile-phase-toughened composites.

The DOE, through subcontracts with Energy Systems and others, provides the RTG to NASA for space missions. As part of that task, efforts in the group include qualification and characterization of the currently used DOP-26 iridium alloy, used for shielding of plutonium fuel in the RTG, and alloy development to produce iridium-based alloys with improved properties. As part of our qualification and characterization studies, we were able to show that new- and old-process DOP-26 iridium alloys have essentially identical high-temperature impact ductilities, and the same oxidation compatibility when exposed to low-pressure oxygen at temperatures over 1250°C. As a result of our alloy upgrade efforts, two modified Ir-0.3 W alloys have been developed which have substantially better weldabilities and comparable high-temperature impact properties. These alloys appear to be extremely promising as potential replacements for the currently used DOP-26 alloy because, not only do they have better weldabilities, but they also contain a much lower amount of radioactive thorium, making handling and transportation much easier, safer, and economical. Our efforts as part of this task have contributed to the success of past space missions, such as Galileo, and are essential for the success of the future Cassini mission to Saturn.

Progress has been made on a program funded by the Development Division at the Oak Ridge Y-12 Plant to produce lithium hydride flakes and powder. Bulk LiH previously crushed into small particles is processed into flakes/powder by the melt-spinning process prototype. This melt-spinning process provides uniaxially solidified flakes with few voids that, in turn, have produced compacts of almost theoretical density with very high compression strengths. Unfortunately, the bulk material used in this work contains iron impurities from both the salt manufacturing and the subsequent crushing operations, which presents a large problem in melt spinning. The iron stops up the orifice in the melt crucible after a few runs, requiring a shutdown of the system. A method has been set up within a glovebox to magnetically screen the crushed material to remove the iron particles prior to the melt-spinning processing. Additionally, stainless steel screens are used inside the melt crucibles to separate the iron from the LiH. These removal methods have been only partially successful, and it is suggested that improvements in the original salt manufacturing should be made to yield material with less contamination.

Overall structure-properties characterization has been done in support of three major General Motors Research/Energy Systems CRADA tasks: (1) formability/uniformity of high-strength steels (HSS), (2) fatigue of MMCs, and (c) wear resistance of MMCs. In the area of HSS, large pieces of a full-scale commercially produced and processed heat of steel were examined to completely characterize the microstructural evolution during various stages of the actual industrial processing sequence. These stages included hot-rolled strip, fully hard cold-rolled sheet, and galvanized and temper-rolled sheet processed through a continuous annealing line (CAL). These characterization efforts of industrially produced material and some supporting laboratory tests provided critical input for the development of computational modeling of CAL processing, particularly recrystallization. The ultimate objective is to provide a tool that allows consistent processing of HSS to meet more demanding automotive forming applications. In the area of wear-resistant MMCs, characterization of aging, combustion environment, and mechanical wear effects on the microstructure and microcomposition of a base-metal engine-tested piston helped provide critical input into the design of an appropriate wear-testing device to simulate in-service piston performance. In the area of fatigue of MMCs, microcharacterization of mechanical failure surfaces was used to identify the life-limiting defects that initiate fracture. Moreover, analysis of the complex, coarser reinforcement/intermetallic and ultrafine matrix precipitate components of the

microstructure is in progress to better understand the fatigue behavior of the MMC and processing avenues to improved MMC performance.

### 3.5 RADIATION EFFECTS IN MATERIALS SCIENCE — *L. K. Mansur*

The capability to irradiate materials is a dimension of materials science that, like temperature, potentially affects all processes and properties. Research in the group covers the science underlying the effects of displacement- and ionization-producing particle fluxes on materials. The two specific areas on which the work currently focusses are the behavior of structural materials for fission and fusion reactor applications and the development of new materials and properties by ion beam treatments. Theory and a variety of experimental techniques are combined to attack major research issues.

#### 3.5.1 Radiation Effects in Reactor Environments

The primary research is on the mechanisms of pressure-vessel steel embrittlement. Part of this work is focussed on questions surrounding embrittlement of the HFIR pressure vessel, but the work is also broader, covering the more general aspects applicable to power reactor vessels, support structures, and other components. In addition, we are working with the ANS design team to project and assess materials performance, especially related to aluminum alloys, in that system.

Our work during the past year has led to the discovery that high  $\gamma$  fluxes are the probable cause of accelerated embrittlement at some locations on the HFIR pressure vessel. This work is the first to identify  $\gamma$ -rays as a major source of embrittlement in metallic alloys and to uncover the conditions under which they are important. In the course of this work, we raised new perspectives in embrittlement and drew wide attention to the role of neutron spectrum, particularly thermal neutrons. We demonstrated clearly that the impurities, copper and boron, are not the cause of the accelerated embrittlement. We investigated low displacement rate and softened neutron spectrum as major suspects. Using a combination of experiments and a model we have developed for embrittlement at low temperatures during the point defect buildup transient period, we have deduced that a low displacement rate is not responsible. With regard to a softened spectrum, we are pursuing experiments to quantify the magnitude of the effect. At locations where the neutron spectrum is highly thermalized, our analysis indicates that the  $(n, \gamma)$  recoils produced by thermal neutrons may make a significant contribution to the embrittlement. Where the ratio of hard  $\gamma$  flux-to-fast neutron flux is greater than about  $10^3$ , our analysis indicates that  $\gamma$ -induced displacements caused by Compton scattered electrons and pair production will be the primary factor in the embrittlement. Our work in this area has stimulated international efforts to focus on the possible effects of  $\gamma$ -irradiation.

In related experimental work on the basics of embrittling microstructures, a program is under way to measure the barrier strengths of small defect clusters. Iron ion irradiations are being employed to obtain irradiated specimens of nine model alloys at a range of doses in a timely way. The microstructural characterization of the unirradiated materials and the first irradiations of five of the alloys have been completed. Preliminary examination of the irradiated specimens by TEM and hardness measurements on the

ORNL Nanoindenter have begun. These first data have demonstrated the qualitative feasibility of our plan to correlate radiation-induced hardness with the observed cluster densities and thereby determine the required dislocation obstacle strengths. More detailed measurements of the defect cluster density, size distributions, and hardening will be obtained.

The modeling work has been advanced on two fronts. The method of molecular dynamics (MD) has been used to simulate the formation and evolution of displacement cascades in iron. The MD work is being carried out in collaboration with investigators from the University of Liverpool and the Harwell Laboratory in the United Kingdom. The goal of this work is to characterize the evolution of displacement cascades as a function of primary knock-on atom (pka) energy and temperature. The ability to conduct simulations of high-energy cascades (pka energy > 5 keV) is limited by the cost and availability of computer time, but seven 10-keV cascades have been completed at ORNL. A greater number of low-energy cascades have been completed at Liverpool, Harwell, and ORNL. Analysis of the results indicates that some of the general trends observed in face-centered cubic simulations are also seen in iron. However, the fraction of the residual point defects that are observed to be in clusters appears to be reduced in iron when compared to copper, and the clustering fraction also appears to be reduced. The primary defect production parameters obtained from the MD have been used to improve the kinetic model discussed below.

The second advance in the embrittlement modeling was the development and incorporation of a copper precipitate hardening model. The use of this composite model is permitting a comparison of the relative importance of point defect clusters and copper-rich precipitates in pressure-vessel embrittlement. Preliminary results indicate that both defect types have regimes in which they dominate. Point defect clusters dominate at lower temperatures, higher fluxes, and lower fluences with the precipitates being more important at the alternate conditions. The results imply that data extrapolations in these variables could be misleading if the relative importance of both defect types is not considered.

Despite sporadic claims in the literature, we have found no radiation softening in pre-hardened aluminum alloys that have been neutron irradiated over a wide range of fluence, including a simulation of the fluence and flux conditions used in one of the previous studies. One difference between ours and the earlier experiments is that we ensured that our specimens were in direct contact with cooling water during irradiation so that the temperature remained about 60°C. In work by others, the specimens were irradiated in a sealed can, where nuclear heating would have raised the temperature. In another softening report, the authors used bombardments with very high-energy protons (600 to 800 MeV) to demonstrate softening in tensile specimens. We could not duplicate their bombardment conditions. However, we conducted an experiment in which we used lower energy protons (2.5 MeV) to create similar damage levels in the near-surface regions of targets held at well-controlled low temperature. Interrogation of the bombarded regions by microhardness measurements and TEM revealed no evidence of softening.

The phenomena of radiation-induced conductivity (RIC) and radiation-induced electrical degradation (RIED) have been identified as potentially limiting the use of ceramic

insulators in near-term fusion reactor designs. We have designed and built an irradiation chamber that will permit the measurement of electrical properties at temperatures of up to 600°C on ceramic thin films while the specimen is being irradiated in the ORNL triple-ion accelerator laboratory. The technique of growing acceptable ceramic films has been demonstrated, and we shortly expect to conduct our first irradiations. Both single and multiple ion beams will be applied to examine the relative importance of ionizing and displacive irradiation; the initial experiments will focus on aluminum oxide (alumina). These experiments should help resolve a controversy that recently arose in which some investigators stated that RIED may be an artifact arising out of certain experimental conditions.

### 3.5.2 Radiation Effects in Materials Modification

This work is an outgrowth of our more "classical" research on radiation effects. The profound modification of materials that is made possible by ion irradiation derives both from energy loss processes (primarily displacement damage, ionization, and excitation) and from the contributions of the implanted ions themselves. Highly defected microstructures and substantially altered compositions are produced. These, in turn, give rise to unlimited possibilities to develop new materials and properties.

Our emphasis at the present time is on polymers because of the highly successful results we have obtained recently. The work consists of both fundamental research and technologically oriented development of new materials and properties. Ion irradiation offers a unique way of modifying the properties of polymers because the ion irradiation produces orders of magnitude higher density of ionization and atomic displacement than do electron or gamma irradiation.

Significant progress has been made in understanding the primary processes and mechanisms of polymer properties modification by energetic ion beams. The results indicate that (1) among the four major energy loss processes (electronic, nuclear, phonon, and plasmon), the first two are largely responsible for changes in the material properties; (2) electronic processes are the major cause for the hydrogen evolution and crosslinking; (3) the nuclear processes are responsible for the loss of large carbon molecules by chain scission; (4) material properties depend upon ionization and displacement linear energy transfer (LET); and (5) LET effects can be controlled by ion energy and species. Optical density, electrical conductivity, hardness, and wear resistance are all enhanced by ion bombardment, showing similar dependence on ion energy, fluence, and ion species. Electrical conductivity is enhanced by the increased charge carrier concentration,  $\pi$ -electrons from the unsaturated bonds, and the increased charge carrier mobility due to crosslinking. Hardness and wear resistance are also increased by the enhanced crosslinking.

To oversimplify, higher ionization LET generally leads to improved properties; higher displacement LET generally leads to degradation. Wear resistance is, however, often limited by the deformation of the soft bulk polymer underneath the ion-beam-hardened surface layer. We have made a major advance on this problem by employing graded ion bombardment: first, bombardment is carried out with high-energy light ions, such as He, to introduce a stiff layer throughout a greater depth, which can resist local deformation;

second, bombardment is carried out with a high-ionization LET ion species (e.g., MeV range N, O, Ne, Ar) to introduce a more highly crosslinked and shallower surface layer. In polymers treated with these dual beams, wear resistance improved remarkably.

In order to understand the mechanisms by which ion irradiation alters the surface properties of polymers, we have been measuring and modeling the absolute magnitude and time dependence of gases released during ion irradiation. When the energetic ions are slowed down, a violent disruption of the molecular bonding in the macromolecules takes place along the ion track. As atoms recombine, new bonds and smaller molecules are formed, and a net rearrangement of remaining macromolecules results. Small molecules with low chemical activity, such as H<sub>2</sub> and CO<sub>2</sub>, diffuse until they escape the surface of the polymer. The composition of the remaining polymer is thus altered. In general, this alteration cannot be produced by heat-induced chemical reactions because such reactions are homogeneous and subject to chemical equilibrium constraints. We have continued measuring the residual gases evolved during our ion implantation of polymers and determining G-values (number of molecules released per 100 eV of absorbed ion beam energy). Theoretical models of the chemical reaction rates between radicals formed during ion irradiation are being developed. In a homogeneous reaction-rate model that we have developed, all of the radicals are assumed to be produced from the average LET and distributed uniformly in the target; the radicals continually interact at a rate determined by their average concentration. In our very recent nonhomogeneous reaction rate model, the early time evolution of H<sub>2</sub> in a single ion track during the ion irradiation of polymers is calculated.

In the next year, our more technologically oriented research will focus on the development of (1) corrosion-resistant, impermeable, hard-surface polymers and (2) scratch-resistant and attractive, colored optical lenses. Chemical resistance and water absorption characteristics of selected polymers, such as polystyrene, Mylar, and polycarbonate, will be studied as a function of ion energy, fluence, and ion species. The work will consist of three distinct major activities: (1) multiple ion beam processing of polymers; (2) measurement of hardness, wear, and erosion resistance, as well as other properties, such as permeability, resistance to swelling produced by solvents, and optical transmission and absorption characteristics; and (3) characterization and modeling of ion-beam-modified polymer structures and their relation to properties using a number of analytical tools including TEM, residual gas analysis, Rutherford backscattering spectroscopy, FTIR, and Raman spectroscopy.

### 3.6 STRUCTURAL MATERIALS — A. F. Rowcliffe

The primary focus of the group is the development of structural materials for fusion and fission reactor applications. The multinational program to design and build the ITER began a detailed engineering design phase in 1993. During this phase, "ITER credits" were awarded to various groups for all aspects of engineering, design, and technology R&D. ORNL work in support of ITER materials R&D is concentrated in three areas: (1) structural alloys for the first wall and shield, (2) heat sink materials for the divertor, and (3) ceramic materials for diagnostic systems and for ion cyclotron heating systems. In non-ITER-related areas, work is continuing on reduced-activation materials based on ferritic-martensitic steels and on silicon carbide composites.

In the area of austenitic stainless steels, two spectrally tailored capsules operated in HFIR removable beryllium positions. These experiments reproduce the temperatures, damage rate, and helium generation rate characteristic of ITER. Candidate U.S. and Japanese alloys will be irradiated to the goal ITER fluence of 20 dpa; measurements include tensile, irradiation creep, fatigue, electrochemical, and swelling properties. To assess the effects of irradiation on fracture toughness in the ITER-relevant temperature regime of 50 to 300°C, three HFIR capsules containing 130 compact tension specimens were irradiated in target positions to a dose of 3.5 dpa at ~90 and 270°C. All materials experienced a greater decrease in toughness following irradiation at 270°C than 90°C. However, with the exception of one air-melted heat of 316 stainless steel, fracture toughness values remained high. Values of  $K_J$  for all test temperatures remained above 150 MPa  $\sqrt{m}$  with no indications of brittle behavior. Cold-working and welding was observed to produce lower toughness values in both unirradiated and irradiated specimens.

It has been demonstrated that the irradiation swelling behavior of austenitic stainless steels is sensitive to temperature, neutron fluence, dose rate, helium generation rate, and temperature history. A single variable experiment in helium generation rate was designed using American Iron and Steel Institute (AISI) 316 alloys containing various ratios of the isotopes  $^{58}\text{Ni}$  and  $^{60}\text{Ni}$ . During neutron irradiation in the HFIR, helium was generated via  $(n, \alpha)$  reactions with  $^{58}\text{Ni}$ . The ratio of the two isotopes was adjusted so that helium generation rates of 1, 4, and 12 appm He/dpa were produced. Swelling data were obtained via high-precision density measurements on 3-mm-diam disks following irradiation to 18 dpa at 400 and 500°C. For all three helium generation rates, swelling in solution-annealed AISI 316 was found to be less than 0.1% at 400°C and less than 0.5% at 500°C. Further analysis of these and other related data is in progress to assess the possible influence of He on irradiation behavior. Three additional HFIR target capsules were assembled and inserted into the HFIR this year. Each capsule contains several hundred isotopically tailored steels in order to expand the alloy-dose-temperature-He generation rate test matrix.

Repair and replacement of ITER components will involve welding irradiated stainless steels containing helium. In a cooperative program with Auburn University, helium was introduced into stainless steels by tritium decay. The threshold for GB embrittlement during welding was found to be between 0.5 and 1.0 at. % helium in type 316 stainless steel. It was shown that application of a compressive stress can reduce the incidence of cracking by suppressing helium bubble growth on boundaries oriented perpendicular to the applied stress direction.

The high thermal conductivity and low thermal expansion of vanadium alloys makes this class of materials very attractive for high-heat-flux structural applications in fusion devices. A new program was launched this year under an ITER R&D task to examine Charpy impact, fatigue, elastic properties, physical properties, environmental compatibility, and welding. Room-temperature elastic modulii were determined for V-5Cr-5Ti, as well as elevated-temperature-specific heat, thermal expansion, and thermal conductivity. Room-temperature fatigue was determined for fatigue lives up to 100,000 cycles. The fatigue life under these conditions was found to be similar to that of type 316 stainless steel. Preliminary welding results indicate that V-5Cr-5Ti is resistant to hot-cracking. Welding in a controlled glove box environment is being extended to thick sections. Thus far, a successful weld has been made in 6-mm plate.

Earlier studies reported by workers at ANL had indicated large variations in ductile-to-brittle transition temperatures (DBTTs) between various heats of V-Cr-Ti alloys within a narrow range of Cr and Ti concentrations. One explanation advanced to explain this sensitivity is the presence of significant concentrations of chlorine derived from the Ti sponge feedstock used in alloying. A careful Auger investigation of GBs exposed by *in situ* fracture failed to detect the presence of chlorine. Sulfur, however, may be playing a role in the initiation of the intergranular fracture that develops in the transition temperature region.

Oxide-dispersion-strengthened copper is a leading candidate material for the ITER first wall structure and divertor heat sink. Several different grades of dispersion-strengthened copper have been irradiated in the Russian SM-3 reactor in a collaborative experiment with the Efremov Institute, St. Petersburg. Cadmium shielding was employed in the irradiation capsules to produce solid transmutation rates comparable to fusion levels, and some of the specimens were doped with boron to produce fusion-relevant He generation rates. The experiment will provide tensile, swelling, conductivity, and creep data at irradiation temperatures of 100, 250, and 350°C at doses of 0.5 and 5 dpa. Postirradiation mechanical property testing will be carried out under subcontract in Russia, and TEM and swelling measurements will be performed at ORNL.

Microstructural analysis of several different copper irradiation experiments was completed. The small triangular platelets of  $\text{Al}_2\text{O}_3$  particles in dispersion-strengthened copper (average edge length of 10 nm and  $\sim$ 2 nm thickness) were observed to gradually transform into nearly circular disks (average diameter  $\sim$ 6 nm) during 4-MeV  $\text{Ar}^+$  ion irradiation up to doses of about 20 dpa, due to ballistic dissolution associated with the energetic displacement cascades. This gradual ballistic dissolution is not expected to produce any serious degradation in the mechanical properties of the dispersion-strengthened copper. The dose and temperature dependence of "black spot" defect cluster formation was measured in pure copper irradiated with 750-MeV protons up to 2 dpa at 50 to 200°C. The 750-MeV protons produce displacement cascades that are more energetic than D-T fusion neutrons, and are useful for investigating fundamental aspects of radiation damage in metals. Solid-solution alloying additions such as Ni and Mn caused a decrease in the density of stacking fault tetrahedra and a dramatic increase in the interstitial dislocation loop density compared to irradiated pure copper.

Ceramic materials play an essential role in the operation of diagnostic and beam-heating systems in the ITER. However, the fundamentals of radiation damage in ceramics are not as well known as in metals. Several *in situ* experiments were performed to measure the electrical properties of ceramic insulators during irradiation. These types of experiments have recently become very important due to reports that indicate a severe, permanent degradation in the electrical resistivity of ceramic insulators may occur if the specimens are irradiated with an applied electric field. In a collaborative effort with researchers from Forschungszentrum Jülich in Germany, evidence was obtained that previously reported accounts of permanent RIED in ceramics may be simply an experimental artifact associated with surface contamination effects. Electrical measurements were made on  $\text{Al}_2\text{O}_3$ ,  $\text{MgAl}_2\text{O}_4$ ,  $\text{AlN}$ , and  $\text{Si}_3\text{N}_4$  during irradiation with 28-MeV  $\text{He}^+$  ions while an electric field of 500 V/mm was applied. No evidence for permanent degradation in the bulk resistivity was obtained up to damage levels of about 0.05 dpa. However, detailed electrical measurements showed that significant surface leakage currents associated with hydrocarbon deposition from the residual gas in the target chamber could occur even when guard rings were used. This surface leakage

current may be responsible for the previous reports of RIED. A capsule was designed for irradiation in the High Flux Beam Reactor (HFBR) at the BNL to further study the RIED phenomenon. Polycrystalline alumina and hot-pressed silicon nitride specimens were irradiated at 80°C to 0.11 dpa under an applied voltage of 133 V/mm. These initial results did not indicate a permanent degradation in electrical properties under these conditions. A second capsule is currently being constructed to irradiate alumina specimens in the HFBR to a higher fluence and over a wide temperature range.

The radiation-induced microstructural changes in ion-irradiated oxide ceramics was examined with cross-section TEM. The relative influence of ionizing and displacive radiation was studied by systematically varying the mass and energy of the bombarding ions between 1-MeV H<sup>+</sup> and 4-MeV Zr<sup>+</sup> ions. The measured ion ranges in Al<sub>2</sub>O<sub>3</sub> were between 1 and 15% greater than the ranges calculated by the TRIM computer code, with the largest discrepancies occurring for intermediate mass ions. The implanted ions exerted a strong influence on the overall microstructural evolution of the irradiated ceramics. Numerous microstructural features such as amorphization and colloid formation were produced in the implanted ion regions but were never observed in irradiated regions that were well separated from the implanted ions. The microstructural evolution in regions well separated from the implanted ions was found to depend strongly on the mass and energy of the bombarding ion. Light ion irradiation produced a significant enhancement in point defect diffusion compared to heavy ion irradiation at the same damage rate. Similarly, irradiation with a given ion at a higher flux generally produced an increased amount of observable diffusion. In some cases such as 1-MeV proton irradiation, observable defect clusters did not form. This suppression in defect cluster formation may be due to the high amount of point defect recombination associated with ionization-enhanced diffusion that occurs during energetic light ion irradiation.

The effects of radiation on the thermal conductivity of alumina have been calculated over a wide temperature range. The phonon scattering relaxation times for various scattering mechanisms have been used in the evaluation of the thermal conductivity integral in order to determine the effect each mechanism has on the lattice thermal conductivity of alumina. Among the scattering mechanisms studied were vacancy scattering due to radiation-produced vacancies and phonon-electron scattering due to radiation-produced vacancies and phonon-electron scattering due to RIC. It was found that vacancy scattering can significantly reduce the thermal conductivity over a wide temperature range; for example, a vacancy concentration of 0.01 per atom leads to a fractional change in the thermal conductivity of about 90% at 77 K versus 39% at 400 K. It was also concluded that RIC does not lead to a significant reduction in the thermal conductivity at any temperature.

The use of silicon carbide composites for fusion reactors offers the potential benefit of a very low-activation structural material with high-temperature strength. The effects of radiation on advanced SiC fibers and SiC fiber composites are being studied. Composites fabricated with standard Nicalon™ fibers have shown substantial degradation in strength at neutron damage levels as low as 1 dpa. This degradation has been linked to fiber pullout caused by densification of the Nicalon during neutron irradiation. Such a radiation-induced densification is contrary to the behavior exhibited by silicon carbide and ceramics in general. TEM studies of neutron and carbon ion-irradiated Nicalon have associated the densification with the presence of a glassy phase in the fiber. Tensile testing and density measurements have been conducted on advanced fibers including low-oxygen Nicalon. This fiber exhibited moderate changes in mechanical properties

following 100°C HFIR irradiations and underwent a slight volumetric expansion typical for irradiated silicon carbide. This fiber should yield superior radiation resistance, and composites are now being fabricated for irradiation testing.

Reduced-activation ferritic martensitic steels have been developed with compositions tailored to reduce long-term-induced radioactivity by three to four orders of magnitude. A 9Cr-2W-0.25V-0.7Ta-0.1C (9Cr-2WVTa) steel developed at ORNL showed the least degradation of impact toughness during fast reactor irradiation in the Fast Flux Test Facility to  $\approx 13$  dpa at 365°C. Based on this observation and observations on the unirradiated properties of this type of steel, the International Energy Agency (IEA) is obtaining a large heat of this type of reduced-activation steel to be used in an international collaborative program to prove the feasibility of using a ferritic steel for fusion. The fusion programs in Japan, Europe, and the United States will participate in the IEA program.

Coated-particle fuels were analyzed for two programs: The DOE MHTGR Program and the NPR Program. Both fuels were of the coated-particle type where a uranium oxide fuel kernel is coated with a multilayered jacket of pyrocarbon and silicon carbide. In fission product release tests of irradiated fuels, the rate of release was found to be unacceptably high. Particles with high rates of fission product release were separated and tested with a variety of analytical techniques. The particles were mounted and polished to reveal the cross sections of the layers. Fission products were found to penetrate various layers differently in retentive and non-retentive particles. Barium and Pd were found to partially penetrate SiC in non-retentive particles but not in retentive particles. Cesium was found to penetrate SiC in non-retentive particles but to not go beyond even inner layers in the case of retentive particles. Cerium and La were found to penetrate inner layers in non-retentive particles but not found outside the kernel in retentive particles.

### 3.7 SUPERCONDUCTING MATERIALS — *D. M. Kroeger*

The Superconducting Materials Group is studying cuprate compounds which become superconducting at temperatures as high as 134 K. The goal of the research is to develop the technology needed to produce conductors that can carry large supercurrents in high-magnetic fields. The compounds studied can be divided into two main groups, those showing near-term promise for applications at temperatures below  $\sim 30$  K and a second group which may be developed into practical liquid-nitrogen-cooled conductors. Silver-clad conductors containing the Bi-Pb-Sr-Ca-Cu oxide "2223" phase are being developed for use at lower temperatures. Examples of the second group of compounds, which have good intrinsic current-carrying capabilities at  $\sim 80$  K, include  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (Y123),  $\text{YBa}_2\text{Cu}_4\text{O}_8$  (Y124), and the Ti-Pb-Sr-Ca-Cu 1223 phase.

The first step in producing research samples is synthesis of high-purity ceramic powders. Aerosol pyrolysis of nitrate solutions is a route which the Superconducting Materials Group has frequently employed, and this process has attracted industrial support. The advantages of the process include precise compositional control, product homogeneity, production of ultrafine particles, and suitability for large-scale production. Problems associated with  $\text{NO}_x$  and carbon contamination have been minimized, and losses of volatile metallic ions such as Pb can be controlled.

Research samples have been produced by sintering of cold-pressed pellets, liquid-phase processing, deformation and annealing of silver-clad powders, annealing powders deposited on silver tapes, and by high-pressure oxygen synthesis. Many techniques are used to characterize the samples. The superconducting properties are defined by magnetic susceptibility, transport critical current density, magnetization, and electrical resistivity measurements. The relationship between superconducting properties and microstructure is emphasized, and microstructural characterization involves XRD, SEM, TEM, microprobe analysis, AES, and optical microscopy.

Studies of moderate-to-low-temperature conductors have been conducted in cooperation with industrial researchers and the staffs at Los Alamos National Laboratory (LANL), ANL, and the University of Wisconsin as a participant in the Wire Development Group led by American Superconductor Corporation. The emphasis of the studies has been on developing silver powder-in-tube and tape conductors containing the Bi(Pb) 2223 phase. Precursor powders generated by aerosol pyrolysis have been utilized because they have no large or hard agglomerates and have shown the ability to react more rapidly to form the Bi-2223 phase than powders produced by solid state reaction. The rate of conversion of a precursor powder to Bi(Pb) 2223 seems to depend critically on the crystalline phase content of the powder, specifically the presence or absence of a yet unidentified metastable phase. For a particular silver-clad precursor powder having overall Bi(Pb) 2223 composition and containing primarily 2201, Ca<sub>2</sub>PbO<sub>4</sub>, CuO, and the metastable phase, sintering for 6 h at 810°C and 7.5% O<sub>2</sub>/Ar leads to the development of a sharp 110-K resistive transition. For the same precursor powder that has been given a 650°C pretreatment to remove only the metastable phase, no such development is observed for the same sintering treatment in the silver-clad geometry. Complimentary XRD shows crystalline Bi(Pb) 2223 to be present in only the sintered deposit showing the sharp 110-K transition.

Deposits containing the Tl 1223 compound (Tl<sub>x</sub>Ba<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>y</sub>, 0.7 < x < 0.95) have shown promise for liquid-nitrogen-cooled applications. These materials have been studied extensively during this reporting period. Researchers at GE Research and Development Center, Schenectady, New York, have developed a process for preparing high-critical-current density J<sub>C</sub> deposits of Tl 1223 on yttria-stabilized zirconia substrates. Tl-free precursor deposits are prepared by spray pyrolysis and subsequently reacted with Tl<sub>2</sub>O vapor to form 3-μm-thick Tl-1223 deposits having J<sub>C</sub> near 10<sup>5</sup> A/cm<sup>2</sup> at 77 K in zero field. Studies of these conductors have been conducted in conjunction with GE and have emphasized two areas: understanding the current-carrying capability and determining the effects of processing variables on the properties of the deposits.

The deposits are polycrystalline and have excellent c-axis alignment. Electron back-scatter diffraction patterns (EBSPs) obtained from widely separated regions on the deposits indicate a lack of in-plane texture. Nevertheless, the deposits show little evidence of weak link behavior. At 77 K, the J<sub>C</sub> vs H curve for H||c has a plateau extending to 1 T. Microstructural analysis and additional J<sub>C</sub> measurements have helped to explain these observations and led to a new percolative conduction model. Results of basal plane texture determinations from XRD azimuthal scans were found to depend on the size of the X-ray beam, indicating the presence of local texture in deposits which, overall, appear to be nearly isotropic. This local texture indicates the presence of colonies of grains with similar orientations. These results have been complemented by EBSP measurements, and these results are similar to the XRD data. The grain colonies can be up to ~1 mm in size, and within the colonies, the a-axis orientations are distributed over an angular range of 10 to 15°. Current conduction within the colonies

## 4. CERAMIC SCIENCE AND TECHNOLOGY

*R. L. Beatty*

Research in the Ceramic Science and Technology Section focuses on (1) the development of strong, tough CMCs; (2) the characterization of carbon and graphite materials for nuclear, space power, and other applications; (3) the understanding and development of supporting processing technologies; and (4) the evaluation and development of materials for advanced thermal insulation systems. Consisting of approximately 35 professional and 20 technical support personnel, the section is organized into five groups: Carbon Materials Technology, Ceramic Processing, Ceramic Surface Systems, Structural Ceramics, and Materials Thermal Analyses (formerly Building Materials).

### 4.1 CARBON MATERIALS TECHNOLOGY — *T. D. Burchell, E. L. Fuller, O. C. Kopp, R. A. Lowden, J. M. Robbins, G. R. Romanoski, L. L. Snead, J. P. Strizak, and C. E. Weaver*

#### 4.1.1 Fusion Energy Carbon Materials

Research is being conducted in the area of the radiation performance of graphites and carbon-carbon (C/C) composites for fusion plasma-facing component (PFC) applications. Graphite and C/C composites are used in every major fusion tokamak reactor in the world, principally due to the low atomic number of carbon (which reduces plasma poisoning) and their potential high strength and high thermal conductivity.

The proposed ITER raises new problems regarding the use of these materials as PFCs. Principally, this high-power reactor will produce the first significant amount of neutrons from a fusion machine. Therefore, for the first time, the radiation damage in PFCs is of primary concern. Research has focused on the dimensional stability, strength, and thermal conductivity of high-quality C/C and high-thermal-conductivity graphites. It has been shown that, in the radiation regime of interest, the strength of candidate C/C composites increases marginally, and the dimensional stability is tolerable. The thermal conductivity has been measured in a series of HFIR hydraulic tube experiments from 0.01 to 1.0 dpa at 100°C resulting in a high reduction in thermal conductivity. Specifically, for the highest quality composite studied, the thermal conductivity was reduced from 650 W/m-K (approximately twice copper) to a saturated value about 60 W/m-K. Saturation occurs at approximately 0.25 dpa.

Annealing studies were carried out to yield a conservative estimate of the effect of elevated-temperature irradiation on thermal conductivity. Samples were heat treated for 1 h in the range of 300 to 1200°C. A significant recovery of thermal conductivity was seen. For example, a one-dimensional C/C had decreased to 30% of its original thermal conductivity by 0.01 dpa, but recovered to 70% original following the 1200°C anneal. These results indicate that C/C materials can be considered for applications such as the ITER, though higher temperature operation would be necessary than the previous design had specified.

Studies are also being planned and carried out for alternate low-atomic-numbered PFC materials. The effects of processing conditions, heat treatment, and irradiation on thermophysical properties are being collaboratively studied with Sandia National Laboratories and LANL. This work is being focused on plasma-sprayed material which typically has a thermal conductivity half that of conventionally processed beryllium.

As the ITER Program combines the research programs from the United States, European Community, Japan, and Russia, an effort has been made to coordinate research among the ITER partners for the plasma-facing materials (PFM) irradiation program. Specific collaborations are under way in the areas of thermal conductivity of high-quality Russian graphites and the design of the divertor with JAERI in Japan.

#### **4.1.2 Commercial MHTGR Program**

Our activities for the MHTGR Program centered around our participation in the USDOE-JAERI collaboration on gas-cooled reactors (GCRs) as detailed in Annex 3, "Graphite," of the agreement. Two specific technology development areas were active during this reporting period: (1) fracture toughness testing of IG-110 graphite, a grade manufactured by Toyo Tanso of Japan for the Japanese nuclear program; and (2) the continued development of a fracture model for graphites. In the latter area, we concentrated on the incorporation of extensive microstructural data for grade H-451 in the model. These data were developed using a PC-based image analysis system coupled to a Nikon-FXA microphoto optical microscope. Pore size and shape distributions were obtained for numerous specimens taken from a production billet of H-451. The mean pore size, or pore breadth, defined as the distance between the left-most and right-most pixels in the digitized object (pore), was found to be 42  $\mu\text{m}$ . The fracture model, using these data and a particle critical stress-intensity factor of  $0.285 \text{ MPa}\cdot\text{m}^{1/2}$ , was found to give excellent agreement with the experimentally determined tensile strength probability distribution for grade H-451 graphite.

#### **4.1.3 New Production MHTGR**

##### **4.1.3.1 Thermal Properties**

Thermal physical properties data were obtained to validate graphite moderator thermal physical design codes which are used to predict peak temperatures, thermal gradients, and thermal stresses in the core of an MHTGR. Specimens of grade H-451 graphite were prepared from a production class I billet (R58-357) and tested to determine: (1) specific heat, (2) room-temperature thermal conductivity, and (3) the temperature dependence of thermal conductivity. Moreover, room-temperature thermal conductivity data were acquired from specimens cut within one of three orientations (axial, tangential, or radial) to assess the extent of anisotropy in the graphite billet.

The specific heat of H-451 was determined using the thermal flash technique. The temperature dependence of H-451 graphite specific heat was found to be well represented by National Institute for Standards and Technology (NIST) standard specific heat data for graphite. Room-temperature thermal conductivity data for H-451 were found to be normally distributed with mean values of 150, 137, and 135  $\text{W}/\text{m}\cdot\text{K}$  in the axial,

radial, and tangential directions, respectively. The anisotropy of the thermal conductivity was attributed to the preferred textural orientation known to exist in extruded graphites.

#### 4.1.3.2 Mechanical Properties

Over 1700 room-temperature tensile tests were conducted on specimens from 6 billets of H-451 graphite in order to evaluate the effects of specimen orientation and spatial location within the billet, and variabilities from billet to billet, class to class, and lot to lot. A concentrated effort was made to complete the tensile testing by the closeout of the NP-MHTGR Program. Detailed analyses of the data could not be completed in time, but the initial observations for one of the billets were interesting. A strength gradient was observed through the cross section of the billet. Strength decreased from the billet perimeter on toward the central axis of the billet. Also, the data suggested that radial and axial orientations of the test specimens with respect to the billet axis may have nearly the same tensile strength. Tangential specimens, on the other hand, appeared to be as strong as the axial specimens at the perimeter of the billet, but the stress gradient was not as steep, resulting in generally higher tensile strengths for the tangential orientation in the central portion of the billet. The test specimens and raw data for the six billets have been stored so analysis could be resumed at a later date.

In the fatigue data testing program that was planned for the NP-MHTGR Program, fatigue life over the range of  $10^2$  to  $10^5$  cycles would be determined as a function of stress amplitude under constant amplitude cyclic loading at room temperature. The effect of stress ratio, R (minimum stress/maximum stress during a fatigue cycle) on cyclic life would be determined over the range R = -1 to R = +1 with the maximum stress ranging from 65 to 100% of the mean ultimate tensile strength. Further, since H-451 graphite billets are generally inhomogeneous and exhibit significant variations in properties, the test matrices were designed to expose any gradient in fatigue properties. The effects of specimen orientation and spatial location within the billet, as well as billet-to-billet and lot-to-lot variations, would be determined.

Nearly 1600 fatigue test specimens were machined along with the tensile specimens that were taken from the 6 billets of H-451 graphite. This was done purposefully since the popular relationships (modified Goodman, Berber, Sonderberg, and Morrow) for determining the effect of mean tensile stress on fatigue life are expressed in terms of the yield and ultimate tensile strengths of the material. No fatigue testing was conducted during the program closeout period since a concentrated effort was made to complete the tensile testing task. The fatigue specimens have been stored for possible future use.

#### 4.1.3.3 Fracture Mechanics

Although design of the MHTGR core is based on a maximum principal stress failure criterion, additional fracture mechanics analysis is needed to assess defect tolerance and inspection requirements. The chevron-notched short-rod test method per ASTM E1304-89 was used to provide fracture toughness data to address material variables. The scope of fracture toughness characterization of core graphite completed to date includes a within-billet evaluation of specimen orientation and spatial location as well as a billet-to-billet comparison of four billets representing the four minimum-strength classifications of H-451 graphite.

No significant trend in fracture toughness with respect to strength classification or axial position was observed. The axial crack plane orientation, i.e., transverse to the graphite billet extrusion direction, exhibited the highest toughness. Higher toughness values were also observed for near-surface regions. It can be concluded that the fracture toughness of H-451 graphite can be represented by a single value:  $K_{\text{QV}}(\text{avg}) = 1.48 \text{ MPa}\cdot\text{m}^{1/2}$  and  $K_{\text{QV}}(-3 \text{ sigma}) = 1.26 \text{ MPa}\cdot\text{m}^{1/2}$ . Effects of spatial location and crack plane orientation are bounded by the -3 sigma limit.

#### **4.1.3.4 Oxidation Studies**

Detailed examination of energetics, intrinsic kinetics, and mechanisms of the interaction of water vapor and oxygen with nuclear-grade graphites has been completed. Final reports have been issued and published in open literature.

#### **4.1.3.5 Coke Source Examination**

PIE of the H-451 specimens from capsules HTN-2 and -3 was finished. HTN-2, a 600°C irradiation experiment, completed 16 cycles, and HTN-3, a 900°C irradiation experiment, completed 9 cycles in the HFIR. PIE of the specimens from HTN-2 and -3 included measurement of dimensional changes, thermal conductivity, elastic constants, and strength.

#### **4.1.3.6 Alternate Vendors Program**

Two irradiation experiments were planned under the NP-MHTGR Program to evaluate graphites from several alternate vendors. The specimens for these two irradiation capsules were machined and underwent full pre-irradiation examination during this reporting period.

#### **4.1.3.7 C/C Composite Control Rods**

A technical plan describing a technology program for high-temperature control rods manufactured from C/C composites was published in January 1992. The plan presented a development strategy along with arguments for the selected materials, carbon fibers, architectures, and processing details. A prototype control rod design based upon General Atomic's (GA's) conceptual drawings was presented, and the required program of irradiation, mechanical, thermal-physical, and oxidation testing was briefly outlined.

In February 1992, a purchase order was placed with Fiber Materials, Inc., for the various C/C materials required for the initial work covered in the technical plan. These long-lead-time items were received during the closeout period for the NP-MHTGR Program. Two demonstration prototype control rod assemblies were machined, and planning had begun for mechanical, thermophysical, and oxidation tests on the remaining materials.

#### **4.1.3.8 NPR-MHTGR Fuel Compact Thermal Conductivity**

Thermal conductivity data are needed to validate core design methods for the calculation of fuel compact temperature, thermal gradients, and thermal stress. Accurate prediction

of compact temperature is important because fission product transport phenomena are extremely temperature sensitive. Prior to making measurements of the thermal properties of fuel compacts, procedures were developed for handling and cutting the fuel sticks. Data were taken for the thermal diffusivity and specific heat of compacts loaded with 10 or 20 vol % fuel particles and were the first obtained for the 8-layer TRISO fuel-containing NP-MHTGR fuel compacts.

#### **4.1.4 Improved Graphite Impact Shells (GIS)**

The GPHS provides power for space missions by transmitting the heat of  $^{238}\text{Pu}$  decay to thermoelectric elements. Because of the possibility of an aborted mission, the heat source must be designed and constructed to survive both reentry heating and earth impact. Cylindrical GIS contain the iridium alloy-clad fuel pellets and serve a principal role in impact protection. The present GIS configuration is machined from an orthogonal-weave C/C composite and is susceptible to longitudinal fracture during impact. Cylindrical architecture C/C composites are being evaluated as a potential improvement to GIS impact performance. Characterization includes measurement of physical, thermophysical, and mechanical properties.

A gas gun test facility has been prepared for impact testing of candidate GIS materials to evaluate the effect of architecture and density on energy absorption. Impact tests will be conducted at room temperature using one nickel-clad hafnia fuel simulant in a half-length GIS specimen. Each GIS specimen plus fuel simulant will be fired at 55 m/s (reentry terminal velocity) into an instrumented steel target that measures the force versus time pulse during deceleration of the projectile. A GIS material that yields a lower deceleration rate and lower peak force will provide greater protection to the  $\text{PuO}_2$  fuel under simulated impact conditions.

#### **4.1.5 CBCF Insulator Material**

CBCF thermal insulators fabricated in earlier production campaigns for the RTG Program are currently flying in the NASA Galileo and Ulysses deep space probes. Since completion of the production schedule for the Cassini mission, the emphasis has been on more complete characterization and improvement of CBCF insulating material. Additional CBCF billets and plates have been processed using standard procedures under which material was produced for fabrication of flight-quality parts for the Cassini mission. Density mapping and compressive strength studies have been conducted to evaluate material uniformity. A similar study of the variation in thermal conductivity in billets and plates is under way. In the interest of reducing high-temperature thermal conductivity of CBCF material, impregnation studies have been initiated to evaluate the effect of an addition of carbonaceous filler material on the radiation heat conduction at high temperatures. It has been postulated that such additions would lead to a reduction of radiation heat transfer, which is a major contributor to total thermal conductivity at high temperatures.

#### **4.1.6 Chemistry and Structure of Coals**

Efficient uses of coal for nonfuel purposes (pitches, cokes, molecular sieves, etc.) require detailed knowledge of the various feedstocks from mines across the country. Comprehensive studies have been made to characterize the Argonne Premium Coal Samples to serve as a reference database for future studies on field samples of coals and/or processed materials. Diffuse reflectance infrared spectroscopic analysis is used to characterize the general chemical nature of the samples. Moreover, TGA, supplemented with mass spectrometric gas analysis, is used to evaluate the chemical reactivity and pyrolysis process. Techniques have been developed to define the relative contributions of chemical reactions occurring during thermal decomposition of the various coals, and the reaction products. Morphological properties are obtained by microscopy and porosity analyses.

#### **4.1.7 Microstructure and Reactivity of Automobile Emission Catalysts**

Pore-size analyses of commercial and prototype catalysts have been used to evaluate the regions where hydrocarbon oxidation occurs (catalyzed by platinum/palladium on the surfaces of powdered alumina). Aged catalysts have been studied to aid in the evaluation of the various factors related to catalyst deactivation ("poisoning"). The results define the chemical and temperature limits for ensuring that the catalysts will achieve the 50,000- to 100,000-mile lifetime imposed by current and future regulations. This program is directly under the auspices of the GM-AC Rochester CRADA.

#### **4.1.8 Carbon Fiber Composite Molecular Sieves (CFCMS)**

CBCF technology has been used to fabricate material for evaluation as molecular sieves in gas adsorption applications. The material has been fabricated in cylindrical-billet and flat-plate geometries and carbonized at 650°C prior to activation by researchers at the University of Kentucky and subsequent evaluation as molecular sieve material. Processing parameters have been varied to yield plate thicknesses of approximately 1.5 to 5 cm. Test results to date have been very encouraging and have resulted in the filing of a patent application identifying the material as a novel material with a potential for wide industrial application.

Characterization has been performed on the as-manufactured material, including SEM studies and fiber length distribution determinations. The activated CFCMS materials have been shown to possess high Brunauer-Emmett-Teller surface areas ( $>1600\text{ m}^2/\text{g}$ ) and narrow micropore distributions centered at  $\sim 6\text{\AA}$  pore width. Preliminary evaluations, performed in collaboration with the University of Kentucky, have demonstrated the efficacy of the material for separating  $\text{CO}_2$  from  $\text{CH}_4$ .

Additional studies have been carried out to evaluate the optimum procedures for carbonization and activation of CFCMS. Detailed studies of the micropore structures of these materials are under way to aid in tailoring molecular sieves for various applications (hydrogen purification, hydrocarbon traps, natural gas storage, etc.).

#### **4.2 CERAMIC PROCESSING — *R. J. Lauf, M. A. Janney, J. O. Kiggans, H. D. Kimrey, A. J. Moorhead, S. D. Nunn, O. O. Omateete, and T. N. Tiegs***

Group R&D activities include ceramic forming by gelcasting, MW processing, in situ-toughened silicon nitride materials development, materials characterization, and sensor and electronic materials development. These activities are supported by the CTAHE Program, Office of Transportation Systems; the AIC and Advanced Industrial Heat Exchanger Programs, Office of Industrial Technology (OIT); the Fossil Energy AR&TD Materials Program, Office of Technical Coordination; the RTG Program, Office of Special Applications; and the Office of Energy Research, USDOE.

Ceramic gelcasting is being developed as a net-shape-forming method that may offer both manufacturing advantages and improved reliability of products when compared with forming by slip casting or injection molding. The gelcasting process involves the dispersion of commercial ceramic powders in an aqueous organic monomer solution. After casting the ceramic slurry in a mold, gelling is accomplished by thermal polymerization of the monomer. Recent developments have included the demonstration of highly repeatable processing using low-toxicity chemical systems, enhanced ceramic green body strength when compared to dry pressed or slip-cast ceramics, and improved green machining characteristics. Important advances have been made in the gelcasting of silicon metal for cost-effective reaction-bonded silicon nitride (RBSN) and sintered reaction-bonded silicon nitride (SRBSN).

The gelcasting process has generated considerable interest in the commercial ceramic processing industry. During the past year, we completed our CRADA with AlliedSignal Ceramic Components Company for gelcasting of silicon nitride turbine engine components, amended our CRADA with Coors Technical Ceramics Company to evaluate gelcasting of an alumina component used in the semiconductor processing industry, and finalized a CRADA contract with Ceramic Magnetics to study the gelcasting of soft ferrite materials. In addition, we are in various stages of negotiations with three other companies to sign CRADAs.

One staff member from our group began a 3-month onsite assignment at AlliedSignal in Torrance, California, to assist them in the industrial scale-up of our gelcasting process. Not only does this assignment demonstrate the value of the national laboratory to American industrial competitiveness, but it also helps our staff gain a better understanding of the manufacturing environment.

In work for the AIC Materials Program, it was demonstrated that good agreement between temperature measurement devices is possible inside an MW furnace. Simultaneous measurements of temperature in ceramic samples (alumina, zirconia, and silicon carbide) were made using a thermocouple (TC), an optical fiber probe, and a 2-color infrared pyrometer. Agreement among the three types of measurement ( $\pm 20^\circ\text{C}$ ) was well within the range typically achieved for conventional furnaces. Careful attention was paid to the proper shielding of temperature probes such as TCs and light pipes to prevent inaccurate measurements caused by arcing and "shine-through," respectively.

Work for the FEMP was initiated (August 1993) to develop an MW-heated diesel particulate trap and burner. This work is jointly funded by the Heavy Duty Transport Program and includes a CRADA with Cummins Engine Company. Preliminary results have demonstrated that filter materials infiltrated with silicon carbide by chemical vapor infiltration (CVI) can be heated effectively using MW energy.

Two additional CRADAs on MW work were conducted last year under ER Programs. One is with AVX Tantalum Corporation to evaluate MW sintering of tantalum capacitors, and the other is with Microwave Laboratories, Incorporated (MLI), to develop a variable-frequency furnace.

MLI has licensed the variable-frequency furnace and has developed a broad line of products ranging from a 200-W benchtop unit to large, multikilowatt systems. They have demonstrated very exciting results in materials processing applications.

Also, working with AVX Tantalum, we have observed several potentially exciting phenomena in MW-processed Ta capacitor anodes. These unique products are the subject of U.S. patent 5,184,286, entitled "Process for Manufacturing Tantalum Capacitors," and are currently being evaluated at AVX Tantalum.

The primary MW work for the CTAHE Program has been the study of MW heating as an alternative for producing cost-effective SRBSN materials. SRBSN raw materials costs are less than about one-fourth those for high-purity silicon nitride materials, which improves the cost competitiveness of these materials with metal parts. Conventional processing of SRBSN requires long nitridation times and a two-step firing process. MW heating reduces the reaction times and is performed in a one-step process, thereby simplifying the operation and making it more cost effective. The flexural strength of the MW-SRBSN is equivalent to the strength of some materials made from higher-cost powders. Thus, these materials may be appropriate for a number of applications at temperatures up to approximately 1000°C.

While, MW processing has generated considerable interest in the scientific community, it has been generally limited to laboratory-scale experiments of small quantities of parts. To produce cost-effective ceramics for advanced heat engine applications, fabrication processes need to be appropriate for large-scale production of parts. An initial series of experiments was performed to investigate the scale-up of the SRBSN process using MW heating. Successful scaling-up of the process was found to be dependent on two important factors, heating uniformity and temperature control. By using normal heating rates ( $\leq 5^{\circ}\text{C}/\text{min}$ ) and heat distributors of dense BN around the parts, these factors can be controlled. Recent tests have demonstrated that both multiple parts and also complex-shaped SRBSN parts can be processed by MW heating. Individual parts as large as 0.5 kg have been successfully nitrided. More importantly, the mechanical properties of the materials were equivalent or superior to those obtained by conventional heating.

Two CRADAs in the area of MW processing of silicon nitride were completed. The first CRADA was with Norton Advanced Ceramics Company and involved MW annealing of dense silicon nitride ( $\text{Si}_3\text{N}_4$ -4%  $\text{Y}_2\text{O}_3$ ) with a range of  $\alpha$ -phase contents. MW annealing showed increases in fracture toughness and high-temperature fast-fracture strength with annealing between 1200 to 1650°C. High-temperature dynamic fatigue measurements revealed an increased stress to failure for specimens MW annealed between 1400 to 1550°C for periods  $>5$  h. It was speculated that silicon nitrides with different sintering additives would require different conditions for optimum crystallization.

Norton Advanced Ceramics Company was also involved in the second CRADA where RBSN was fabricated in the MW. The results were directly compared to materials fabricated

by conventional heating. These tests showed that the MW-processed materials exhibited higher strengths in most cases and higher toughness in all cases.

Other work on silicon nitride under the CTAHE Program is directed toward in situ-toughened materials. Toughening is achieved by the development of elongated grain microstructures that have controlled intergranular phases to promote crack-bridging mechanisms. Gas-pressure sintering (GPS) is one technique used to grow elongated grain structures and obtain high-toughness silicon nitrides with refractory GB phases. Numerous studies have shown that the silicon nitride powder used in fabrication is an extremely important variable in the microstructural development and final properties of the materials. At ORNL, several  $\text{Si}_3\text{N}_4$  powders were examined having different fabrication methods, surface areas, oxygen contents, and impurity levels. During early stage densification, all powders showed similar shrinkage, and the diimide-derived powder exhibited delayed  $\alpha/\beta$  transformation compared to the other powders. Powders derived from diimide and gas-phase processes achieved the highest final densities. Improved densification was observed by increasing the oxygen content, and this also resulted in high toughness for some materials with rare-earth apatite additives. However, the increased oxygen resulted in reduced high-temperature strength.

During densification of  $\text{Si}_3\text{N}_4$  by GPS, gradients in density, composition, and properties develop from the exposed surfaces to the interior bulk material. Minimization of these gradients is important for reducing the necessity for machining. It is well established that machining represents a major portion of the cost of silicon nitride parts, and efforts are under way to reduce its impact on the economics of these materials for engine applications. Consequently, it is desirable to minimize the reaction layers and thereby minimize the machining necessary. In studies at ORNL, formation of the gradients was found to be dependent on the process conditions and near-surface surroundings used during sintering. It was determined that in a graphite furnace environment, the gradients are minimized by using closed RBSN crucibles without excess powder packing at low  $\text{N}_2$  overpressures. Along with the compositional gradients, mechanical property differences also exist between the as-sintered surfaces and the bulk materials. For example, the strengths of as-sintered surfaces of  $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3\text{-Al}_2\text{O}_3$  were observed to be reduced by more than 30% compared to the bulk material.

Work in the Ceramic Processing Group (CPG) is also directed at immobilization of high-sodium nitrate content/LLW. This type of material represents a large portion of the radioactive wastes at sites such as Hanford and SRL. Researchers in the CTD have developed a process for destruction of sodium nitrate referred to as the Nitrate-to-Ammonia and Ceramic (NAC) Process. In the process, metallic aluminum is reacted at high pH to produce sodium aluminate and aluminum trihydrate. The CPG was involved in final steps in the NAC process to heat-treat the reaction product to remove chemically bound water and react the Na, Al, and Si components together to obtain a dense material that is resistant to environmental degradation. High-density, leach-resistant materials were made by drying, calcining, pressing, and sintering techniques. Work was also initiated to investigate the formation of a vitreous product from the NAC process.

Work continued on the CRADA with the Electric Power Research Institute (EPRI) on the development of deposition techniques and substrates for thin-film polycrystalline silicon photovoltaic cells. The program is a joint effort with researchers in the Solid State Division. One of the most important drivers for the effort is low-cost materials and manufacturing techniques. During this period, a number of silicon wettability tests, both in high-purity argon atmospheres and vacuum, were conducted on both

commercially supplied substrate materials as well as those being developed in-house. To date, the most promising results have been achieved on an experimental composite substrate fabricated into thin sheets by gelcasting. The composite is strong and readily wetted by molten silicon. We think that by varying the proportions of reinforcement and matrix, we will be able to make the substrate conductive or nonconductive as required for a particular cell design.

We joined a team whose mission is to develop the design for a new shipping cask for the californium ( $Cf^{252}$ ) radioisotope sources that are produced in our HFIR. The present shipping casks are large spheres, 5 to 6 ft diam, containing concrete for the neutron and gamma ray shielding. Unfortunately, the certifications on these casks will expire in 1996, so we are in the initial phases of designing a replacement cask that will meet today's much more stringent government regulations. Presently, a very wide variety of materials, from reinforced concrete to yttrium and hafnium hydrides, is being considered.

Work was initiated on a CRADA with GM to develop engine systems electronics suitable for high-temperature and/or high-voltage under-hood applications. To date, our efforts have centered on the analysis, through nondestructive and destructive means, of spark plug insulators or spark plugs that have failed either during quality assurance tests or engine tests, respectively.

Work continues on adapting CBCF thermal insulation to other applications. U.S. patent 5,243,464, "Damage-Tolerant Light-Absorbing Material," and a U.S. patent application, "Improved Blackbody Material," represent the first products of this initiative. The light-absorbing material was a finalist in the Commercial Technology Achievement Award competition sponsored by *Laser Focus World* magazine.

Development work continues on a simple, resistive hydrogen sensor. A thick-film sensor is being developed in cooperation with DuPont, for which two patent applications have been filed.

A new CRADA project on the development of improved zinc oxide surge arrestors, supported by ER Programs, is being carried out with Ohio Brass, in collaboration with the Solid State Division.

Work continues with Saphikon, Inc., under a High-Temperature Superconductor Pilot Center (HTSC) Agreement to produce continuous ribbons of rare-earth perovskites suitable as substrates for oxide superconductors. We have successfully demonstrated epitaxial deposition of YBCO superconductor material onto the ribbons.

#### **4.3 CERAMIC SURFACE SYSTEMS — *T. M. Besmann, P. J. Blau, W. Y. Lee, J. C. McLaughlin, D. P. Stinton, and C. S. Yust***

A research effort sponsored by the Division of Advanced Energy Projects, Office of Basic Energy Sciences, has been continuing to create novel, self-lubricating materials comprised of solid lubricants embedded in a hard ceramic matrix CVD. TiN, which was selected as a hard coating matrix, is prepared using a unique organometallic precursor,  $Ti\{N(CH_3)_2\}_4$ , and  $NH_3$  at 300 to 400°C. The solid lubricant,  $MoS_2$ , is deposited from  $MoF_6$  and  $H_2S$  in the temperature range of 300 to 500°C. The effects of processing

conditions on the microstructure and adherence of  $\text{MoS}_2$  have been studied. Experiments are being performed to assess the possibility of incorporating  $\text{MoS}_2$ ,  $\text{TiS}_2$ , and  $\text{TiO}_2$  as dispersed solid lubricant phases in the TiN matrix. Friction studies suggest that fullerenes ( $\text{C}_{60}$ ), which were initially considered as a potential solid lubricant material, are not superior to ordinary graphite in lubrication.

$\text{Si}_3\text{N}_4$  and SiC are susceptible to hot corrosion induced by molten salts such as  $\text{Na}_2\text{SO}_4$  under certain combustion environments. In a research program sponsored by the Ceramic Technology Project (CTP),  $\text{Ta}_2\text{O}_5$  is being considered as a potential coating material for the protection of the Si-based materials from hot corrosion. A CVD process has been successfully developed and optimized to produce crystalline, void-free  $\text{Ta}_2\text{O}_5$  coatings. The effects of process parameters such as temperature and reagent concentration on coating thickness and morphology were studied. The effectiveness of the  $\text{Ta}_2\text{O}_5$  coating in a variety of laboratory and burner-rig corrosion tests is being assessed.

An EE Program is investigating the introduction of ceramic materials into the hot-gas path of stationary gas turbines. Such a system has the potential to increase combustion temperatures and significantly improve the operating efficiencies. However, very stiff ceramic materials with low thermal expansions must be joined to more ductile metals with high coefficients of thermal expansion (CTE). During thermal cycling, the difference in material properties creates residual and thermal stresses sufficient to damage ceramic materials or spall ceramic coatings away from metallic substrates. A development effort was initiated to design, fabricate, and test functionally graded materials (FGMs) as abradable seal coatings for turbine shrouds and TBCs for turbine blades that overcome these problems. A contract was placed with Lehigh University to develop a model to determine the stresses present in FGMs of different compositions and thicknesses. FGMs were prepared by plasma spraying and will be characterized to verify Lehigh's model. FGMs will be designed and fabricated from optimum material combinations identified by the model.

The Air Force Wright Patterson Laboratories has sponsored an effort to model CVI for the fabrication of CFCCs. A 3-D process model using a steady state finite volume technique has been developed together with Georgia Tech Research Institute. An additional task requested by Wright Laboratories was to design and construct a scale-up forced CVI unit to demonstrate the fabrication of turbine rotor subelement. The scale-up infiltration system was designed, procured, and operated during this period. The program was successfully completed with the production of high-quality disks densified in a remarkable 40 h.

An AIC project to develop  $\text{TiB}_2$ -matrix composite cathodes for the reduction of aluminum continued this period. Efforts focussed on the fabrication of composites of fairly complex geometry for testing by the Alcoa Company for use as Hall-Heroult cell cathodes. Initial results are encouraging, although the material's surface did not wet uniformly.

Several efforts are ongoing under a Fossil Energy AR&TD Materials Program. Fiber-reinforced thick-walled tubular composites of different fiber architectures were fabricated using the forced CVI process. Unfortunately, the process required a long time (150 to 200 h) to thoroughly densify the fibrous preforms. The process was improved during the

year so that composite tubes could be densified in only 40 h. Increased cool-face temperatures and improved seals for the reactant gases greatly accelerated the SiC deposition rate.

Fiber-reinforced SiC-matrix composites often fail because of degradation of the interfacial carbon coatings. Mechanical stresses cause cracks to penetrate the SiC matrix and permit the ingress of oxygen that degrades the carbon interlayer. Thermomechanical modeling suggests that oxidation-resistant coatings with low elastic moduli and low CTE should be ideal for this application. Great progress was made during the year to produce coatings from materials with low moduli and low CTE. A CVD process was developed to deposit porous SiC coatings, and a particulate deposition process was developed to deposit mullite and sodium-zirconium-phosphate coatings. Evaluation of composites with oxidation-resistant interfacial coatings is currently under way.

Full-scale, fiber-reinforced candle filters (60 mm diam and 1.5 m long) have been fabricated in a collaborative effort with the 3M Company. Filters tested at the Westinghouse Science and Technology Center exhibited excellent filter efficiency (<5 ppm), cleanability, and thermal shock resistance. These very promising results have interested 3M in licensing the ORNL filter patent.

Friction and wear research in the Ceramic Surface Systems Group is supported by several DOE programs: Office of Transportation Materials, Tribology Project; Advanced Energy Programs; and DPs. Primary emphasis is on understanding the influence of materials structure and thermomechanical properties on wear and friction behavior. During 1993, friction and wear analyses were performed on in situ-reinforced silicon nitride materials to establish the contact pressure and velocity boundaries for transitions from acceptable sliding behavior to unfavorable friction and wear regimes. That involved CRADAs with two U.S. companies (AlliedSignal Aerospace, Inc., and Dow Chemical Company). In another project, the potential benefits of carbon-graphite materials as pistons were evaluated. The effort was performed on specimen materials supplied by two graphite producers (Poco Graphite and U.S. Graphite). In other work, a group staff member has been chairing an ASTM task group to develop a reciprocating wear test standard. Another CRADA project with an automotive company involved developing a special engine wear simulator. The tribology staff also helps the DOE/ORNL CTP in the design and testing of repetitive impact wear of emerging materials for ceramic engine valves.

#### **4.4 STRUCTURAL CERAMICS — *P. F. Becher, K. B. Alexander, A. Bleier, C.-H. Hsueh, H. T. Lin, and S. L. Hwang***

Advances in the technology and commercially important areas of energy production and conversion, transportation, chemical processing, manufacturing, electronics, and communications are predicated upon the development of materials with greatly improved performance. In a great number of these areas, brittle materials, e.g., ceramics, will be employed in devices (e.g., optical, electronic) with greater performance demands and in applications involving severe structural loads. Many of these components will be exposed to tensile stresses, and possibly quite high temperatures, which will require that the materials be designed to achieve greater fracture resistance and resistance to

deformation at elevated temperatures, as well as perform the main component function. This project combines theoretical modeling with characterization of toughening, deformation, and material degradation processes in ceramics and composites. Studies of materials processing are employed to both develop the microstructural features dictated by the mechanical properties models and understand the mechanisms involved in the generation of such microstructures. The underlying objective is to understand how to develop advanced ceramics with greater mechanical performance capabilities and adapt this understanding to the needs of the DOE programs. Current projects involve several interactive tasks supported by the Materials Sciences Program, Office of BES; the CTAHE, Office of Transportation Technologies; the AIM, OIT; and the CFCC Project, OIT. The research also contributed to the transfer of technology to industry through licensing agreements in the area of whisker-reinforced ceramics (WRC) and in transformation-toughened zirconia alloys.

#### **4.4.1 Fiber-Reinforced Composites**

Theoretical studies of interface properties have a strong collaborative component with efforts to characterize fiber-matrix interfaces in CFCCs now under development. In this way, techniques such as fiber pullout, pushout, push-in, and slice compression tests have been developed. Stress analyses have also been performed to determine the interfacial properties from experimental results. However, the analysis of the stress transfer between the fiber and the matrix is very complex, and past studies have been forced to make a number of simplifications. For example, the radial expansion/contraction (i.e., Poisson's effects) of the fiber was often ignored. Until recently, researchers were forced to employ an iterative process in attempts to derive interfacial properties from experimental results. By considering all the essential factors, we have succeeded in developing rigorous analyses that are now used for computation of interfacial properties. Methodologies for each testing technique have also been established to evaluate interfacial properties (e.g., bonding, residual clamping stress, and frictional coefficient) sequentially and uniquely. The analytical solutions are routinely employed for characterizing composite interfacial properties and are used extensively by industrial and university researchers.

A new task was initiated to evaluate the mechanical behavior of fiber-reinforced ceramics at elevated temperatures. The creep rates of an SiC-infiltrated plain-weave cloth (carbon-coated, 0.3- $\mu$ m-thick, Nicalon fiber bundles) composite were found to be quite low ( $< 5 \times 10^{-9}/s$ ) at temperatures between 1100 and 1400°C and applied stresses of 50 to 100 MPa in air. Creep resistance appears to be maintained through the creep-resistant SiC matrix. However, after creep exposures, the composite exhibits a fourfold increase in the interfacial debonding stress at room temperature that accounts for its completely brittle fracture behavior. These studies are being expanded to broaden the temperature and stress range of creep conditions and to evaluate time-to-failure response at elevated temperatures.

#### **4.4.2 Silicon Nitride and Related WRC**

Studies of silicon nitrides reinforced by elongated grains indicate that toughening due to formation of elongated silicon nitride grains should scale with the grain diameter and volume content of the larger elongated grains. Earlier, this response was predicted based on crack-bridging models developed for WRC. In silicon nitride and other ceramics, optimization of this self-reinforcement toughening response requires

knowledge of (1) the mechanisms controlling elongated grain growth and (2) the debonding of the interfaces between grains during crack extension. Recently, experimental techniques were developed that allow us to characterize interfacial debonding for such systems and assess the influence of the GB glass composition.

The design and fabrication of two test stages for both optical microscopy and SEM studies are now completed and will allow ceramics and composites to be loaded under control conditions while observing the crack. Both three- and four-point flexural specimens and a double-cantilever-beam specimen can be subjected to a variety of loading conditions. Thus, the fracture resistance versus crack length curves can be accurately measured while the crack path and crack wake characteristics are observed over a magnification range of ~ 50 to 100,000x. This capability will provide unique details of crack-tip characteristics and crack-wake-bridging parameters critical to the design of advanced ceramics.

Because the creep response of silicon nitride ceramic is also influenced by the elongated grain structure and by the GB phase(s), it is imperative that microstructural design concepts also address creep behavior. Collaborations with Max-Planck-Institut für Metallforschung have investigated tensile creep in silicon nitride materials containing  $\text{Yb}_2\text{O}_3$  additive. The study indicates that the creep rate of this particular silicon nitride is reduced by decreases in the  $\text{Yb}_2\text{O}_3$  content and decreases in the size of the elongated grains at 1200 and 1300°C. Under these conditions, the lifetime is inversely dependent upon creep rate and is limited by crack formation by cavitation.

In companion studies, preliminary flexural creep results of SRBSN indicate that the MW-sintered SRBSN ceramics exhibit creep rates that are two orders of magnitude lower than those for conventionally sintered samples at 1200 and 1300°C. Studies are currently evaluating the effects of microstructure, composition, and silicon powder purity on creep rates of MW SRBSN ceramics.

#### 4.4.3 Transformation-Toughened Composites

Recent neutron diffraction and Raman measurements reveal that the internal tensile stresses, due to thermal expansion mismatch, in zirconia grains in an alumina matrix increase as zirconia content decreases in agreement with analytical models. However, our analytical models also predict that dependence of these internal stresses on zirconia content is quite different if the zirconia is present as isolated grains or as a continuous phase (i.e., a thin film along alumina grain boundaries). Concurrent quantitative analyses of microstructural evolution show that both zirconia grain growth rates and the spatial distribution of zirconia are a function of the zirconia content. The zirconia grains remain isolated at low zirconia contents (e.g., <15 vol %). Above this level, grain growth evolves to form a more interconnected structure (i.e., formation of a continuous zirconia phase). As a result, the measured internal stresses in zirconia grains exhibit a transition from those predicted for an isolated phase to those based on a continuous phase with increase in zirconia. As our experiments show, the martensite transformation initiation temperature,  $M_s$ , for zirconia grains embedded in alumina (1) increases with the increase in zirconia grain size, and (2) the dependence of  $M_s$  upon zirconia grain size increases as the zirconia content decreases. These effects are a direct result of residual stresses imposed on the zirconia due to the thermal expansion mismatch with the matrix. The key here is that the toughness achieved by triggering the transformation during fracture is dependent upon the martensite transformation initiation temperature attained in the composite. Therefore, the ability to quantitatively

account for the effects of thermal expansion mismatch stresses and zirconia grain size is a critical factor in optimizing the transformation toughening in composites where it is often desirable to alter either the zirconia content or the matrix material. Until now, this was accomplished primarily by trial and error as sufficiently detailed models were not available for the design of transformation-toughened composites.

#### 4.5 MATERIALS THERMAL ANALYSES — *T. G. Kollie, R. S. Graves, G. M. Ludtka, G. Mackiewicz-Ludtka, K. E. Wilkes, and D. W. Yarbrough*

The Materials Thermal Analyses Group conducts research and monitors subcontracts to other installations primarily for the DOE's Assistant Secretary for Energy Efficiency and Renewable Energy in the areas of advanced thermal insulation for buildings, evaluation of existing materials for building insulation, and technology transfer to the buildings industry. A new area of research for the group, which is conducted for DOE's DPs, is modeling of thermal effects occurring during processing of materials to predict microstructures, thermal stresses, and properties in the materials. Other research is performed for the Environmental Protection Agency (EPA), the Department of Defense, and other DOE programs and facilities in areas related to thermal insulation, properties of materials, and thermal processes.

Field management is provided for the materials part of the Building Thermal Envelope Systems and Materials Program conducted by ORNL for the Building Systems and Materials Division of the Office of Buildings Energy Research of DOE. This program addresses the development and characterization of new and existing insulation materials for buildings and building equipment. The Materials Program goals are to develop new materials that can reduce building energy consumption by 20% by 2010 and which are cost competitive for their application.

Powder-filled evacuated panels (PEPs), with absolute pressures near 1-mm Hg pressure, provide an alternative insulation with a much higher initial R-value (R-25/in.) than CFC-blown foam insulation (R-8/in.). The major efforts of the program have been directed toward development of lower-cost, higher-R-at-higher-pressure powders to improve the thermal performance of PEPs. A secondary research effort has been directed toward identification of low-permeability barrier material to encapsulate the powder and retain the vacuum and thereby extend the lifetime of the PEPs.

Both the ORNL Radial Heat Flow Apparatus (RHFA) and the Heat Flow Meter Apparatus (HFMA) were used to study heat-transport mechanisms in PEP powders as a function of gas pressure in the powder. With the RHFA, the powder is not subjected to the load of the atmosphere, whereas it is in the HFMA technique. An application was filed with the U.S. Patent Office for a calcium silicate powder for use as a PEP filler material. This powder was of interest because it costs only about 55¢/lb and has a thermal resistivity (R/in.) of about 60 at 0.01-mm Hg pressure as measured in the RHFA. Comparative values for FK500LS precipitated silica powder, the most commonly used PEP filler material, are 75¢/lb and about 40 R/in. at 0.01-mm Hg pressure. We also studied the thermal performance of several mineral-based powders. One of these powders performed as well as FK500LS at higher pressures and performed superior below 1-mm Hg pressure.

The tapped density of this powder is 0.42 g/cc; it costs 29¢/lb. We are in discussions with the manufacturer to explore modifying their processing to reduce the density of the powder, while not raising costs or decreasing performance; the manufacturer believes these goals can be accomplished. Because we do not believe that atmospheric load will increase the density of this powder more than a few percent, we also believe that these powders may prove to be better than FK500LS under atmospheric load. The thermal performance of a biodegradable powder was investigated. This powder is made from a process similar to an aerogel. The cost of this powder was not available; the tap density of this powder is 0.13 g/cc. Our first measurement on this powder in the RHFA was at about 0.05-mm Hg pressure, and the R/in. of the powder was about 92. When the pressure was raised, however, the R/in. dropped dramatically (25 at 1-mm Hg). At 7-mm Hg pressure, the R/in. was less than ten. Based on the experience with other powders, we asked the manufacturer to reprocess the powder, which essentially doubled the R/in. at 1-mm Hg pressure and vastly improved the thermal performance at all intermediate pressures.

We submitted a continuation patent application to the U.S. Patent Office for the gauge we invented to measure the internal pressure of PEPs nondestructively; patent 5,249,454 was granted for the first application on this invention. A second license for this gauge was written with VacuPanel, Inc. Three invention disclosures were filed with the DOE: a method to measure the density of the powder in a fabricated PEP, a method to measure the thermal conductivity of PEP powder as a function of pressure and density while under atmospheric load, and a method to predict the lifetime of PEPs. Proof-of-principle was accomplished on these three disclosures in conjunction with work on CRADAs.

We are developing a procedure to measure accurately the resistivity of high-R insulation such as PEPs. We are using our HFMA for these measurements. Because specimens of the high-R materials must be measured as a composite with lower-R materials, a computer model of the heat-flow patterns must be employed to compute the R of the specimen from that measured by the HFMA. A heat flux transducer (HFT) array was also used to map the heat fluxes across the composite test specimens. The measured heat flux distribution compared favorably with computed values obtained with the conduction code HEATING-7. The HEATING-7 code has been modified to run on a work station. This computer code is an extension of a previously developed, special purpose code that was written last year. The original code was written for specific PEP design and test configurations, and it is not applicable to newer designs. The new code allows users to specify any HFT array they wish as well as removing several other limiting assumptions built into the previous version. Work has begun on a more sophisticated version of the code. With a single sensor, the thickness and conductivity of the cladding material encasing the powder must be known in order to extract a meaningful, effective R-value for a PEP being tested. It is hoped that, with the new array, this cladding information will no longer need to be supplied. The goal of this effort is to produce the software necessary to extract the effective R-value for a panel with unknown cladding conductivity from the experimental data. This analysis will also derive the cladding conductivity.

The ORNL Unguarded Thin Heater Apparatus was modified so that the heat-transfer conditions in attics insulated with loose-fill insulation could be simulated for wintertime

conditions. Results with this device showed that the R of fiberglass insulation decreased when the temperature difference across the insulation exceeded about 20°C, in agreement with the results obtained in the ORNL Large-Scale Climate Simulator (LSCS). An application for this apparatus was filed with the U.S. Patent Office. The decrease in R of the loose-fill insulation is due to the establishment of natural convective cells within the insulation. The temperature gradient necessary to produce these cells in loose-fill insulation can be predicted from measurements of the air-flow permeability of the insulation. Air-flow permeabilities were measured with an apparatus fabricated last year at ORNL to meet the requirements of ASTM C 522. The air-flow permeabilities of the as-blown loose-fill insulation specimens were in the range 20 to  $80 \times 10^{-9} \text{ m}^2$  and were inversely proportional to the density raised to about the third power. The shape of the thermal resistance-temperature difference curve is characteristic of heat transfer by natural convection. The onset of the decrease in thermal resistance is consistent with measured values of air-flow permeabilities and a critical Rayleigh number of 29 or less for an attic insulation with an open top.

A CRADA (ORNL 91-0042) with the Appliance Research Consortium was amended to test advanced insulations immersed in blown foam insulation. A CRADA with PPG Industries (ORNL 91-0071) was amended to extend the work to determine the relationship between morphology of silica particles and the thermal conduction mechanisms operative in the silica powders.

The CRADA with Clayton Homes and Tennessee Technological University (TTU) (ORNL 93-0191) was approved by the DOE-Oak Ridge Operations Office (ORO) on September 22, 1993. PEP superinsulation will be substituted for the existing insulation in parts of the structure, and the energy savings will be determined at TTU and ORNL by comparison to a standard unit. The research will include insulation materials development and characterization, performance evaluations using the LSCS at ORNL, and field testing of full-size units at TTU. Three levels of roof-cavity insulation will be included in the project. One manufactured housing unit will be insulated to present standards, a second unit will have an increased level of conventional insulation, while the third unit will have PEP superinsulation in the roof cavity. The project will also evaluate the use of high-efficiency heat pumps in manufactured home units. The project will last for 3 years. DOE's South Eastern Power Administration will co-sponsor work at TTU on this project, and the PEPs will be supplied by Degussa Corporation.

The CRADA with E. I. DuPont de Nemours, Inc., and VacuPanel, Inc. (ORNL 92-0123), was approved by DOE-ORO on September 30, 1993. The purpose of this CRADA is to identify new, low-permeability barrier materials to contain the vacuum in PEPs and to optimize the cost versus performance of the barrier films. This CRADA is scheduled to last 18 months.

The Cellulose Insulation Manufacturers Association/TTU CRADA (ORNL 92-0147) was approved in March 1993. This cooperative effort will develop a physical property database, improved methods for testing cellulose, and perform a survey of cellulosic insulations manufactured and marketed in the 1990s.

A CRADA (ORNL 90-0028) was amended with the Polyisocyanurate Insulation Manufacturers Association, the Society of the Plastics Industry, and associated trade

organizations and industrial members to evaluate the thermal performance of rigid foam blown with CFC-11 (control) and HCFC-141b. Industry produced boards are being evaluated by field tests in the ORNL Roof Thermal Research Apparatus and by lab thermal resistance (R-value) tests of thin-board specimens. Aging at 75 and 150°F showed the foams blown with HCFC-141b had a thermal resistivity 3 to 16% (average 9.4%) less than that obtained by CFC-11 blowing gases under similar conditions. Laboratory and field tests of the change in K with time are in excellent agreement; the average field exposure temperature was 73°F, which is very close to the aging test of the laboratory results. Work on the original CRADA was completed this year and a summary report written.

Besides the above-mentioned CRADAs, we performed technology transfer in several ways. For example, we measured the thermal conductivity of experimental PEP powders for AlliedSignal Corporation. We participated in the writing of ASTM test procedures by attending meetings of the ASTM C-16 Committee on Thermal Insulation and by participating in interlaboratory comparisons using proposed test procedures to establish the required precision and bias statements.

The work for DOE's DPs is conducted as two CRADAs. The first is with GM (ORNL 92-0113) to develop lightweight materials for transportation applications. The work on this CRADA began in October 1992. The second CRADA (ORNL 92-0077) is with the NCMS and seeks to develop a computational tool/methodology (based on finite element techniques) to predict the effects of heat treatment on the size and shape of industrial, quenched parts. Work on this CRADA began in October 1993. The initial work will develop finite element models to scope distortions in automotive powertrain gears that develop during water quenching. When fully developed, the model will predict gear distortion and residual stress patterns. Eventually, the model will produce a computer-aided engineering tool for predicting heat-treat distortions in any shaped part.

We have performed R&D for the U.S. EPA for their Super-Efficient Refrigerator Program. This work is very synergistic with our work on the Building Materials Program for the DOE. For example, several tasks for the EPA Interagency Agreement concern work on the development of materials for PEPs. In addition, we are studying replacement blowing gases for the environmentally harmful CFC-11 gas-blown foam insulations. The NASA in Huntsville, Alabama, is also involved in this work. NASA is fabricating foam specimens for use in the ORNL thin-slicing-accelerated aging technique. Gases with zero ozone depletion potential, substantially reduced global warming potential, and low thermal conductivity are being studied. Future work will study gases with zero global warming potential.

## 5. NUCLEAR FUEL MATERIALS

*M. J. Kania*

The Nuclear Fuel Materials Section has as its primary objectives (1) the qualification of advanced nuclear fuel materials performance during in-reactor operation and under off-normal conditions, (2) the characterization of fission product transport and behavior in core materials under normal and off-normal conditions, and (3) the development of performance models and codes for use in confirmatory design analysis and safety-related assessments. These objectives are accomplished through management of fuel development activities at the subtask level and the coordinated activities of five technical groups within the section with shared expertise and resources in fuel materials characterization, modeling, irradiation testing, unique remote equipment development, and facility operation.

Research activities of the five technical groups include the operation and development of specialized remote facilities for PIE and handling of irradiated fuel materials [Irradiated Fuels Examination Laboratory (IFEL)]; the evaluation of irradiated fuel performance and fission product characterization, including microstructural characterization and specialized remote equipment development (Fuel Materials Evaluation Group); in-reactor testing and performance evaluation of fuel materials (Fuel Materials Testing Group); characterization/evaluation of fuel and fission product behavior under off-normal conditions [High-Temperature Fuel Behavior (HTFB) Group]; and model development based on fundamental understanding of fuel materials and fission product behavior (Fuel Performance Modeling Group).

Support for these efforts is provided through three main DOE sources: (1) the Commercial MHTGR Program Fuel Materials Development and Fission Product Behavior subtasks; (2) the NPR-MHTGR Fuel Performance Subtasks (normal operating conditions and off-normal operating conditions), the Fuel Performance Model/Code Development Subtask, and the Fission Product Transport Subtask; and (3) the ANS Fuel Materials Development Subtask. Funding for the NPR-MHTGR Program was terminated at the end of September 1993, and the level of support for the Commercial MHTGR Program was reduced significantly. As a result, a significant number of the professional staff from the Fuel Materials Testing Group, the HTFB Group, and the Fuel Performance Modeling Group were transferred to other positions within ORNL. These three technical groups were eliminated, and the remaining technical staff were reassigned to the Fuel Materials Evaluation Group. Together with the IFEL, the Nuclear Fuel Materials Section has positioned itself to meet the needs of the downsized Commercial MHTGR Fuels/Fission Product Behavior Subtasks and the ANS Fuels Subtask. New areas of support for the section are most likely to be with the National Environmental Restoration and Waste Management Programs.

## 5.1 IRRADIATED FUELS EXAMINATION LABORATORY — C. E. DeVore

The IFEL is a major hot-cell facility located in Building 3525. During the report period, programmatic work, as well as facility upgrade work, was accomplished. Programmatic work included the receipt, remote disassembly or machining, and either transfer to the IMET Group at Building 3025E or examination by the Fuel Materials Evaluation Group at Building 3525. The experimental designations and their program sponsors processed during this report period were HRB-21 (NE-MHTGR); NPR-1, -2, and -1A (NP-MHTGR); JP-14 and -17 (Fusion); and HANS-2 (ANS). Production of Iridium-192, which was begun in 1992, continued in support of the Isotope Production and Distribution Program. A total of 431,200 Ci were received from the HFIR, and 302,110 Ci were shipped to commercial customers.

Many loads of in-cell waste consisting of obsolete task-specific and facility equipment that had been accumulated from many years of operation were transferred to the solid waste storage area.

There were 16 occurrences during 1993 compared to 26 for 1992. For the year 1993, five involved facility condition (system/component degradation), ten involved personnel contaminations, and one involved a near miss (reported electrical shock). During 1992, there were nine reported contaminations. Due to the reporting requirement change with the implementation of DOE Order 5000.3B, which considers company-issued clothing the same as personal clothing, several 1993 reported contaminations would not have been reported in 1992. The use of corrective actions resulting from 1992 occurrences has led to an overall reduction in the contamination occurrences at the facility. The continued use of pre-job briefings involving all personnel has led to a better understanding and working relationship between operations and support personnel.

The facility safety documentation upgrade performed in 1992, which resulted in a hazard classification of "low," was measured against a new DOE standard based on radionuclide inventory. This resulted in a DOE hazard of category 2, which is comparable to an Energy Systems "moderate." During the year, the consideration of the Laboratory to "upset" conditions involving potentially high inventories of radioactive materials resulted in the commitment to begin the process to upgrade the facility to a "moderate" (DOE category 2) facility. Efforts have begun to accomplish this upgrade.

An operator training program, begun in 1992, has been completed for the core requirements for operators. Tests for operator certification remain to be developed. Supervisor certification training and certification testing remain to be completed. The training program was being developed by contracting personnel.

The generation of liquid low-level waste (LLLW) has been held to a minimum due to the Federal Facilities Agreement. The agreement resulted in the suspension of use of the collection tank at the waste disposal facility. In order to continue the generation of waste, a trucking station that pumps LLLW from the facility's LLLW collection tank to a transportable tanker has been used to continue limited generation as the result of programmatic requirements. Under this condition, although limited in the quantity of LLLW generation, facility operation has continued.

In order to provide a more responsive organization to the requirements of the "new culture" desired by DOE for the operation of nuclear facilities, during 1992, new personnel were assigned for the management, operation, and maintenance of the facility. As the result of program terminations and budget uncertainties in 1993, several personnel positions were eliminated during the last quarter of the year.

## 5.2 FUEL MATERIALS EVALUATION — *N. H. Packan and M. J. Kania*

PIE was completed on two major fuel irradiation capsules, NPR-1 and -2, for the NP-MHTGR Program. The test capsules were transferred from the HFIR to the IFEL where they were disassembled, and their components (fuel compacts, unbonded particles, graphite holders, and flux monitors) were subjected to metrological examinations. Extensive destructive and nondestructive PIES including ceramography, compact deconsolidation followed by particle gamma-ray spectrometry, acid leaching, and SEM/microprobe analyses were conducted. These tasks are all directed toward learning what features of the current NP-MHTGR coated fuel particle design contributed to poor in-reactor performance as evidenced by high activity releases during the HFIR test irradiations.

PIE continued on fuel compacts and unbonded particles from experiment HRB-21 for the Commercial MHTGR Program. Destructive and nondestructive examinations including ceramography, compact deconsolidation, particle gamma-ray spectrometry, acid leaching, and SEM/microprobe analyses were conducted. These tasks are directed toward isolating the design- or performance-limiting mechanisms responsible for the poor in-reactor performance of the current-generation commercial MHTGR fuel. Such poor performance was evidenced by the high activity releases in the purge gas of capsule HRB-21 during its irradiation test phase. At the end of this report period, a small amount of PIE remained to be completed on a portion of the HRB-21 irradiated fuel and nonfuel specimens.

## 5.3 HOT CELLS REVITALIZATION PROGRAM (HCRP) — *C. E. DeVore*

The HCRP was initiated in 1987 in response to Laboratory management's request to consolidate metallurgical examination of hot-cell work into two facilities, Buildings 3025E and 3525. The program was to be completed in two phases; Phase I would bring both buildings into fully operational status, and Phase II would include correcting remaining design deficiencies and would modify or add systems that would promote safe and efficient operations in both facilities. Phase II was planned to be completed under a Multiprogram, General-Purpose Facility, FY 1996 Line Item.

During 1993, approximately \$436,000 was spent in support of the revitalization program. The major expenditures were for program support (26%), Building 3525 decontamination activities (35%), Building 3525 equipment upgrade (24%), Building 3025E upgrades (10%), and miscellaneous (5%). The program support provided management of the program.

The major emphasis of the work in Building 3525 included procurement of equipment and services for facility decontamination. Equipment included spray equipment for the application of strippable coatings used in the removal of loose surface contamination of facility areas. Services procured for the facility decontamination were for a demonstration of crystalline ice blast technology in the decontamination of obsolete in-cell facility equipment in order to allow personnel contact handling while maintaining personnel radiation exposures within acceptable levels. The equipment upgrades consisted of maintenance of existing facility equipment to maintain operability until modern replacement equipment is secured and installed.

The effort in Building 3025E for calendar year 1993 involved a small expenditure of funds dedicated to logistical support in preparation for repairs to two shielding window assemblies designated for installation in cell 2. These repairs are scheduled for completion in January 1994.

The funding of the HCRP has been provided by Laboratory overhead funding. As the result of DOE's concern over the use of overhead funds being directed toward facilities which, although multisponsored, could not be defended as being for the benefit of the entire Laboratory, the funding for FY 1994 was redirected from the HCRP to surveillance and maintenance of Building 3525. The justification for the use of overhead funding for this facility is due to the role the facility plays in the infrastructure of the Laboratory's nuclear capabilities for upset response capability. Building 3025E has sufficient programmatic support into the near future, while Building 3525 is struggling to seek new programs and possible new missions.

As the result of this development, the HCRP has been canceled. The future of any facility revitalization program will be determined by the programmatic support provided to the facilities.

#### 5.4 HIGH-TEMPERATURE FUEL BEHAVIOR — W. A. Gabbard

A number of enhanced capabilities were implemented into the Core Conduction Cooldown Test Facility (CCCTF) in support of accident condition testing for the Commercial MHTGR and NP-MHTGR. Expanded diagnostic improvements and additions were in the areas of: on-line chromatography, moisture ingress event initiation, control and monitoring, beta-active fission product detection and analysis, more efficient fission gas trapping detection and trapping, and improved data collection and handling system. These enhancements expand the range of MHTGR licensing-based events the CCCTF can simulate and provide improved control of test environments.

The HTFB Group completed the planned removal of the CCCTF from its original installation site in Building 4501 during the first quarter of FY 1993. The new site was selected (Building 3525), and layouts, including engineering analysis of floor loading, were made for installation of a vendor-supplied custom hot cell. The hot cell with manipulators was specified to accomplish accident mode testing of ceramic-coated nuclear reactor fuel under high-temperature conditions. The hot cell was designed and purchased during the second quarter of the year.

During the third quarter, site preparation in Building 3525 was completed. The custom hot cell was delivered and reassembled on site. A shield integrity test was performed using existing MHTGR fuel in the 50-Ci range. Additional shielding modifications were performed to the custom hot cell to meet the shielding criteria whereupon the hot cell was accepted.

The pursuit of other applications for the CCCTF resulted in the direct involvement of M&C Division manpower and facilities in the Oak Ridge Reservation (ORR) Waste Management activities. The HTFB Group successfully conducted vitrification tests of surrogate K-25 Pond Sludge Program test samples using the furnace assembly in the HTFB laboratory. The promotion of CCCTF capabilities was met with considerable interest from waste management personnel due to the facility's ability to perform thermal treatment of radioactive samples using Building 3525 equipment and to perform surrogate (nonradioactive) sample testing in the lab with identical equipment. Comparison of test data from real and surrogate samples was recognized as a significant aspect of radioactive waste treatment, testing, and modeling.

Current installation plans are to bring the CCCTF on line during FY 1994 with test modes of high-temperatures and inert gas using a cold finger and cryogenic traps for studies of fission product kinetics. Further fuel testing appears to be in serious doubt, and the CCCTF will be tailored to waste management interests.

#### **5.5 FUEL MATERIALS TESTING — *J. T. Parks and M. J. Kania***

Qualification was completed on the in-reactor test data obtained during the HFIR irradiation of three major MHTGR fuel test capsules. This process integrates selected reactor data, such as neutron flux level, power, coolant flow, temperature, and pressure, with the principal test data, such as TC readings, purge gas flow, and mixtures, into an experimental database. Also incorporated were the analysis of the in-reactor dosimetry test data and modeling of each test capsule using the DORT and ORIGIN computer codes. Successful completion of this effort provided a valid benchmark for the specific test capsule thermal and neutronic design methodologies employed.

Enhancements were made to the Materials Irradiation Facility system to significantly improve the on-line monitoring of fission gas release and test data evaluation for MHTGR fuel irradiation test capsules in the HFIR. New instrumentation capabilities include: on-line moisture monitors, local gamma-ray spectrometry data acquisition and analysis, and installation of a real-time data link between the experimenter and the HFIR test capsule instrumentation facility. The additional on-line instrumentation will improve the quality of real-time data, increase efficiency, improve the quality of on-line data, and provide better control of the irradiation test environment.

#### **5.6 FUEL PERFORMANCE MODELING — *O. M. Stansfield and M. J. Kania***

A program sponsored by the Commercial MHTGR Program was developed and executed for utilizing noninvasive optical techniques for the characterization of SiC structure and

chemical composition on MHTGR TRISO-coated fuel particles. The proposed SiC characterization techniques will improve the uniformity of coatings and assist in the development of particle coatings that meet MHTGR fuel quality requirements of  $\leq 5 \times 10^{-5}$  coating defects.

A remote technique was developed for use in hot-cell environments to measure the fraction of defective coatings for irradiated MHTGR fuels. The technique, called leach/burn/leach, subjects an irradiated MHTGR fuel compact to a 100°C aqueous acid leach, followed by a burn step to remove carbonaceous materials, and then another acid leach. The fraction of particles with failed coatings is determined at each step by an analysis of the leach solution for actinide elements or specific fission products. The measurement of irradiated fuel particle failure fractions in MHTGR fuels is necessary to validate performance models. The leach/burn/leach procedure will provide an additional capability for failure fraction determination independent of existing metallographic, gamma spectrometric, and fission gas release methods.

A direct physical correlation was established for the high level of fission gas activities observed in-reactor with the performance of the structural coatings on fuel particles in four recent, major MHTGR fuel test capsule irradiations. Massive failure, up to 100%, of the outer pyrocarbon layers along with percent-level SiC layer failures was observed in particles irradiated to moderate fluences. These data compare poorly with precharacterization quality control data, which indicate as-manufactured SiC layer and pyrocarbon layer defect levels at  $\leq 5 \times 10^{-5}$ . The PIE data suggest an irradiation-induced failure mechanism for the structural coating layers not previously observed in in-reactor experiments.

### 5.7 ADVANCED NEUTRON SOURCE FUEL DEVELOPMENT — G. L. Copeland

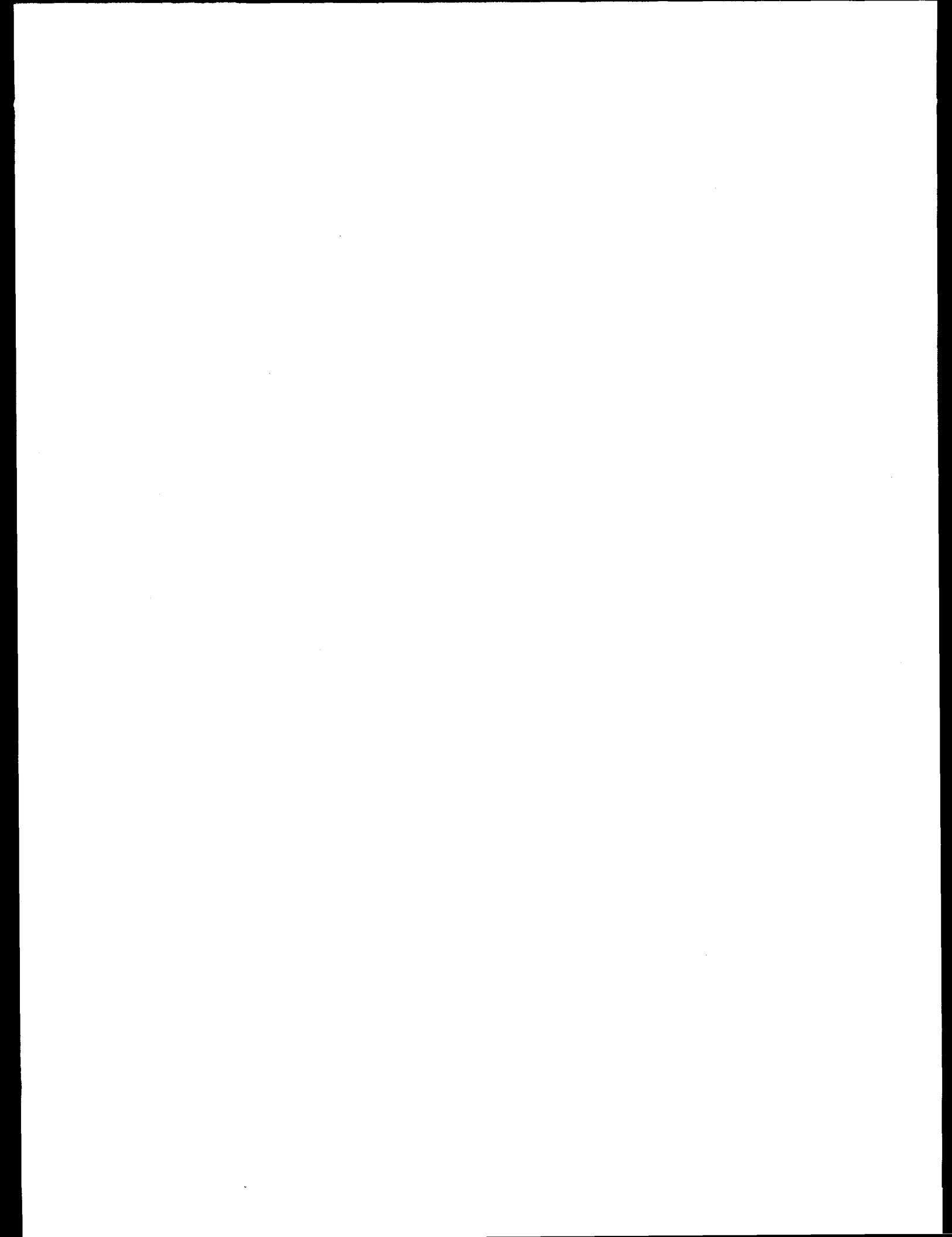
Work continued in support of a  $\text{U}_3\text{Si}_2\text{-Al}$  dispersion as the reference fuel for the ANS. The work was concentrated in the areas of irradiation of fuel specimens, fuel performance modeling, and fuel fabrication.

Fabrication efforts this year produced a series of plates which show the feasibility of producing fuel gradients in both the axial and radial directions as required in the current ANS design. Additionally, two series of miniature plates were fabricated which are now undergoing accident testing in a pulse reactor in Japan.

Evaluation of the first capsule irradiated in the target region of the HFIR was completed. The results are encouraging for the performance of the fuel at ANS conditions. A second capsule was irradiated and is now undergoing evaluation at ANL. Results from the first test have been incorporated into the refined fuel performance model.

### **5.8 CORROSION, MIGRATION ET DISTRIBUTION IRRADIATION EXPERIMENT (COMEDIE) FISSION PRODUCT TRANSPORT PROGRAM — O. F. Kimball**

The first interactive irradiation test (BD-1) of the COMEDIE Fission Product Transport Program sponsored by the Commercial MHTGR Program was successfully executed in the SILOE reactor pool at the Centre d'Etudes Nucleaires de Grenoble (CENG), France. Following completion of a 64-d irradiation, the test rig was subjected to four depressurization events, each involving more than 100 separate operations. All were successfully executed and the respective test data gathered. This effort was effectively managed among the four major participants [USDOE, Commissariat a l'Energie Atomique (CEA), GA, and ORNL] by ORNL. Post-test examinations and analysis are currently under way at CENG. The information from this test will contribute to a validation database that will be used by the Commercial MHTGR Program to modify or validate their fission product distribution, MHTGR core corrosion, and transient thermal/fluid hydraulic codes.



## 6. PROGRAM ACTIVITIES

This section of the report deals with the program activities in which the M&C Division was engaged to a major extent during the report period. Brief statements of the purpose, nature, and scope are presented on the following programs sponsored by the USDOE: BES-Materials Sciences, Electric Energy Systems, Reactor Materials, EE Materials, Space and Defense, FEM, and HTS.

### 6.1 BASIC ENERGY SCIENCES PROGRAMS — *L. L. Horton*

The Office of BES funds a wide range of research activities in the M&C Division. The majority of our funding comes from the Division of Materials Sciences. This program provides the fundamental basis for the division's core programs and a framework to foster the development of innovative materials and processes. The overall goal is to develop an understanding of materials and materials processes at all levels, from the atomic structure to the macroscopic properties. The program includes the research components required to better understand and utilize materials: synthesis, processing, fabrication, characterization, and development of models/mechanisms. The R&D effort has close ties to the DOE technology programs, especially the materials-related efforts funded by the EE, Fossil Energy, and Fusion Energy Programs, as well as the NRC. In addition, there are more than 12 CRADAs with industry that have grown from the BES-funded tasks in M&C.

Research in synthesis and processing science includes the development of fabrication and joining techniques for advanced intermetallic alloys, ceramics, and composite structures. Intermetallics research includes fundamental studies of phenomena related to physical and mechanical properties, ranging from atomic bonding to environmental embrittlement. Investigations in the ceramics program are focused on establishment of basic microstructure-property relationships for the design of advanced ceramics and ceramic composites, including self-reinforced silicon nitrides. Future directions in processing R&D under assessment include magnetic materials and toughened ceramics coatings/scales for gaseous corrosion protection. A strong first-principles theory effort is integrated with the alloy development program. During the past year, a preliminary evaluation of capabilities and status of first-principles theory of ceramic materials was initiated. The goal is to establish a first-principles effort that complements the experimental program.

The welding and joining program continues to investigate the evolution and stability of microstructures and properties of weldments, including mathematical modeling and experimental verification of transport phenomena. This task serves as the coordinator for the national BES Welding Program. With the expansion of the process modeling efforts in the M&C Division, the modeling component of this task has broadened somewhat to include the basic component of related solidification modeling efforts, including superplasticity and related process modeling.

Important to all of the tasks is the development and application of advanced characterization techniques, including AEM, APFIM, MPM, XRD, and ion beam techniques.

Ion implantation is used to study defect interactions, radiation effects, and to modify surface-related properties of polymers and ceramics. New techniques and facilities for high-temperature MPM studies and the 3-D multielement atom probe are near completion. During the past year, orders were placed for a new 200-kV FEG analytical microscope to replace a 14-year-old instrument and an advanced analytical SEM. An AFM was added to the MPM facility. A major ORNL initiative related to our characterization efforts is the Advanced Materials Science and Engineering Complex which includes the Materials Research and Development Laboratory. This facility would provide facilities designed for the specific requirements (vibrations, electric fields, etc.) of modern analytical equipment.

M&C continues to develop new and improved techniques for characterization with x rays and neutrons, both in-house and at synchrotron facilities. EE and BES are jointly supporting a new facility, the HFIR-HTML Neutron Residual Stress Facility. R&D with this facility will use neutron diffraction techniques to measure the residual stresses in parts after manufacturing processes such as welding. Together with Solid State Division, M&C is actively developing the optics and design for a new, multipurpose X-ray beamline facility (UNICAT) and the conceptual design for an X-ray microprobe (MICROCAT) for the Advanced Photon Source under construction at ANL.

There is continuing increased emphasis on radiation effects, especially with regard to the effects of neutron environments. This program continues to make major contributions to the understanding of mechanisms of RPV embrittlement. In the past year, studies of the HFIR pressure-vessel embrittlement have uncovered the likely contribution of gamma irradiation to the displacement damage in the pressure vessel for HFIR and similarly configured reactors (e.g., reactors with a long water path between the core and pressure vessel). Recently, the M&C BES Radiation Effects Task was named coordinator for the national BES Radiation Effects Contractors Group. In related activities, M&C staff continue to play a major role in the fuel element and corrosion studies for the development of the ANS.

Through ORNL and the Oak Ridge Institute for Science and Education (ORISE), BES supports the SHaRE Cooperative Research Program and the Oak Ridge Synchrotron Organization for Advanced Research (ORSOAR). The SHaRE Program allows scientists from universities, industries, and other national facilities to have access to facilities in M&C, especially the AEMs, APFIMs, and MPMs. The ORSOAR Program supports an X-ray beamline at the NSLS. This facility is used for cooperative research by scientists from more than 20 universities and industrial institutions.

In addition to the Division of Materials Science, M&C also receives support from the Division of Advanced Energy Projects (AEP), BES. An R&D project focussed on the development of novel composite coatings to control high-temperature friction and wear is in its second year. It involves the use of CVD to deposit multiphase coatings containing high-temperature lubricants. In 1993, M&C also organized an AEP workshop on "Innovation in Materials Processing and Manufacture: Exploratory Concepts for Energy Applications."

## 6.2 ENERGY RESEARCH LABORATORY TECHNOLOGY TRANSFER PROGRAM — *L. L. Horton*

The Energy Research Laboratory Technology Transfer (ERLTT) Program sponsors a wide range of activities that involve industrial interactions. A major portion of the funding from this program supports CRADAs, both large, multi-year projects and smaller (<\$100,000), single-year projects. In FY 1993, funded and approved ERLTT CRADAs in M&C included projects on wideband MW processing equipment (MLI with Instrumentation and Controls Division), MW sintering of tantalum capacitors (AVX Tantalum Corporation), advanced deposition and substrate technologies for thin-film polycrystalline silicon (EPRI, with Solid State Division), MW processing of silicon carbide (Dow Chemical), and coordinate measurement standardization studies for high-precision ceramic and metal components (Brown and Sharpe, part of the EE/DP/ER Ceramic Machining effort). A number of other CRADAs were in various stages of negotiation at the end of the fiscal year. Also now available (FY 1994) through the ERLTT are funds for technology maturation, personnel exchanges, and technical assistance. M&C is an active participant in these programs.

## 6.3 REACTOR MATERIALS AND GAS TURBINE (GT)—MODULAR HELIUM REACTOR (MHR) PROGRAMS —*P. L. Rittenhouse*

### 6.3.1 Advanced Liquid-Metal Reactor (ALMR) Materials Technology

A bellows exposed to liquid sodium for 5 years at 260°C in the Experimental Breeder Reactor II (EBR-II) was examined. Some metal deposits were seen at the gas-sodium interface inside the bellows, but there were no changes in the properties of the bellows compared with identical archive hardware.

Samples of a 2.25 Cr-1 Mo steel tube removed from a helical coil steam generator operated by Energy Technology Engineering Center for 17,000 h with sodium as the heat-transfer medium were examined. The tube specimens were in excellent condition with approximately 25  $\mu\text{m}$  of oxidation on the inner (steamside) surface and with only a few occurrences of metal deposits on the exterior surface.

Long-term creep tests of types 304 and 316 stainless steel were continued. The longest test times are approaching 14 years.

### 6.3.2 GT-MHR Fuels and Materials Programs

Emphasis in DOE's GCR activities was changed from the steam-cycle MHTGR to the direct-cycle GT-MHR. This change, for the present time, has had only minimal effects on the content and scope of work in the division and at ORNL. Our areas of focus continue to be technology development relative to coated-particle uranium oxycarbide (UCO) fuel, behavior of fission products, properties of graphites for structural and fuel block applications, and metallic materials used for structural components (e.g., reactor vessels and core internal structures). The graphite and metals activities were supported primarily under the MHTGR-NPR Program as discussed in Sect. 6.3.3.

The PIE of UCO fissile and thoria fertile fuels irradiated in the HFIR in capsule HRB-21 was continued throughout the year. The PIE included visual examination and metrology of fuel compacts and graphite sleeve components, deconsolidation of fuel compacts to allow access to the fuel particles, gamma analysis of individual fuel particles, ceramography of fuel compacts and fuel particles, SEM of fuel particles, and leach/burn/leach tests of the fuels to determine fuel failure fractions. PIE of components from the BD-1 fission product transport experiment conducted last year in the COMEDIE loop at the CEA facility in Grenoble, France, was also completed and the results reported. Finally, a subcontract was placed with the Kernforschungsanlage in Jülich, Germany, to perform fission product-related PIE on graphite specimens irradiated in the HFR-B1 experiment at Petten, The Netherlands.

### **6.3.3 MHTGR-NPR Fuels and Materials Technology**

Although the termination of all NPR programs was announced late in 1992, continued funding was provided by DOE to permit orderly closeout and the completion of various risk-reduction tasks related to fuels, graphites, and metals. The largest risk-reduction task was the completion of the PIE of coated-particle UCO fuels irradiated in two HFIR capsules (NPR-1 and -2) and in a single capsule (NPR-1a) in the advanced test reactor at INEL. The types of examinations completed were as described in Sect. 6.3.2 for capsule HRB-21. A final report on these irradiation experiments was issued in September 1993.

Significant efforts (mechanical and physical properties, oxidation behavior, etc.) aimed at risk reduction were also carried out on structural and fuel block graphites. Additionally, efforts aimed at providing C/C composite control rod bodies to replace the traditional Alloy 800H cans were carried out successfully.

Work also continued throughout the majority of the year on metallic materials associated with the core internals, the steam generator, and the reactor vessel. Of special significance were completion of (1) the determination of the effects of MHTGR-specific irradiation conditions on the NDT shift in SA508 and SA533B reactor vessel steels and (2) detailed metallurgical examinations of components of a steam generator ringheader removed from the Fort St. Vrain GCR.

Finally, all program and quality records associated with the NPR Program were provided to DOE for storage at approved sites. Additionally, funds are being provided by DOE for 1994 to permit the completion of waste disposal, cleanup, and facility restoration activities associated with the fuels PIE that was performed.

### **6.3.4 Materials Technology for the HWR-NPR**

All structural materials activities related to the HWR version of the NPR were terminated early in the year, and all appropriate records were provided to DOE for safe storage.

#### **6.4 ENERGY EFFICIENCY AND RENEWABLE ENERGY MATERIALS PROGRAM— R. A. Bradley, D. R. Johnson, H. W. Hayden, P. J. Blau, P. Angelini, P. S. Sklad, M. A. Karnitz, T. G. Kollie, D. M. Kroeger, and V. J. Tennery**

Materials programs for the DOE-EE comprise about one-third of the division's R&D activities. We have lead laboratory roles and/or major materials support tasks in the following EE Programs: (1) Ceramic Technology, (2) Transportation Materials, (3) AIC Materials, (4) CFCCs, (5) Materials for ATS, (6) Advanced Heat Exchangers, (7) Tribology, (8) Building Materials, and (9) HTS Partnerships Program. In the Ceramic Technology, AIC Materials, CFCC, and ATS Programs, we provide technical support to DOE in the planning, implementation, and management of the national DOE programs. This involves extensive interfaces and subcontracts with industry, universities, and other federal laboratories in addition to research in the M&C Division.

##### **6.4.1 Ceramic Technology Project — D. R. Johnson**

The CTP was initiated in FY 1983 to meet the ceramic materials technology needs of the companion DOE engine programs. The original goal of the program was to establish the technology base to allow private industry to supply reliable and cost-effective ceramics for use in advanced engines and other energy conversion applications. The program was accomplished by using an R&D agenda developed following an extensive industrial assessment of needs that was formatted into a dynamic 5-year project plan and later revised to the complete 10-year plan. The original program included a balanced emphasis on the three technology areas recognized as necessary to achieve reliability in structural ceramics: (1) materials and processing, (2) design methodology, and (3) database and life prediction. The R&D tasks in the program are performed in-house at ORNL, at other national laboratories, and through subcontracts with private industry and colleges and universities.

The program goal of reliable ceramics was largely met upon completion of the 10-year plan in 1993, a conclusion borne out by industry's successful experience in running ceramic components in engines. However, commercial implementation of the benefits of ceramic engine components remains clouded by the relatively high cost of the ceramic components. Based again on extensive input from industry, the direction of the CTP is now shifting toward reducing the cost of ceramics in order to facilitate commercial introduction of ceramic components for automotive and diesel truck applications in the near term. This implies inclusion of moderate-temperature applications as well as the very high-temperature automotive GT application. A systematic approach to reducing the cost of components is planned. The work elements are as follows: economic cost modeling, ceramic machining, powder synthesis, alternative forming and densification processes, yield improvement, system design studies, standards development, and testing and database development.

##### **6.4.2 Lightweight Materials for Transportation Program — P. S. Sklad**

The Lightweight Materials for Transportation Program was initiated within the DOE to focus on the needs of the domestic automotive industry, including automakers, materials suppliers, and component suppliers. The program, developed through the Office of Energy Efficiency and Renewable Energy—Office of Transportation Materials, sponsors R&D in industry, universities, and national laboratories. Two families of materials are most generally applicable under this program: metals and polymers. Within the metals group, aluminum alloys, magnesium alloys, and HSS are considered highest priority,

with MMCs, titanium alloys, intermetallics, and HSS also desirable for longer range applications. General needs include process modeling, process development for improved quality and performance, alloy development to enhance process ability and properties, process development to transition typical defense/aerospace processes to automotive process demands, development of joining techniques, and recyclability. In the polymer area, unreinforced and reinforced plastics are considered highest priority, with advanced high-strength, high-stiffness materials, such as rigid polymers, desirable for future development. Needs in polymers include improved process modeling to optimize design and performance; development of rapid, production-scale processes for reinforced plastics; low-cost and durable tooling; development of low-cost materials and components; joining; improvement in the understanding of crash performance; recycling; and improvements in accelerated testing and modeling of durability.

A Lightweight Materials for Transportation Program Plan has been prepared and considerable interaction with members of the U.S. Automotive Materials Partnership has ensured consistency of technical direction. The goals of the program are fully consistent with those of the USCAR, as well as with those of the Partnership for a New Generation of Vehicle proposed by President Clinton. Initial R&D efforts have focused on advanced forming of aluminum, modeling of superplastic forming, and adhesive bonding of polymer composite materials.

#### **6.4.3 Tribology Project — *P. J. Blau***

ORNL has participated in the Office of Transportation Materials-Tribology Project since its inception. Primary emphasis is on developing the technology base for new ceramics, composites, and other advanced materials so that their potential for use in friction and wear-critical applications can be determined. During 1993, friction and wear analyses were performed on in situ-reinforced silicon nitride materials for engine wear parts and carbon-graphite materials for pistons. The work on silicon nitride involved CRADAs with two U.S. companies, and the effort was performed using specimen materials supplied by two graphite producers. ORNL also works with ASTM on wear test standardization, automotive companies on material screening issues, and with the DOE/ORNL CTP in the area of impact wear of emerging materials for ceramic engine valves.

#### **6.4.4 Advanced Industrial Concepts Program — *P. Angelini***

The mission of the Office of Industrial Processes in DOE-EE is centered on the development of improved technology for industrial plants of the future. The objectives are to develop improved materials and processes that will have the most impact toward increasing energy efficiency, improving the environmental aspects of processes, and enhancing the competitiveness of industry. The programs with which ORNL has interactions include the Pulp and Paper Program, Metals Initiative, Separations Program, and the Advanced Industrial Materials (AIM) Program. Activities related to the Pulp and Paper Program have focused on combustion needs and responses to two calls for proposals from DOE. Activities on the Metals Initiative Program have included attending workshops, plant visits, and preparation of white papers in response to near-term solutions to needs identified by the steel industry. Efforts on the Separations Program have included subcontract activity on advanced membrane technology. The R&D related to the AIM Program is presented below.

ORNL has project management responsibility for technical projects of the national AIM Program. The national program funds projects in various national laboratories and industry. The R&D efforts include work on intermetallic alloys, MW processing, spraying of metal powder, aerogel thermal insulation, coatings, and other advanced materials. Strong industrial interaction is important to the program. The program has initiated Materials Needs and Opportunity Assessments in a number of industries including the pulp and paper industry and the glass industry. The assessments are carried out by sponsoring workshops and plant visits. The AIM Program also has an annual meeting which is coordinated through ORNL.

#### **6.4.4.1 ORNL AIM Program — P. S. Sklad**

Projects under the AIM Program at ORNL, along with subcontracts funded through ORNL, focus on the development of a wide range of materials and processes. A number of computer modeling activities support these development efforts. In the area of high-temperature intermetallic alloys (e.g., Ni<sub>3</sub>Al, NiAl, and FeAl), work within the M&C Division has led to collaboration with industrial partners to develop heat-resistant assemblies for heat-treating furnaces, transfer rolls for heat-treating furnaces and slab-reheating furnaces used during hot-processing of steel ingots, and turbochargers for large diesel engines. Other projects include continuous fiber-reinforced TiB<sub>2</sub> for improved Hall-Cell electrodes in the aluminum smelting industry, ultratough metal-bonded ceramics, evaluation of catalyst materials and emission control systems, materials processing technology, MW processing of materials, and welding development. New efforts will focus on materials and processes in the pulp and paper industry.

#### **6.4.5 Office of Industrial Technologies — M. A. Karnitz**

Three industrial projects are being conducted for the Industrial Energy Efficiency Division of the OIT. The objective of the first project is to provide materials technology support for the development of advanced ceramic heat exchangers for industrial applications. ORNL is determining the corrosion limits of the ceramic materials and developing cost-effective methods for fabricating heat exchanger components cooperatively with several industrial suppliers and heat exchanger designers. These high-temperature heat exchangers can save significant amounts of fuel and increase productivity in the industrial processes by increasing the efficiencies of the process. Problems are resolved by the application of current materials technology or by performing the required analysis and testing in cooperation with the industrial suppliers.

The second project for OIT is to provide assistance in the development of CFCCs. The properties of CFCCs make them attractive in a variety of industrial applications where their use can result in energy savings and increase in productivity. Ten industrial companies are developing the primary processing methods for the fabrication of CFCC components for use in industrial applications. ORNL is leading an effort to support the industrial teams by providing the scientific foundation for the successful process development and scale-up. The supporting technologies effort is being conducted by universities and national laboratories and includes the more basic or generic support tasks of composite design, materials characterization, test method development, and performance-related phenomena. In 1993, the emphasis was on the performance and reliability of CFCC materials.

The third project for OIT is a new program to develop ATS for power generation. The objective of the ATS Program is to develop ultrahigh-efficiency, environmentally superior, and cost-competitive GT systems for utility and industrial applications. One of the supporting elements of the ATS Program is the Materials/Manufacturing Technologies Task. DOE-ORO and ORNL are coordinating this element. The objective of this element is to address the critical materials and manufacturing issues for both industrial and utility GTs.

#### **6.4.6 Building Materials — *T. G. Kollie***

The Building Materials Program addresses the development and characterization of new and existing insulation materials for buildings and building equipment. The goal is to develop new materials that can reduce building energy consumption by 20% by 2010 and which are cost competitive for their application. Projects are conducted in four areas: Advanced Materials Research, Existing Materials Performance, Retrofit Insulation, and Materials Program Management. The advanced materials effort emphasizes high-risk, long-term research on alternative materials for chlorofluorocarbon-based insulations and vacuum insulation. The goal of the existing materials performance effort is to improve test procedures on materials of high thermal resistance and to determine properties of existing commercial materials. The Retrofit Insulation Task seeks to develop a new, more efficient method to retrofit insulation within existing buildings. The Materials Program Management Task is concerned with the technical and administrative work of the Building Materials Program.

The Building Materials Program at ORNL interacts with other EE Materials Programs at ORNL and at other facilities. A by-product of the program is the established capacity to characterize the thermal performance of materials used in buildings and building equipment. The current program provides constructive support to other national laboratories; to other federal and state programs [EPA, Federal Trade Commission; and NIST]; to industry (seven CRADAs) and industry trade organizations; and to universities.

#### **6.4.7 High-Temperature Superconductivity Partnerships — *D. M. Kroeger***

The goal of the ORNL Superconductivity Partnerships Program for Electric Power Systems is to jointly develop with industry the technology base needed for U.S. industry to proceed to commercial development of electric power applications of HTS. The three major elements of the program are: (1) wire development, (2) electric power applications development, and (3) the Superconductivity Partnership Initiative. In collaboration with U.S. industry, the program is developing practical fabrication techniques for HTS precursor materials and conductors. In addition, the technologies for producing practical HTS coils for motors, generator, and other power applications are being developed. The program benefits include: reduced energy losses in electric power systems through the demonstration of critical wire and systems technology; increased critical current density ( $J_c$ ) in powder-in-tube conductor through doping powders produced through the aerosol pyrolysis technique and a demonstrated continuous manufacturing process; increased  $J_c$  in deposited conductors through substrate texturing; demonstration of HTS coil fabrication processes through use of innovative insulating and potting materials; and component demonstration for pre-prototype power devices. This program also integrates the ORNL activities with the other national laboratories, government agencies, university centers, and industry groups, including EPRI. Specific activities of the M&C Division on the HTS Partnerships Program are described in Sect. 6.7.

## 6.5 SPECIAL PROJECTS — *R. H. Cooper*

During the past year, ORNL's DOE-sponsored SNP Programs, combined with activities sponsored by industry and federal agencies other than DOE, have provided diverse opportunities for the application of the division's materials technologies. The status of the DOE SNP Programs and selected non-DOE-sponsored programs is discussed below.

### 6.5.1 DOE Space Nuclear Power Programs — *R. H. Cooper*

The division provides management support to two SNP Programs: Radioisotope Power Systems and the SP-100 Project. Activities in support of DOE's Radioisotope Power Systems Program are the larger of the two DOE SNP Programs. In the area of the RTG system, M&C Division provides managerial oversight of activities performed throughout Energy Systems. These activities include production of critical components for NASA's Cassini mission to Saturn, scheduled for launch in 1997, and materials development.

Two primary production activities are performed at Energy Systems. The first is the manufacture of the iridium-alloy clad vent sets that contain the heat-generating radioisotopes used in the RTG system. This manufacturing activity requires the integration of the capabilities of ORNL and the Y-12 Plant; ORNL produces iridium-alloy blanks and foils for subsequent assembly by the Y-12 Plant into clad vent sets. In addition to the manufacture of the iridium-alloy components, the M&C staff produces CBCF thermal insulators (sleeve and disc) that minimize temperature changes at the surface of the iridium-alloy cladding during off-normal operations of the generator. The production of blanks, foil, and clad-vent-set hardware will be completed by the end of FY 1994.

Materials developmental activities and maintenance of production capabilities for future missions continue to be important tasks paralleling manufacturing. These activities include development and characterization of advanced iridium-based alloys with superior performance for the clad-vent-set or cladding application, design and qualification of a high-temperature carbon-composite material with enhanced kinetic energy absorption capabilities, development of cost-effective methods to refine large quantities of iridium scrap with M&C's existing EB-melting facility, and validation of high-temperature thermal conductivity of current and improved CBCF insulations.

The SP-100 Project was established to develop a nuclear reactor to provide long-term power for space applications. A technology assessment has been performed, and a ground testing phase, in which hardware is built and evaluated, is currently under way. The principal focus for ORNL has been the determination of mechanical properties, characterization of irradiation effects, and evaluation of the compatibility of specific refractory-metal alloys under anticipated SP-100 system operating conditions. Further, M&C also provides managerial oversight for the characterization of materials and their subsequent fabrication into large radiation-shield components, which are performed at the Y-12 Plant. SP-100 Project closeout began in September 1993 and will be completed during 1994.

### **6.5.2 Non-DOE-Sponsored Programs — *R. H. Cooper***

The Special Projects Office provides coordination of division activities supported by 31 different organizations, including private industry, other national laboratories, NASA, EPA, Army, Navy, and Air Force. Support provided by the Special Projects Office includes administration, program development, and technical program management. Through the years, these activities have provided an important means to enhance and leverage existing materials technologies or capabilities to the mutual advantage of DOE and our non-DOE sponsors. Examples of the scope of selected activities follow.

In support of the NASA Marshall Space Flight Center, the division is determining the mechanical properties of superalloys in a high-pressure hydrogen environment. ORNL is currently conducting a round-robin test program in order to establish acceptable test methods for high-temperature, high-pressure testing in hydrogen; these methods include tensile and fatigue properties and will be followed by all NASA contractors.

Work is also being performed for the U.S. Air Force Wright Patterson Laboratories in optimizing and scaling-up the CVI process for the fabrication of ceramic composites for advanced man-rated turbine engine components. This work complements efforts supported by the U.S. Air Force Office of Scientific Research to evaluate the nucleation kinetics of surface films deposited by chemical vapor methods.

Three diverse Navy projects continue in 1993. A project aimed at gaining a fundamental understanding of the development and distribution of residual stresses in welds is ongoing for the Office of Naval Research. Research on the nature of crack initiation and growth in ceramic deep-submergence vessels, utilizing alumina ceramics manufactured by WESGO and Coors Ceramics, has been completed for the Naval Command, Control, and Ocean Surveillance Center, RDT&E Division. Naval Air Warfare Center research continues on the development of SiC-reinforced  $Si_3N_4$  matrix composites currently being developed under Navy sponsorship at AlliedSignal Research and Technology. These coatings will be used to improve the environmental stability and mechanical properties of the AlliedSignal composite systems.

## **6.6 FOSSIL ENERGY PROGRAM — *R. R. Judkins***

The ORNL Fossil Energy Program Office, located in the M&C Division, manages research activities within the division and in several other divisions within ORNL. The focus of ORNL's Fossil Energy Program is on materials R&D, environmental analysis support activities, bioprocessing, coal combustion research, and modeling activities on the operational requirements of the Strategic Petroleum Reserve.

### **6.6.1 Fossil Energy Materials Program — *N. C. Cole***

The ORNL FEMP Office is a part of the M&C Division, and it is from this office that the activities of the program within the M&C Division and at other federal and industrial laboratories and universities are managed. Virtually all the materials research on the program within ORNL is performed in the M&C Division. The focused materials R&D

covers research on ceramics, new alloys, and mechanisms of corrosion. Materials technical support and failure analyses are provided to projects on the Clean Coal Technology Program. Transfer to industry of the technology developed on the program has been an important activity this year. Our strong commitment to technology transfer is reflected in an active CRADA involvement.

The ORNL Fossil Energy Program continued to have the responsibility for technical management with the DOE-ORO for the Fossil Energy AR&TD Materials Program. In addition to traditional structural materials R&D on ceramic composites and advanced alloys, functional materials (i.e., materials that possess properties important to performance of a specific function, such as solid-state electrolytes and inorganic membranes) have been integrated into the materials program. Fiber-reinforced ceramic composites with improved strength and toughness are being produced by forced CVI and deposition, a process developed at ORNL. Ceramic composites have a variety of applications in fossil energy systems, including high-temperature heat exchangers and hot-gas cleanup filters. Achieving the necessary densification for tubular ceramic composites for high-temperature heat exchangers, fabricated by the CVI process, often requires up to 200 h. Recent innovative modifications to the equipment have resulted in an increase of the inner temperature of the tubular component from about 500 to 700°C, resulting in a reduction in infiltration times to about 50 h. Work also emphasized understanding the nature of the fiber-matrix interface with the aim to develop interfaces that are resistant to oxidation and yet optimize the mechanical properties of the composites. In addition, we are developing protective overcoats or oxide matrices that are resistant to corrosion. Ceramic membranes for the separation of gases in high-temperature and hostile environments were developed and tested. Testing was completed on a total of 12 experimental ceramic membranes. Separation factors determined from the data are providing guidance for membrane development efforts, and the substantial database of flow data provides basic information needed for further development of the gas transport model. Development began on CFCMS, fabricated from fibers produced from petroleum and coal-derived pitches. The feasibility of MW-assisted CVI of ceramic preforms was explored.

ORNL is developing advanced austenitic alloys for use in fluidized-bed and pulverized coal combustion power plants. The objective of this work is to modify existing alloys so as to provide the mechanical properties needed in high-temperature, high-pressure, second-generation power plants. The exploratory high-temperature, low-cycle fatigue testing of modified 310 stainless steel was completed. The performance of the steel was found to be comparable to other stainless steels in terms of cyclic life versus strain range. Stress-rupture testing of the first heat of modified 310 stainless steel was carried to times exceeding 10,000 h, and outstanding strength continues to be observed at 871°C (1600°F). Stress-rupture testing of weldments in modified 310 stainless steel was extended to times exceeding 1000 h at 871°C (1600°F). The strength of weldments was found to exceed the strength of the base metal. Intermetallic alloys, primarily iron aluminides, are being developed for applications in which superior oxidation and sulfidation resistance and strength are required. Improved room-temperature ductility, resistance to hydrogen embrittlement, and fabricability were emphasized this year. An iron aluminum alloy containing a reduced aluminum content, designated FAP-Y, was found to not only retain much of the oxidation and corrosion resistance of previous alloys,

but also has a tensile elongation of 40% at room temperature. In notched impact testing, it has a high upper-shelf energy, approximately 250 J, and the DBTT temperature, 150°C, is an improvement over the higher-aluminum alloys. A Cr-Nb alloy developed on the FEMP for very high-temperature applications has outstanding strength but requires improvements in its oxidation resistance. A recently formulated high-strength Cr-Nb alloy showed the best high-temperature isothermal oxidation resistance of any of the developmental compositions examined to date.

Corrosion research centered on studies of the formation and breakdown of protective oxide scales, particularly in sulfur-containing atmospheres, and on the effect of environment on corrosion of iron aluminides. An understanding of the properties and characteristics of oxide scales is evolving. The results of oxidation tests at 1300°C on samples of an Fe<sub>3</sub>Al alloy, mechanically alloyed with 0.5% Y<sub>2</sub>O<sub>3</sub>, showed that the oxide addition increases the useful elevated-temperature strength of the alloy to better exploit its unique high-temperature oxidation properties.

Assessments of materials problems and of the needed research to solve those problems for a variety of fossil energy technologies continued to be a part of our activities this year. Materials failure analyses, a significant factor in the success of advanced clean coal technologies, continue to be conducted for the Pittsburgh Energy Technology Center.

We are committed to transferring the technology developed on the FEMP to industry and to others in the fossil energy community. ORNL has three licenses to produce iron aluminides in powder form (Ametek) and wrought form (Hoskins and Harrison). Several CRADAs have been signed or are under discussion. Cummins Engine Company and ORNL have established a CRADA to develop filters to remove carbon from the exhaust streams of diesel engines. This project, the Direct-Energy-Regenerated Particulate Trap Technology, involves two M&C Division technologies, CVD for producing ceramic composites and MW processing, both sponsored by the FEMP. The initial contribution of ORNL will be to determine the optimum coating composition and thickness for MW coupling, structural strength, and ability to survive multiple regeneration cycles. A CRADA with the 3M Company has been completed for the transfer to industry of a process for fabricating full-size ceramic composites using technology developed at ORNL. Researchers at ORNL and the University of Kentucky have filed an invention disclosure on a gas separation device, the CFCMS. This device appears to have numerous applications such as in pressure swing adsorption gas separation systems and upgrading of subquality natural gas. We are presently pursuing the transfer of this invention to several industrial firms.

#### 6.7 FUSION ENERGY MATERIALS PROGRAM — *E. E. Bloom*

The Fusion Energy Materials Program has three major points of focus: (1) development of reactor structural materials, (2) development of first-wall and high-heat-flux materials, and (3) development of ceramics for electrical applications. Within the Office of Fusion Energy, these efforts are supported by the Neutron Interactive Materials and the Plasma Interactive Materials Programs. The ORNL effort supports U.S. participation in the ITER as well as the ultimate objective of making fusion an economically competitive and environmentally attractive energy source.

In the structural materials program, the primary emphasis remains on qualification of austenitic stainless steels for the ITER and the development of low-activation ferritic steels, vanadium alloys, and ceramic composites (e.g., SiC/SiC).

Austenitic steels are the leading candidate for structural applications in the ITER because of their advanced state of development and commercial practice. In a collaborative program with the JAERI, we are investigating the effects of fusion reactor radiation damage levels on the engineering properties of these alloys. Central to this effort is the irradiation of these alloys in the HFIR with tailoring of the neutron spectrum to produce damage levels (i.e., transmutation-produced helium and dpa) equivalent to those produced in a fusion reactor spectrum. These experiments are providing data and understanding of radiation response at temperatures and damage levels that are precisely those required for the ITER Engineering Design Activity (i.e., 60 to 400°C, up to 30 dpa).

Development of low- or reduced-activation materials is critical to achieving fusion's potential as a safe and environmentally attractive energy source. Development of low-activation ferritic steels requires that metallurgically important elements such as Ni, Mo, Nb, and N be removed or reduced to relatively low levels and that potential impurity elements be controlled to acceptable levels. To develop low-activation martensitic steels, tungsten is being used as a substitute for molybdenum, and niobium is replaced by tantalum and vanadium. The development activities are focused on the most critical or limiting property of this class of alloys—the radiation-induced shift in DBTT and reduction of fracture toughness. The vanadium alloys that are being considered for fusion have attractive activation characteristics, so compositional modification is not required to achieve this goal. The focus of our research on vanadium alloys is chemical compatibility with proposed fusion coolants and the effects of irradiation on fracture toughness. From the viewpoint of induced activation, SiC is the ultimate fusion structural material. Monolithic SiC is not considered because of its fracture properties. SiC/SiC composites offer an approach to improved fracture toughness. Our understanding of the performance of these materials in an irradiation environment is extremely limited. The focus of our present research is to explore the effects of irradiation on properties so as to provide a basis for accurately assessing the potential of SiC/SiC composites as fusion structural materials and to begin efforts to tailor these materials for the fusion environment.

The effects of irradiation on the dielectric properties of ceramic insulators are of critical importance in the successful design and operation of numerous systems in a fusion reactor (e.g., RF heating and plasma diagnostics). Our initial experimental work (initiated in 1991) has been directed at in situ measurements of the loss tangent during ionizing and ionizing-plus-displacive irradiation. Results to date show an increase in loss tangent of nearly two orders of magnitude at a displacement rate of  $\sim 10^7$  dpa/s. A change of this magnitude will impact materials selection and design of RF heating systems for ITER. Measurement of in situ properties will be expanded to investigate radiation-enhanced dielectric breakdown and the effects of irradiation on structural evolution and mechanical properties.

Graphite and C/C research activities are part of the plasma interactive and high-heat-flux materials programs. Graphite and C/C composite materials are selected for these applications because their low Z number minimizes radiative heat losses from the plasma.

However, their application requires graphite and C/C composites with extremely good thermal shock, erosion, and neutron damage resistance. Optimum thermal shock resistance is assumed to be offered by appropriately designed C/C composites (i.e., selected fibers, matrices, and architectures). Current work is directed toward the optimization of these materials for neutron-damage resistance.

#### 6.8 HIGH TEMPERATURE MATERIALS LABORATORY USER PROGRAM — V. J. Tennery

This program includes two major components, the HTML User Program and the HTML Fellowship Program. The user program provides support for the staff of the six HTML User Centers for working with industrial and university users in conducting their research. The HTML is a modern research facility that houses an array of special instruments used to meet research needs for advanced materials, including structural ceramics and alloys. The research instruments in the six HTML User Centers provide a comprehensive set of tools for performing state-of-the-art determination of the structure and properties of solids. Current HTML User Centers include: Materials Analysis, Mechanical Properties, Physical Properties, XRD, Residual Stress, and Ceramic Machining. A key part of the HTML concept includes a staff of highly trained technical personnel who work with industrial and university researchers in this DOE-designated National User Facility. The user centers are organized to provide materials characterization support to appropriate university and industrial users and to research programs throughout the local DOE facilities. Support includes a wide range of involvement with research personnel such as (1) conducting research relating material properties to structure, (2) characterization of one-of-a-kind specimens, and (3) training qualified users and then providing them access to equipment to perform their own materials research. During this year, a seventh center, the CMC, was operating. This center is a partnership between DOE-EE, DP, and ER. This partnership center is described later in this report.

Table 1. HTML FY 1993 user experience

Type of user	Number of		
	Institutions	Individuals	User days
Industry	30	46	2695
University	20	47	1353
Local Oak Ridge users	<u>1</u>	<u>101</u>	<u>9666</u>
Totals	51	194	13,714

The industrial user days varied from 441 to 861 during a particular quarter, and university user days ranged from 175 to 409. An additional number of user days, ranging from 2027 to 3093, were accumulated by researchers employed at ORNL.

To date, 428 research proposals have been submitted to the user program by researchers from universities, industrial companies, and government laboratories other than ORNL. (ORNL research staff are not required to submit a research proposal; however, there is a fee charged to those researchers for use of the instruments.) A breakdown by user category of these proposals is given in Table 2. Access clearances have been initiated on more than 500 individual researchers who were listed as principal investigators on these proposals.

Table 2. Research proposals submitted July 1987 through September 1993 in High Temperature Materials User Program

User type	Research proposals submitted		
	July 87- Sept. 92	Oct. 92- Sept. 93	Total
Industry	126	47	173
University	177	44	221
Other goverment	5	3	8
<b>Total Non-proprietary</b>	<b>308</b>	<b>94</b>	<b>402</b>
<b>Proprietary</b>	<b>23</b>	<b>3</b>	<b>26</b>
<b>Grand Totals</b>	<b>331</b>	<b>97</b>	<b>428</b>

A listing of many of the major accomplishments in the HTM User Program during this reporting period is given here.

1. The first scanning thermal conductivity microscope in existence was installed in the PPUC. This microscope can simultaneously obtain and display a topographic and thermal conductivity image of the surface of a test specimen with a resolution of  $<1 \mu\text{m}$ . Tests have demonstrated the microscope's ability to thermally resolve 2- $\mu\text{m}$  pads of silicon oxide (thermal conductivity,  $K = 1.4 \text{ W/m K}$ ) spaced 1  $\mu\text{m}$  apart on a silicon substrate ( $K = 148 \text{ W/m K}$ ).

2. Capability to determine the thermal diffusivity of coatings has been developed by the PPUC staff. This new capability will greatly enhance the HTML's ability to study the effects of processing conditions, thermal history, and microstructure on the thermal conductivity of high-performance TBCs. The software was tested using a Rene 142 specimen with a plasma-spray zirconia coating and a free-standing plasma-spray zirconia coating supplied by GE-Aircraft Engines.
3. An initial study was conducted to determine the cylindricity and circular trueness of a cylinder liner from an 8V-92 production engine of the Detroit Diesel Corporation. Initial results indicated approximately 15 mm of form error near the top of the liner and <5-mm error at a depth of 5 cm from the top of the liner.
4. At the HTML User Group meeting, personnel from Coors Electronic Packaging in Chattanooga, Tennessee, made a presentation describing how work performed in the MAUC has been instrumental in improving solder seals and connections on electronic packages manufactured by Coors.
5. Research being conducted by personnel from Florida Tile Industries involves utilizing electron microscopes and other instruments in the MAUC to determine causes of defects in the tile body and glaze (localized discolorations and surface irregularities) in tiles produced in the Lawrenceburg, Kentucky, production facility. Minimization of these defects will result in improved productivity and competitiveness in the international tile market.
6. Work has been completed on a preliminary study involving the use of the Nanoindenter as a diagnostic tool in the investigation of component failure.
7. A workshop on measurement of residual stress by X-ray and neutron diffraction methods provided over 30 industrial, academic, and laboratory users and prospective users an opportunity to gain an in-depth understanding of micromechanics, measurement methods, and data analysis. The principal instructor was Dr. I. C. Noyan from IBM's T. J. Watson Research Center. Other contributors to the workshop were from the University of Denver, the University of Missouri, Los Alamos National Laboratory, and ORNL.
8. Caterpillar, Inc., in collaboration with the RSUC staff, has determined the diffraction elastic constants of PSZ deposited by plasma-spray techniques to determine if the stabilizers and/or the thermal spray method lead to differences in elastic properties. Neutron diffraction techniques are being tested as a possible faster, more accurate measurement method.
9. The RSUC facilities have been used in support of a Navy project to characterize the residual stresses that resulted from a series of grinding conditions of aluminum oxide. The residual stresses typically decreased as the grit size decreased. Stresses in typical flexure bars used for flexure strength testing were observed to be lower than the corresponding stresses in large plates ground in an identical manner.

10. Thermal conductivity of TBCs as a function of processing and thermal aging was studied by GE-Aircraft Engines. The TBCs are being developed to increase the efficiency and life of turbine engines.
11. Special procedures required to calibrate the depth-area relationship for the Nanoindenter diamond probe were applied and verified. The verification process involved the use of the AFM to directly measure the indent geometry. The resulting calibration curve relating depth to area is currently being applied to the calculation of hardness and elastic modulus of superconducting materials.

The 95th Annual Meeting of the American Ceramic Society was held in Cincinnati, Ohio, on April 18-22, 1993. Registration for this meeting was in excess of 5500. Over 50 of the many presentations were authored by HTML staff and/or participants in the user program.

**HTML User Exchange Group.** Present, past, and potential HTML users are invited to attend meetings which provide participants an opportunity to give advice on how the user program can be improved and made more effective.

On September 23, 1993, the fourth meeting of this group was held. Thirty researchers from the university and industrial community, the HTML staff members, and other researchers from ORNL attended this meeting. This number included 18 researchers from 5 universities and 12 researchers from 8 companies. The following technical presentations were made:

*Creep Rate and Stress-Rupture Properties of NT154 Si<sub>3</sub>N<sub>4</sub>,* M. N. Menon, AlliedSignal Engines

*Use of Cobalt and Nickel as a Barrier to Diffusion of Nickel Through Gold,* W. K. Baxter, Coors Electronic Package Company

*Thermophysical Properties and Applications of [NZP] Materials,* T. B. Jackson, LoTEC, Inc.

*Measurement of an Elastic Constant in Plasma-Sprayed Zirconia Coatings Using X-Ray Diffraction,* J. L. Bjerke, Caterpillar, Inc./T. Watkins, HTML

## 6.9 CERAMIC MANUFACTURABILITY CENTER

This year has been one of continued creation and startup for the CMC. The CMC evolved from a partnership of the EE, ER, and DP programs of DOE. This partnership program, called "Cost-Effective Machining of Ceramics," has continued to gain momentum. ORNL and EE made laboratory and office space available in the HTML for the CMC, and EE provided capital funds for the purchase of a creep feed grinder. ER funds currently support a CRADA with Brown and Sharpe Co. that provides a state-of-the-art coordinate measuring machine (CMM) in the CMC. DP provided ceramic CRADA support funds for seven CRADAs signed so far, with three more approaching final approval. The equipment from the last of the ten laboratories to be moved was relocated

in 1993. The CMM was delivered and installed in February 1993, and the creep feed grinder was delivered and installed in May 1993.

Since the stated objective of the CMC is "to develop, in conjunction with U.S. industry, advanced technologies and associated scientific concepts necessary to significantly reduce the cost of machining structural ceramics, with an initial focus on heat engine components," the efforts undertaken in the CMC are very much industry driven. This cooperative effort with industry will influence the types of equipment installed in the CMC at any given time. This industry influence is a major factor in the forthcoming installation of a new state-of-the art centerless grinder provided as an in-kind CRADA contribution by Cincinnati Milacron. This grinder will be utilized in developing new manufacturing processes for axially symmetric ceramic components, including engine valves.

Computerization of the CMC was initiated early in 1992 with the procurement of computer-aided design/computer-aided manufacturing equipment and software. The facility also provides extensive computer modeling and graphical capabilities utilizing a SiliconGraphics workstation, Macintosh computers, and IBM-compatible computers. All of the capabilities and equipment are linked together through a DECNet/EtherNet network such that a true "Art-to-Part" process can be followed in the development of manufacturing processes for lower-cost ceramic components.

Presentations on the mission and creation of the CMC have been made to numerous potential industrial CRADA partners, to the Industrial Diamond Association, at a Society of Manufacturing Engineers seminar, and to various DOE representatives.

## 6.10 HTML FELLOWSHIP PROGRAM

This program provides funds for supporting industrial researchers and U.S. citizen graduate students for conducting their research in the HTML. Research was performed by two industrial fellows, one faculty fellow, and one graduate fellow during this year. Three new graduate fellows were selected, and their appointments begin in early FY 1994. One of the industrial projects and the faculty project were completed. The other appointments are ongoing.

### 6.10.1 Industrial Fellowships

The first industrial fellow, Dr. Suzanne Raebel from Cummins Engine Company, completed a 3-month appointment in April 1993. Through her research efforts, she had the opportunity to learn about surface analysis techniques with which she was not familiar. The research she performed was useful to Cummins' diesel engine research in analyzing the surface layers of fuel system components, the surfaces of camshaft lobes, the oxide layers on cylinder liners, the chromium-plated surface of a valve stem, and the indium diffusion through L10-connecting rod bearings that exhibited various degrees of wear.

The second industrial fellow is T. Barrett Jackson, co-founder and Vice President of Manufacturing, LoTEC, Inc. (Utah). At LoTEC, he coordinates manufacturing processes and acquisition of facilities and equipment, and conducts R&D for new products and

manufacturing processes. Before LoTEC was formed in 1992, Jackson was employed by Ceramatec, Inc., in Salt Lake City. There he was involved with research projects concerning processing and characterization of SiCAION ceramics, new low thermal expansion structural ceramics, and high-thermal-conductivity sintered aluminum nitride. While earning his M.E. in Engineering from the University of Utah Department of Materials Science and Engineering in 1992, Mr. Jackson was a participating researcher in the HTML User Program. A summary of his research project to date follows.

Thermo-Physical Properties of [NZP]-Type Ceramics. The unique crystal structure of the [NZP] family of ceramic materials ( $\text{NaZr}_2\text{P}_3\text{O}_{12}$  and its isostructural analogs) allows almost unlimited atomic substitution. The substitution of elements at the various lattice positions has a profound effect on thermal expansion, thermal expansion anisotropy, phase-transformation temperature, melting point, and high-temperature strength. The goal of the research being done in the HTML User Centers is to gain a better understanding of the thermal-physical properties of this class of ceramics and make direct comparisons of chosen compositions. To this end, the following has been completed during the first quarter of this project.

Utilizing equipment in the CMUC, CTE specimens were made by grinding rectangular bars to a precise length with parallel end faces. This was necessary to ensure accurate measurements using the dual push rod dilatometer in the PPUC. Work is continuing on producing large numbers of flexure-strength specimens that will be used to determine the flexural strength and Weibull modulus of the various compositions after being subjected to repeated thermal cycling. The growth of microcracks or microcrack healing will affect the strength of the ceramic and should be a function of thermal cycle temperature and thermal expansion anisotropy.

The thermal stability of these ceramics will be investigated in two ways. One, the change in thermal expansion characteristics after repeated thermal cycling will be determined by measuring the CTE of as-sintered or hot-pressed specimens and then subjecting the specimens to a number of thermal cycles followed by again measuring the thermal expansion. Changes in absolute expansion and amount of hysteresis will be determined. To ensure accuracy, multiple specimens are measured. Currently completed are measurements of as-sintered specimens and those exposed after one cycle to 1230°C. Changes in thermal expansion and hysteresis have been noted, especially in highly anisotropic compositions. The changes may be attributable to microcrack healing at these temperatures. Flexural-strength measurements and electron microscopy will help determine the validity of this assumption. The second method of determining stability will be the measurement of flexural strength. When all of the test specimens have been completed, thermal cycling will commence followed by testing. Already completed is the measurement of flexural strength and Weibull modulus for two as-sintered compositions. This establishes a baseline for further testing.

The thermal conductivity of these ceramics, especially at high temperatures, will be extremely important when being considered as high-temperature insulation. During this quarter, the room-temperature thermal diffusivity of all the compositions has been determined. High-temperature thermal diffusivity measurements have been made on one composition. The heat capacity as a function of temperature has been determined for

half of the compositions under investigation. Coupling the thermal diffusivity with the heat capacity of the material, the thermal conductivity as a function of temperature can be calculated.

This first quarter completes most of the groundwork for the next quarter of work. When all of the test specimen preparation is complete, the thermal stability experiments will commence followed by physical and mechanical property measurements. These measurements in comparison to the initial measurements will help determine the thermal stability of these ceramic materials.

#### **6.10.2 Faculty Fellowships**

The first faculty fellow, Dr. A. Datye from the University of New Mexico, completed a 3-month appointment in August 1993. Dr. Datye utilized the HRTEM and SEM facilities in the HTML MAUC to provide better understanding of the catalysts used for automotive exhaust control, hydrocarbon processing, and as combustion catalysts. Some of the catalysts investigated are used on an industrial scale at AlliedSignal, Ford Motor Company, and at Texaco. Dr. Datye received a Ph.D. in chemical engineering from the University of Michigan, Ann Arbor, in 1984. He has also been a participating researcher in the HTML User Program prior to becoming a faculty fellow. He conducted research on several problems, including using electron holography of hollow nanoscale Pd particles, deactivation mechanisms in automotive catalysts, hysteresis in chemisorption on Pd/SiO<sub>2</sub> catalysts, and interaction of Rh with CeO<sub>2</sub> in auto exhaust catalysts.

#### **6.10.3 Graduate Fellowships**

The first graduate fellow, Mr. Alex Cozzi of the University of Florida, performed research on his Ph.D dissertation on MW joining of alumina using sol-gel techniques.

Three new graduate fellows were appointed and will start research in the HTML in early FY 1994:

Mr. A. Haynes, University of Alabama  
Mr. R. Ott, University of Alabama  
Mr. D. Taylor, University of Arizona

### **6.11 DEFENSE PROGRAMS TECHNOLOGY TRANSFER INITIATIVE — *P. Angelini***

The DP Technology Transfer Initiative is based on the dual-use concept of enhancing the competitiveness of U.S. industry and the capabilities of the DP activities. There are five main focus areas in the technology transfer initiative, including materials and materials processing, manufacturing, energy and environment, microelectronics and photonics, and computing. During this past year, proposals have been submitted with the Y-12 Plant in one call for proposals. The initiative utilizes the CRADA mechanism, thus enabling close cooperation between the industrial partner(s) and Energy Systems. The proposals contain strong industrial components, tasks, and commitments of in-kind funding and are focused on technical issues driven by the industrial partners. Work that has been successfully funded and initiated relates to materials, materials process development, materials and process modeling, and computing.

## 7. COLLABORATIVE RESEARCH FACILITIES AND TECHNOLOGY TRANSFER

### 7.1 ORNL/ORISE SHARED RESEARCH EQUIPMENT PROGRAM — *E. A. Kenik and N. D. Evans*

The SHaRE Program allows participants from universities, industries, and other national laboratories access to the wide range of often unique microanalytical facilities within the M&C Division. The program promotes collaborative research in materials science areas pertinent to the USDOE and ORNL missions and emphasizes areas under current investigation within the M&C Division. Facilities and techniques included under the SHaRE Program are analytical and intermediate-voltage electron microscopy, APFIM, AES, nuclear microanalysis, XRD, ion implantation, and mechanical properties microanalysis. A number of SHaRE projects complement the advanced materials development programs in the M&C Division, such as advanced ceramics, ordered intermetallics, radiation effects, and austenitic alloys. During this period, special funding permitted the purchase of three advanced facilities for SHaRE: a Gatan post-column imaging filter for the acquisition of electron energy-loss maps, a new analytical FEG-SEM, and a new 200-keV analytical FEG-TEM to replace the aging Philips EM400T/FEG AEM.

The Division of Materials Sciences, Office of BES, provided funds through the ORISE to support the SHaRE activity. Program funds were used to reimburse travel and living expenses of university participants while at ORNL and for the support of Neal Evans, whose principal responsibilities included participating in various SHaRE projects and familiarizing SHaRE participants with the electron microscope and computer facilities. His presence as a liaison between the M&C Division research staff and the SHaRE participants allowed a high level of SHaRE participation with minimal interference with ORNL in-house programs.

A steering committee reviews all proposed SHaRE projects and defines SHaRE Program policy. The members in FY 1993 were: E. A. Kenik, ORNL; J. Bentley, ORNL; C. B. Carter, University of Minnesota; B. D. Fabes, University of Arizona; N. D. Evans, ORISE; and R. Wiesehuegel, ORISE.

During FY 1993, 19 of 24 approved SHaRE projects were active; six of the active projects did not require travel support. The active projects involved 37 outside participants (users), including 18 students. There were at least 33 papers based on SHaRE research published and approximately 37 presentations made at technical meetings. Currently, another ~20 papers based on SHaRE research have been accepted for publication, or are in press. Furthermore, this was a very fruitful period for the SHaRE Program in terms of graduate students whose participation in the program benefited their recently completed dissertation studies; five Doctor of Philosophy degrees were completed. Additionally, 15 more graduate students are progressing in their dissertation studies by participating in the SHaRE Program.

### 7.2 ORNL/ORISE SYNCHROTRON ORGANIZATION FOR ADVANCED RESEARCH — *C. J. Sparks*

The M&C Division X-Ray Research and Application Group, in collaboration with the Materials Research Laboratory at the University of Illinois, operates an intense X-ray

beamline at the NSLS to study the crystallographic structure of weakly scattering materials. Long- and short-range crystallographic structure and phase purity have among the most important influences on materials properties. This beamline has improved our capability to measure the weak scattering from local structural changes. Though heavily subscribed, this facility is open to outside users for qualified experiments. During this past year, different experiments were performed by 63 scientists from 7 different universities, 4 industrial, and 3 government laboratories. Sixteen published papers, including one *Physical Review Letters*, and one Masters and two Ph.D. theses were completed. Among the research highlights of this reporting period are the following:

1. Measurements of the structure-property relationships in Fe-base alloys relate the role of local atomic arrangements to many of their properties. Recent measurements on local pair correlations are providing new data to better understand the role of atomic size difference on phase stability. The prediction and understanding of phase transitions will advance our ability to tailor materials properties.
2. X-ray scattering from magnetic Dy/Lu superlattices and thin films related the structure and interfaces to the enhanced curie temperature.
3. Resonant X-ray scattering near absorption edges of heavy fermion uranium alloys revealed differences in the density of unoccupied 5f(7/2) and 5f(5/2) states and coupling of the ground state to these excited states. This is a promising new tool for the study of electronic properties.
4. Crystallographic characterization of the atomic positions in Al-Cu-Fe quasi-crystals has improved our understanding of their translational symmetry.
5. Measurements of the preferred orientation of high-temperature superconducting grains along a textured tape revealed the orientation change at a resolution of 0.1 mm. Critical current density was shown to be strongly influenced by the change in orientation.

An updated and more user friendly version of our software was installed which runs the computer-controlled beamline and data output. The annual meeting of ORSOAR users was held in May 1993 at BNL following the NSLS User's Meeting. Again, discussion centered on the need for more funding for the beamline to increase the efficiency of our experiments and to increase support for ORSOAR users. Increased funding of ~\$50,000 per year would make a marked improvement in our research capacity.

### 7.3 TECHNOLOGY TRANSFER — J. R. Weir, Jr.

Substantive activities this year involved providing technical assistance to current and potential licensees, negotiating collaborations to further develop ORNL technologies, and providing information on technologies through oral presentations and written communication. There were no new licenses signed this year. However, there were 14 new CRADAs signed.

To date, the 29 licensees to the M&C Division technologies are:

1. Advanced Innovative Technologies\*; American Matrix; Advanced Composite Materials; Cercom, Inc.; Dow Chemical; GTE; Hertel; High Velocity; Inland Industries\*; Iscar; Kennametal; and Keramont Corporation (SiC whisker-reinforced ceramics);
2. Instron (ceramic gripper assembly for tensile testing);
3. Ametek; Armada Corporation (Hoskins); Armco, Inc.; Cummins Engine; Harrison Alloys; Metallamics; and Valley Todeco (Ni<sub>3</sub>Al alloys);
4. 3M (novel ternary ceramic alloy);
5. Coors Ceramics (gelcasting method of making complex ceramic shapes);
6. Ametek; Cast Masters\*; Harrison Alloys; and Hoskins Manufacturing Company (iron aluminides);
7. Microscience (atom-probe software/field-ion microscope);
8. Sigma Tech\* (ultralight electromagnetic shielding); and
9. Microwave Laboratories\* (variable-frequency microwave furnace).

Of the royalties from licenses of Energy Systems technologies in FY 1993, over half were from M&C Division patents.

The ASM International Technology Transfer Committee (chaired by a staff member) consists of 22 members of federal and national laboratories.

Following is the status of the 28 CRADAs in the M&C Division of which 14 were signed in FY 1993:

<u>Client</u>	<u>Technology</u>	<u>CRADA approved</u>
Eaton & Johnson Controls	Shape Memory Alloy	06/13/91
Garrett (Allied Signal)	Microwave Annealing of SiN with High Additive Content	08/22/91
Norton Company	Microwave-Sintered Reaction-Bonded SiN	09/05/91
Norton Company	Microwave Annealing of SiN with Low Additive Content	09/05/91
Appliance Research Consortium	Powder-Evacuated Panel	07/22/91

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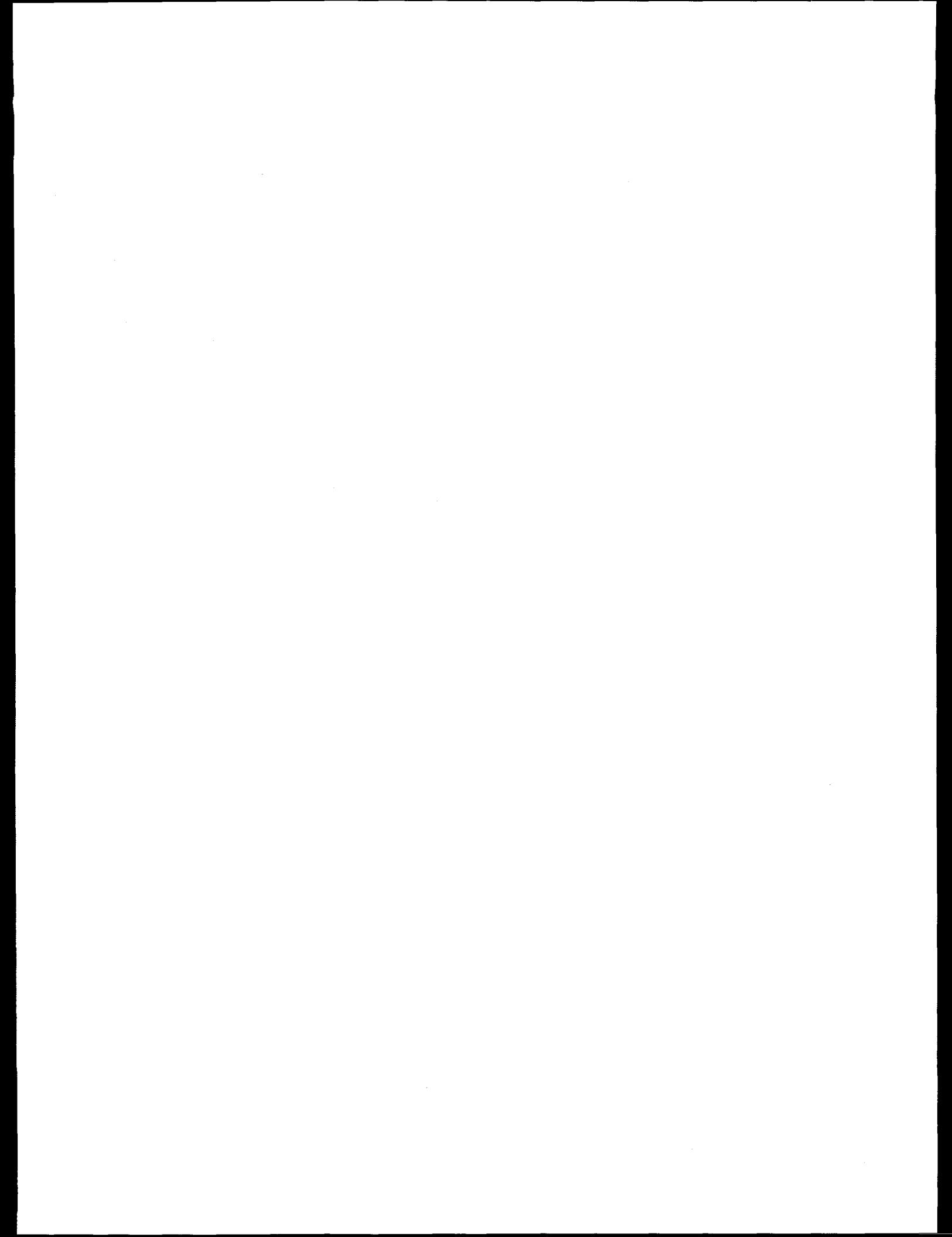
\*Licenses signed in FY 1992.

<u>Client</u>	<u>Technology</u>	<u>CRADA approved</u>
Microwave Labs	Wideband Microwave Processing Equipment	11/04/91
AVX Tantalum Corporation	Microwave-Sintering Tantalum Capacitors	11/04/91
3M Company	Chemical Vapor Infiltration of Ceramic Composites	02/03/92
General Motors	Alloy Heat-Treating Furnaces	02/03/92
Coors Ceramics	Machining and Inspection of Ceramics	02/18/92
PPG Industries	Powder-Evacuated Panel	06/24/92
Detroit Diesel	Machining Ceramics	08/26/92
Coors Ceramics	Gelcasting Ceramics	09/28/92
Metallamics	Nickel Aluminide Rolls	10/08/92
General Motors	Lightweight Materials	10/08/92
DuPont	Heat Pump System Modelling	09/26/92
Lanxide	Machining Ceramics	10/08/92
Concurrent Technologies	Weld Process Model	10/08/92
Dow	Microwave Silicon Carbide	05/06/93
DuPont	Cladding for PEP Insulation	09/30/93
Pratt & Whitney	Ceramic Bearings	09/30/93
AlliedSignal	Microwave Silicon Nitride	04/06/93
CIMA/TTU	Data Base Insulation	03/02/93
Dow	Wear Silicon Nitride	08/30/93
AlliedSignal	Wear Silicon Nitride	04/13/93

<u>Client</u>	<u>Technology</u>	<u>CRADA approved</u>
Caterpillar	Machining Ceramics	09/13/93
IBM	Modeling Magnetic Data Storage	09/14/93
Pratt & Whitney	Titanium Intermetallics	09/30/93

Thirteen invited technology transfer presentations were made describing 20 M&C Division technologies. The technology transfer exhibit was shown at seven meetings. We responded to over 100 inquiries for information and sample materials.

A state outreach initiative was funded in FY 1993 to utilize the technology transfer concepts developed in a previously funded DOE project called the University-Laboratory-Industry Technology Brokerage System. The project involved visits to two selected states (Vermont and Oklahoma) to present seminars and workshops to state economics development and community college officials to develop state networks to access the technology at federal laboratories on behalf of small companies.



## 8. EDUCATION PROGRAMS

*L. L. Horton*

The M&C Division's involvement in educational activities continues to grow. In FY 1993, there were more than 150 "paid" guests (~25 professors, 2 high school teachers, over 88 graduate/undergraduate students, and ~ 37 postdoctoral fellows) in the division. These personnel are brought into the division by a host of programs coordinated by the ORISE and the Southeastern University Research Association, by the ORNL co-op program, and under university and personal services subcontracts. In addition, ~3000 pre-collegiate students and over 350 pre-college teachers participated in programs sponsored or co-sponsored by M&C. The involvement with these students ranged from on-site tours to hands-on research experiences for pre-college students and teachers.

About 375 pre-college students and over 60 teachers actually toured division facilities. These tours included presentations about materials science and hands-on demonstrations of electron microscopy (most participants operated an SEM and took a micrograph), ceramic processing, and superconductivity. School outreach programs involved nearly 2000 students and 125 teachers. Other major educational outreach programs included "Science in Action," the UT Academy for Teachers of Science and Mathematics, the DOE-Appalachian Regional Honors Workshop, and the National Junior Science and Humanities Symposium.

The Science in Action Program is a 3-day multidisciplinary program held during Engineer's Week in February. It is affiliated with the WATTeC conference and involves local technical professional societies. M&C provides one of the co-chairmen, several of the speakers, and exhibits. Over 625 students and over 110 teachers participated. M&C-sponsored presentations included "Fun with Materials" and "What is a Scientist/Engineer?" as well as exhibits on microscopy.

The largest single effort was the ORNL High School Honors Workshop, co-sponsored by DOE and the Appalachian Regional Commission. The Honors Workshop, co-hosted by M&C and the Environmental Sciences Divisions, was a 2-week research experience for 110 high school students and 8 teachers. Forty of the students and all of the teachers were located in M&C for the program. The student activities included research projects, theme sessions focussed on materials-environmental issues, and diverse social activities. During the second week of the program, the students wrote a short report of their research and gave an oral presentation of their results. The research topics in M&C were:

- "Laboratory Growth of the Hardest Material in the World: Diamond"
- "Microwave Processing of Ceramics"
- "Mechanical Properties of Materials"
- "Exploring the Nature of the Chemical Bond: Computing the Structure of Atoms and Molecules"
- "Damage-Tolerant Ceramics"

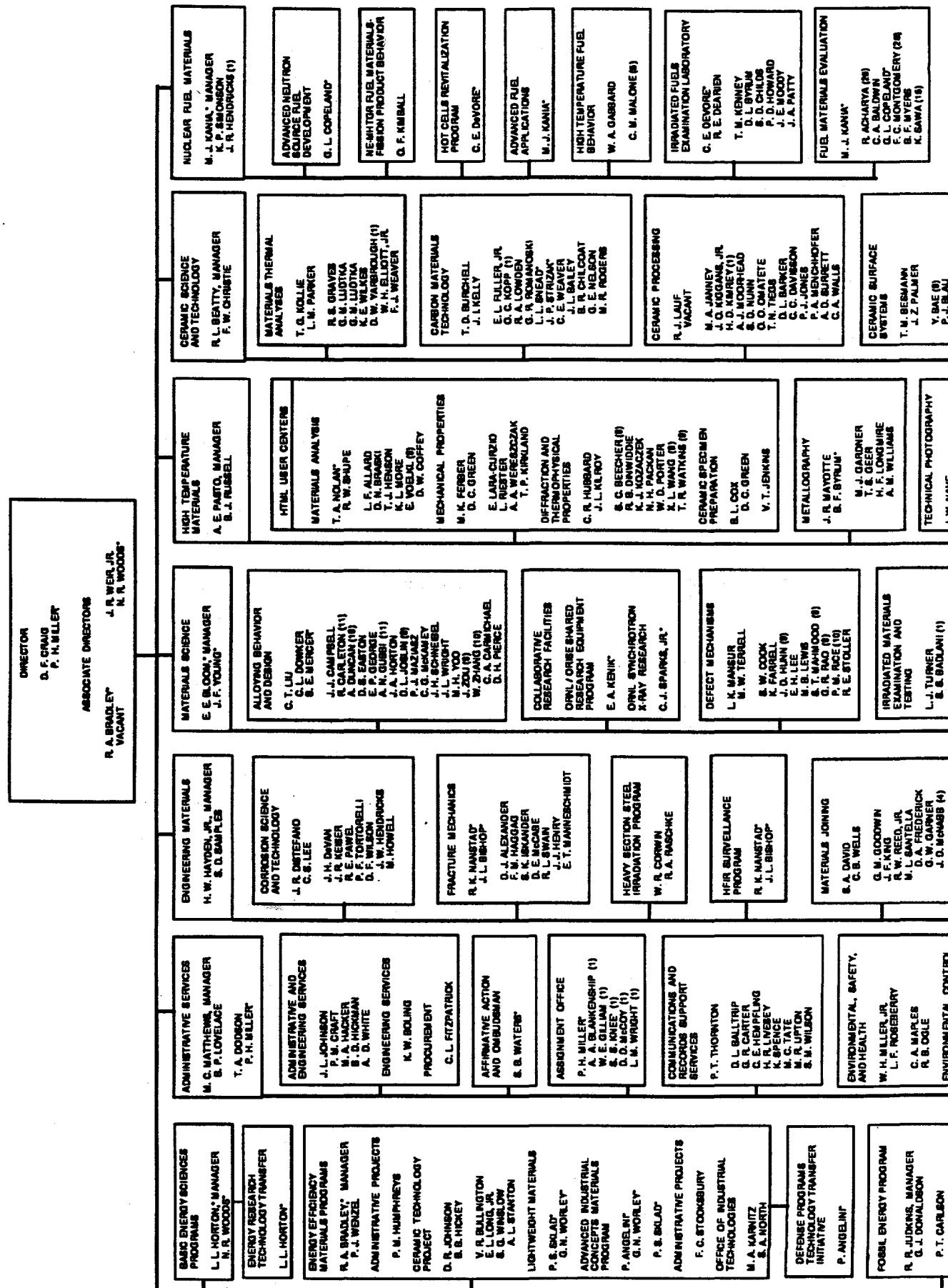
- "Metals Processing – Melting, Rolling, and Testing of Aluminum Alloys"
- "Near - Net-Shape Processing of Engineering Ceramics: The Next Generation"
- "High-Tc Superconductors: Synthesis, Processing, and Characterization"
- "Correlation of Microstructure and Heat Flow Characteristics in Microwave-Processed Ceramics"
- "The Microscopic World: Characterization of a Microwave-Annealed Ceramic Using Various Microscopy Techniques"

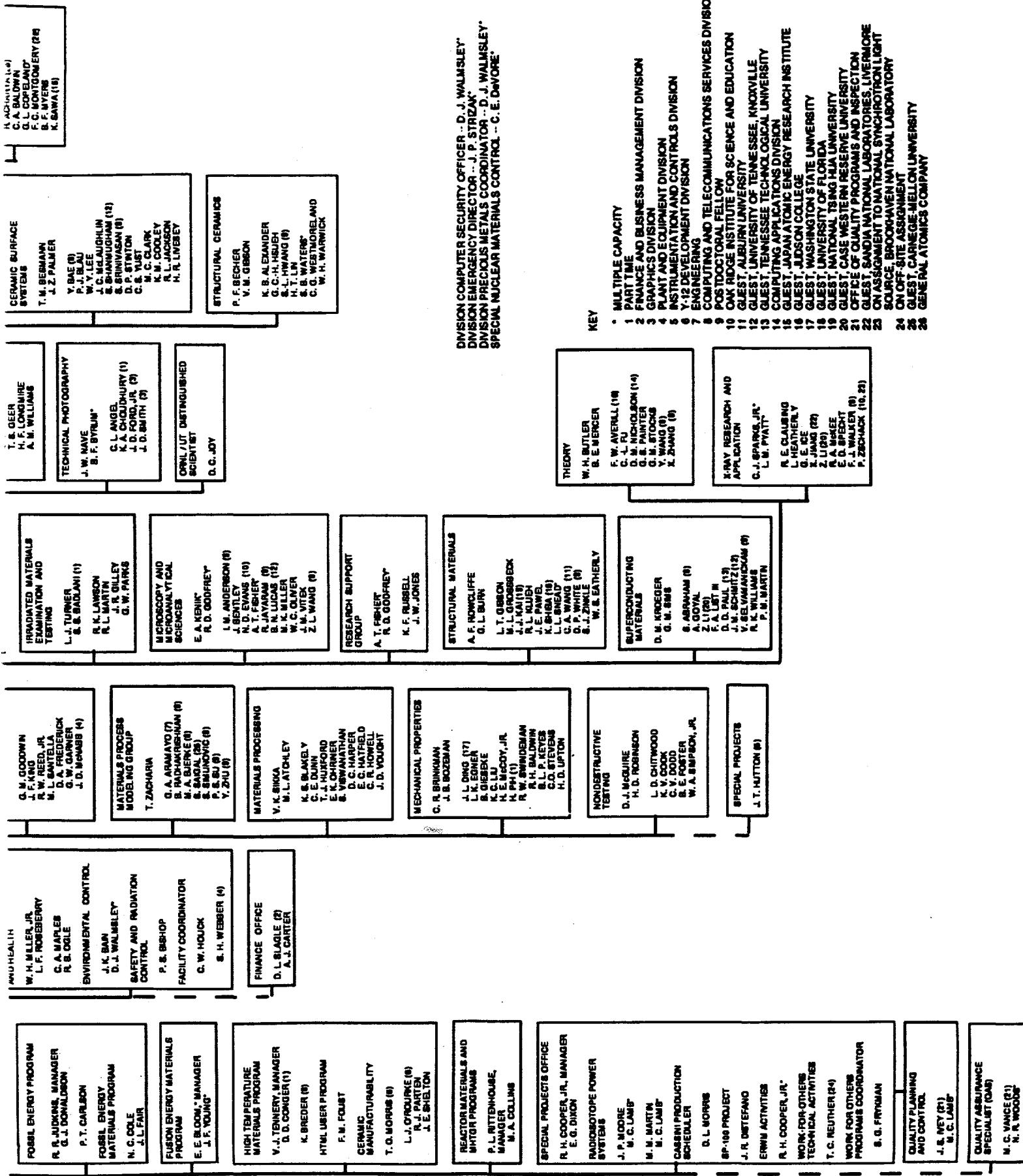
M&C staff are also involved in outreach activities focussed on women students. We participate in the ORNL Women in Science Program for college-age and pre-college women (presentations and tours) and in the "Sharing Adventures in Engineering and Science" Program for middle school girls (off-site presentations).

Appendix A

METALS AND CERAMICS DIVISION

HOMEWORK 1 189





## Appendix B

### PERSONNEL SUMMARY

January 1, 1993, to December 31, 1993

*Compiled by Barbara Lovelace*

#### Scientific Staff

##### A. New Staff Members

A. Goyal	Superconducting Materials Group
K. J. Kozaczek	X-Ray Diffraction & Physical Properties Group
E. Lara-Curzio	Mechanical Properties Users Group

##### B. Retirements/Terminations

G. E. C. Bell	Voluntary Resignation
O. B. Cavin	Retirement
D. R. Childress	Voluntary Resignation
L. C. Emerson	Retirement
F. M. Foust	Retirement
R. L. Heestand	Retirement
R. N. Hengstler	Retirement
E. H. Lee	Retirement
M. Payne	Retirement
J. M. Robbins	Retirement
T. K. Roche	Retirement
L. G. Shrader	Retirement
G. M. Slaughter	Retirement

##### C. Transfers

P. E. Arakawa	Transferred to Waste Management & Remedial Action from Hot Cells Revitalization Program
G. L. Bell	Transferred to Fusion Energy Division from Fuel Materials Testing Group
A. Bleier	Transferred to Chemical Technology Division from Structural Ceramics Group

R. A. Buhl	Transferred to Robotics & Process Systems Division from Radiation Effects in Materials Science Group
B. L. Cox	Transferred to Y-12 Quality Division from Ceramic Specimen Preparation Group
D. O. Hobson	Transferred to Engineering Technology Division from Reactor Materials Programs
J. A. Horak	Transferred to K-25 Technical Division from Mechanical Properties Group
J. D. Lentz	Transferred to Fusion Energy Division from Hot Cells Revitalization Program
C. M. Malone	Transferred to Instrumentation & Controls Division from High Temperature Fuel Behavior Group
R. C. Martin	Transferred to Chemical Technology Division from Fuel Performance Modeling Group
J. H. Miller	Transferred to Chemical Technology Division from Ceramic Surface Systems Group
R. N. Morris	Transferred to Computing Applications Division from High Temperature Fuel Behavior Group
D. L. Moses	Transferred to Advanced Neutron Source Project from NPR Project Office
D. M. Nicholson	Transferred to Computing Applications Division from Theory Group
J. T. Parks	Transferred to Chemical Technology Division from Fuel Materials Testing Group
D. F. Pedraza	Transferred to K-25 Technical Division from Carbon Materials Technology Grou
O. M. Stansfield	Transferred to K-25 National Security Program from Fuel Performance Modeling Group
R. D. Taylor	Transferred to Chemical Technology Division from Fuel Materials Evaluation Group

#### **Administrative and Technical Support Staff**

##### **A. Retirements/Terminations**

D. A. Ellis	Voluntary Resignation
M. L. Hodges	Voluntary Resignation
N. H. Rouse	Retirement
M. D. Threat	Retirement

##### **B. Transfers**

C. W. Boggs	Transferred to Waste Management & Remedial Action from Fuel Materials Evaluation Group
K. C. Brunson	Transferred to Office of Environmental Compliance & Documentation from Conservation Materials Program
C. L. Cheverton	Transferred from Space & Defense Programs to Central Management Organization

J. M. Cole	Transferred to Chemical Technology Division from Research Support Group
R. E. Dearien	Transferred from Engineering Technology to High Temperature Materials Section
K. R. Grubb	Transferred to Office of Operational Readiness & Facility Safety from Administrative Services Section
P. D. Hughes	Transferred to Chemical Technology Division from High Temperature Fuel Behavior Group
J. R. Lowe	Transferred to Y-12 Office of Legal Counsel from Ceramic Surface Systems Group
J. E. Moody	Transferred from Plant & Equipment Division to Irradiated Fuels Examination Laboratory
A. E. Parker	Transferred to Waste Management & Remedial Action from Irradiated Materials Examination & Testing Group
M. W. Terrell	Transferred to Biology Division from Radiation Effects in Materials Science Group

#### **Co-Op Assignments**

A. M. Abeel	Virginia Polytechnic Institute and State University
A. Arora	Alfred University
N. V. McAdams	Virginia Polytechnic Institute and State University
K. M. Ploetz	Alfred University
D. C. Schenk	Tennessee Technological University
O. J. Schwarz	Tennessee Technological University
R. E. Simpson	Georgia Institute of Technology
M. W. Stott	Tennessee Technological University
M. D. Teske	Georgia Institute of Technology
N. L. Vaughn	Tennessee Technological University

#### **Summer Assignments (1993)**

##### **A. Summer Research Interns**

M. L. Auger	Alfred University
T. M. Beavers	University of Tennessee
T. Chang	University of Michigan
J. A. Cook	University of Tennessee
K. L. Ploetz	Alfred University
M. J. Swindeman	University of Tennessee

##### **B. Administrative Support Staff**

J. D. Baker	Lincoln Memorial University
V. M. Brawner	Tennessee State University
D. A. Hargreaves	Tennessee Technological University

J. L. Houston	East Tennessee State University
T. L. Karnes	Lincoln Memorial University
K. M. Marsh	University of Tennessee
D. D. Neal	Roane State Community College
J. E. Nunn	University of Tennessee
S. R. Odom	Roane State Community College
B. D. Reed	Roane State Community College
L. A. Reid	Samford University
S. M. Stout	University of Tennessee

### Guest Assignments

#### A. Scientific Staff

R. Acharya	General Atomics
J. D. Allen	Midwest Technical
W. R. Allen	Midwest Technical
N. M. Atchley	Midwest Technical
F. W. Averill	Judson College
B. P. Bandyopadhyay	University of North Dakota
G. E. C. Bell	M. J. Schiff & Associates, Inc.
M. J. Bennett	Consultant
D. A. Bolce	Midwest Technical
A. Boltax	Consultant
E. S. Bomar, Jr.	Consultant
T. A. Burtseva	D. V. Efremov Research Institute of Electrophysical Apparatus, Russia
O. B. Cavin	Consultant
K. K. Chawla	New Mexico Institute of Mining & Technology
B. A. Chin	Auburn University
W. A. Coghlan	Grand Canyon College
C. Cooperrider	Hitachi Scientific Instruments
J. L. Ding	Washington State University
W. P. Eatherly	Consultant
N. D. Evans, III	Oak Ridge Associated Universities/ORISE
R. M. Evans	Consultant
W. H. Farmer	Consultant
J. S. Faulkner	Florida Atlantic University
J. B. Flynn	Consultant
R. J. Gray	Consultant
T. M. Gray	Gilbert Commonwealth
J. C. Griess	Consultant
D. M. Griffith	Hitachi Scientific Instruments
G. Y. Guo	SERC, Daresbury Laboratory, UK
B. L. Gyorffy	University of Bristol, UK
R. W. Harrison	Consultant
C. W. Holland	Consultant

H. S. Hsu	Innovative Materials Technology Company
D. C. Joy	University of Tennessee
J. J. Kai	National Tsing Hua University, Taiwan
B. T. Kelly	Consultant
P. G. Klemens	University of Connecticut
J. I. Koike	Oregon State University
F. W. Kutzler	Tennessee Technological University
E. H. Lee	Consultant
T. B. Lee	United Energy Services Corporation
B. C. Leslie	Consultant
W. D. Manly	Consultant
M. Z. Martin	University of Tennessee
R. W. McClung	Consultant
D. L. McElroy	Consultant
C. J. McHargue	University of Tennessee
J. Miltenberger	Hitachi Scientific Instruments
F. Montgomery	General Atomics
A. V. Naberennkov	D. V. Efremov Research Institute of Electrophysical Apparatus, Russia
J. M. Okoh	University of Maryland Eastern Shore
S. Peterson	Consultant
G. M. Pharr	Rice University
F. J. Pinski	University of Cincinnati
W. T. Potter	ARC, Inc.
R. M. Prader	Graz University of Technology, Austria
N. Prince	Consultant
M. C. Rao	Consultant
D. W. Richerson	Consultant
J. A. Rifkin	University of Connecticut
S. Saigal	Carnegie Mellon University
S. Sarin	North Carolina A&T State University
K. Sawa	Japan Atomic Energy Research Institute
J. O. Scarbrough	Consultant
W. C. Schreiber	University of Alabama
L. B. Shaffer	Anderson College
K. Shiba	Japan Atomic Energy Research Institute
G. M. Slaughter	Consultant
G. D. W. Smith	Oxford University, UK
L. C. Smith	United Energy Services Corporation
M. N. Srinivasan	Texas A&M University
K. Suzuki	Hitachi Scientific Instruments
Z. Szotek	SERC, Daresbury Laboratory, UK
W. M. Temmerman	SERC, Daresbury Laboratory, UK
H. Ugachi	Japan Atomic Energy Research Institute
C. R. Vander Linden	Vander Linden & Associates
F. J. Walker	University of Tennessee
B. L. Weaver	The 3M Company
D. E. Wittmer	Wittmer Consultants, Inc.

E. H. Wood	Jeol USA, Inc.
N. Yamamoto	National Research Institute for Metals, Japan
Y. Ye	University of Tennessee
D. E. Zelmon	U.S. Air Force, Office of Scientific Research
P. Zschack	Oak Ridge Associated Universities/ORISE

#### B. Post-Doctoral Program

I. M. Anderson	University of Minnesota (ORAU/ORISE)
S. S. Babu	Penn State University
Y. Bae	Stevens Institute of Technology (ORAU/ORISE)
S. C. Beecher	University of Delaware (ORAU/ORISE)
K. Breder	Royal Institute of Technology, Sweden (ORAU/ORISE)
H. Cai	Ohio State University (ORAU/ORISE)
B. G. M. Frost	University of Tennessee
A. Goyal	University of Rochester (ORAU/ORISE)
J. D. Hunn	University of North Carolina (ORAU/ORISE)
S. L. Hwang	University of Michigan (ORAU/ORISE)
R. Jayaram	University of Pittsburgh (ORAU/ORISE)
X. Jiang	Sandia National Laboratory/Associated Western Universities
D. L. Joslin	University of Tennessee (ORAU/ORISE)
E. Lara-Curzio	Rensselaer Polytechnic Institute (ORAU/ORISE)
Z. Li	University of Tennessee
C. K. Lin	University of Illinois (ORAU/ORISE)
S. T. Mahmood	North Carolina State University (ORAU/ORISE)
B. Radhakrishnan	University of Alabama (ORAU/ORISE)
G. R. Rao	Auburn University (ORAU/ORISE)
P. M. Rice	Arizona State University (ORAU/ORISE)
J. L. Robertson	Sandia National Laboratory
V. Selvamanickam	University of Houston (ORAU/ORISE)
S. Simunovic	Carnegie Mellon University (ORAU/ORISE)
M. Sokolov	Moscow Institute of Atomic Energy (ORAU/ORISE)
S. Srinivasan	North Carolina State University (ORAU/ORISE)
P. S. Su	Georgia Institute of Technology (ORAU/ORISE)
E. Voelkl	University of Tuebingen, Germany (ORAU/ORISE)
X. L. Wang	Iowa State University (ORAU/ORISE)
Y. Wang	Florida Atlantic University (ORAU/ORISE)
Z. L. Wang	University of Tennessee
T. R. Watkins	Penn State University (ORAU/ORISE)
D. P. White	University of Connecticut (ORAU/ORISE)
C. Xu	Iowa State University (ORAU/ORISE)
W. Zhang	University of North Texas (ORAU/ORISE)
X. Zhang	University of Kentucky (ORAU/ORISE)
Y. Zhu	University of Leige, Belgium (ORAU/ORISE)
J. Zou	Washington University (ORAU/ORISE)

## C. Graduate Students

S. Abraham	University of Tennessee
A. Bolshakov	Rice University
M. Borst	University of Tennessee
M. Carballo	Florida International University
R. L. Carleton	Auburn University
N. Chawla	University of Tennessee
J. A. Cook	University of Tennessee
A. D. Cozzi	University of Florida
C. A. Dobson	Georgia Institute of Technology
A. J. Duncan	University of Florida
D. W. Graham	Virginia Polytechnic Institute & State University
D. J. Grellinger	University of Wisconsin
A. N. Gubbi	Auburn University
J. A. Horn	University of Alabama
T. B. Jackson	LoTEC, Inc.
D. L. Joslin	University of Tennessee
Y. Katoh	University of Tokyo, Japan
S. Khosla	University of Tennessee
K. S. Leshkivich	University of Tennessee
Y. Lin	Auburn University
B. N. Lucas	University of Tennessee
W. M. Matlin	University of Tennessee
P. Mehta	University of Tennessee
N. Miriyala	University of Tennessee
M. C. Osborne	Rensselaer Polytechnic Institute
J. R. Pate	University of Illinois
D. C. Patton	University of Alabama
D. D. Paul	Tennessee Technological University
L. M. Pike	University of Wisconsin
F. Rebillat	University of Bordeaux, France
J. M. Schmitz	University of Tennessee
S. Shammugham	University of Tennessee
J. B. Sipf	University of Tennessee
N. Sukidi	North Carolina State University
D. M. Walukas	University of Tennessee
C. A. Wang	Auburn University
X. Wang	Iowa State University
H. J. White	University of Tennessee

## D. Undergraduate Students

S. R. Agnew	Cornell University
E. J. Brinson	North Carolina A&T State University
J. M. Canon	University of Missouri
R. T. Collins	University of Tennessee
S. M. Duma	University of Tennessee

M. W. Golla	Penn State University
N. Gorey	Alfred University
L. G. Gunn	Washington State University
J. P. Maria	Penn State University
N. A. Moody	Allegany High School
M. W. Patout	University of Tennessee
I. N. Pedraza	Swarthmore College
C. L. Robinson	Tennessee State University
S. E. Russ	University of Tennessee
S. J. Vance	Penn State University
S. Vitek	Massachusetts Institute of Technology
C. A. Wijayawardhana	Denison University
M. R. Zeher	Virginia Polytechnic Institute & State University

**E. Science and Engineering Research Semester Program (SERS)**

V. K. Andleigh	Cornell University
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**F. Southeastern University Research Association (SURA)**

E. L. Hines	Florida Atlantic University
R. G. Jordan	Florida Atlantic University
Y. Liu	Florida Atlantic University

**G. Science/Math Action for Revitalized Teaching Program/Appalachian Regional Commission (SMART/ARC)**

L. E. Long	Lookout Valley Middle School
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**H. Teacher Research Associate Program (TRAC)**

L. T. Hixson	Cleveland High School
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**I. Project SEED (Summer Educational Experience for the Disadvantaged Program)**

C. E. Matos	Colegio Catolico Notre Dame High School, Puerto Rico
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**User Facilities**

**A. High Temperature Materials Laboratory (HTML)**

K. S. Ailey-Trent	North Carolina State University
M. K. Akhtar	University of Cincinnati
P. B. Aswath	University of Texas
F. D. Baldwin	University of Florida
A. Bandyopadhyay	University of Texas
W. K. Baxter	Coors Electronic Package Company

Y. Berta  
J. L. Bjerke  
R. P. Boisvert  
D. A. Bowers  
C. H. Cadden  
S. Cao  
G. Carrasquillo  
R. V. Carter  
R. H. Chand  
J. Chang  
K. K. Chawla  
N. Chawla  
T. P. Cheng  
C. G. Cofer  
A. D. Cozzi  
A. K. Datye  
J. R. Dick  
D. Dragoi  
S. H. Evans  
M. R. Foley  
B. G. Frost  
J. M. Ghinazzi  
R. E. Gonzalez  
S. T. Gonzcy  
L. P. Hehn  
W. E. Hollar  
A. T. Hunt  
T. B. Jackson  
D. W. James  
D. Y. Jang  
J. Jo  
C. R. Johnson  
D. L. Joslin  
P. Khandelwal  
C. A. Knepfler  
E. H. Liu  
M. S. Love  
M. D. Mello  
M. N. Menon  
C. S. Moore  
J. M. Moyer  
T. J. Mroz  
B. A. Nagaraj  
M. J. Nguyen  
J. R. Olive  
N. R. Osborne  
S. S. Park  
P. K. Predecki

Georgia Institute of Technology  
Caterpillar, Inc.  
Dow Corning Corporation  
McDonnell Douglas Corporation  
Allison Gas Turbine  
University of Tennessee  
Rutgers University  
Northrop Corporation  
Chand Kare Technology  
Allison Gas Turbine  
New Mexico Technological University  
University of Tennessee  
Applied Materials, Inc.  
University of Illinois  
University of Florida  
University of New Mexico  
Florida Tile Industries  
University of Denver  
Alpha Optical Systems  
Norton Company  
University of Tennessee  
Coors Technical Ceramics  
University of Kentucky  
AlliedSignal, Inc.  
University of Florida  
The Carborundum Company  
Georgia Institute of Technology  
LoTEC, Inc.  
Carbomedics, Inc.  
University of Missouri  
Concurrent Technologies  
University of Alabama  
University of Tennessee  
Allison Gas Turbine  
Northwestern University  
Ultramet  
Florida Tile Industries  
Quadrax Corporation  
AlliedSignal, Inc.  
General Electric Aircraft Engines  
Teledyne Allvac  
Advanced Refractory Technologies, Inc.  
General Electric Aircraft Engines  
General Motors Corporation  
Vanderbilt University  
University of Dayton Research Institute  
University of Tennessee  
University of Denver

B. H. Rabin	Idaho National Engineering Laboratory
S. P. Raebel	Cummins Engine Company
S. Raghuraman	University of Illinois
C. A. Randall	Penn State University
R. G. Rateick	AlliedSignal, Inc.
F. Reidinger	AlliedSignal, Inc.
G. N. Riley	American Superconductor Corporation
W. R. Russ	Virginia Polytechnic Institute & State University
P. G. Sanders	Northwestern University
F. A. Schwartzman	Brown University
S. Shanmugham	University of Tennessee
B. W. Sheldon	Brown University
R. J. Shinavaski	Clemson University
O. M. Spaldon	North Carolina State University
R. Srinivasan	University of Kentucky
N. Sukidi	North Carolina State University
S. E. Sund	AlliedSignal, Inc.
C. M. Sung	University of Massachusetts
D. J. Taylor	University of Arizona
W. T. Toreki	University of Florida
M. J. Tricard	Norton Company
X. L. Wang	University of Tennessee
J. R. Weertman	Northwestern University
P. J. Whalen	AlliedSignal, Inc.
J. S. Wolf	Clemson University
Y. Yang	Virginia Polytechnic Institute & State University
R. L. Yeckley	Norton Company
J. Y. Yung	Sundstrand Aerospace
X. Zhang	University of Tennessee
4 users	(Proprietary)

#### B. Shared Research Equipment Program (SHaRE)

I. Baker	Dartmouth College
M. G. Burke	Westinghouse Science & Technology Center
D. L. Callahan	Rice University
C. B. Carter	University of Minnesota
A. Castagna	Rensselaer Polytechnic Institute
T. M. Cheek	University of Florida
Y. L. Chen	North Carolina State University
P. Dewo	University of Florida
G. L. Edgemon	Georgia Institute of Technology
J. J. Hoyt	Washington State University
W. Jiang	Washington State University
M. J. Kaufman	University of Florida
P. G. Kotula	University of Minnesota
L. S. Lin	United Technologies-Pratt & Whitney
D. M. Maher	North Carolina State University

M. P. Mallamaci	University of Minnesota
S. McKernan	University of Minnesota
J. C. Morris	Rice University
M. G. Norton	Washington State University
W. H. Poisl	University of Arizona
C. J. Simon, III	University of Tennessee
G. Sundar	Washington State University
S. Tanaka	North Carolina State University
T. Y. Tsui	Rice University

C. Oak Ridge Synchrotron Organization for Advanced Research (ORSOAR)

R. Aburano	University of Illinois
G. Aeppli	AT&T Bell Laboratories
J. Arnold	University of Akron
H. Chen	University of Illinois
S. Z. D. Cheng	University of Akron
T. C. Chiang	University of Illinois
F. Chiu	University of Akron
K. Chung	University of Illinois
M. DeBoissieu	State University of New York, Stony Brook
W. Dmowski	University of Pennsylvania
M. Durbin	Northwestern University
P. Dutta	Northwestern University
T. Egami	University of Pennsylvania
B. Everitt	University of Illinois
S. Fu	University of Pennsylvania
B. Gaulin	McMaster University, Canada
R. Ghaskadvi	Northwestern University
R. Ho	University of Akron
H. Hong	University of Illinois
E. D. Isaacs	AT&T Bell Laboratories
N. Kishiragura	University of Illinois
K. Koga	University of Houston
S. W. Lee	University of Akron
L. Leibowitz	Argonne National Laboratory
J. G. Lussier	McMaster University, Canada
A. Malik	Northwestern University
S. Moss	University of Houston
M. H. Mueller	Argonne National Laboratory
M. Nelson	University of Illinois
R. Newman	Dow Chemical Company
M. C. Petri	Argonne National Laboratory
K. Pettit	University of Illinois
M. Radler	Dow Chemical Company
J. H. Richardson	Argonne National Laboratory
H. Robota	AlliedSignal, Inc.
J. Roesler	University of Illinois

D. Rosenfield	University of Pennsylvania
M. Salamon	University of Illinois
P. Schelling	University of Illinois
A. Schroeder	McMaster University, Canada
T. Sendyka	University of Pennsylvania
M. C. Shih	Northwestern University
R. Simmons	University of Illinois
S. Teslic	University of Pennsylvania
C. Venkataraman	University of Illinois
H. Williams	AT&T Bell Laboratories
P. Wochner	University of Houston
S. X. Zeng	University of Illinois
A. Zhang	University of Akron

## Appendix C

### HONORS AND AWARDS

*Compiled by Muriel Tate*

The Metals and Ceramics Division at Oak Ridge National Laboratory has established a longstanding tradition of excellence. The quality of its research and the success of its development work have been the result of the established ability of its scientific and engineering staff. Since the division's initial achievements, this ability has been formally recognized in the many professional honors received.

Presented below is a listing of special honors and awards accorded to divisional staff personnel during the report period. The type of recognition received varies, but tends to fall into one of the following generic categories: honorific and professional society awards, appointments, conference involvement, and patents issued.

### HONORIFIC AND PROFESSIONAL SOCIETY AWARDS

**K. B. ALEXANDER, H-T. LIN, AND J. W. GEER** received a Martin Marietta Energy Systems, Inc., Licensing Support Award, December 13, 1993.

**M. L. ATCHLEY, R. H. BALDWIN, AND C. R. HOWELL** received a Martin Marietta Energy Systems, Inc., Licensing Support Award, December 13, 1993.

**J. K. BAIN** received a Martin Marietta Energy Systems, Inc., Administrative and Office Support Award for outstanding support and service to the M&C Division in the area of environmental compliance, May 28, 1993.

**T. M. BESMANN** received the President's Award for Continuous Improvement as part of Energy Systems Total Quality Management and in recognition of personal contributions toward those efforts, May 5, 1993.

**T. M. BESMANN, M. C. CLARK, K. M. COOLEY, R. A. LOWDEN, J. C. MCCLAUGHLIN, AND D. P. STINTON** received a Martin Marietta Energy Systems, Inc., R&D Accomplishment Award for the development of chemical vapor deposition technology that accelerates ceramic composite fabrication time from weeks to hours and tailors special ceramic coating properties, May 28, 1993.

**P. S. BISHOP** received the 1993 M&C Division Technical Support Award for Distinguished Achievement, September 16, 1993.

**K. S. BLAKELY** received a Martin Marietta Energy Systems, Inc., Licensing Support Award, December 13, 1993.

**P. J. BLAU** was named a Fellow by ASM International for innovative research leading to a new level of understanding of the mechanisms of friction and wear and for original contributions to the science of tribology, October 19, 1993.

**B. F. BYRUM** was honored for 38 years of chapter membership by Professional Secretaries International, Oak Ridge Chapter, July 12, 1993.

**B. R. CHILCOAT** received a Martin Marietta Energy Systems, Inc., Licensing Support Award, December 13, 1993.

**F. W. CHRISTIE** received the 1993 M&C Division Sustained Contribution Award for Distinguished Achievement, September 16, 1993.

**S. A. DAVID** was awarded the Comfort A. Adams Lecture and 1992 Charles H. Jennings Memorial Awards by the American Welding Society, April 27, 1993.

**S. A. DAVID** was named a Fellow by the American Association for the Advancement of Science, November 5, 1993.

**H. W. HAYDEN** was named Inventor of the Year by the International Hall of Fame in recognition for his work in Metallurgy, November 12, 1993.

**R. N. HENGSTLER** received a Martin Marietta Energy Systems, Inc., Administrative and Office Support Award for sustained and outstanding contribution to the efficient and cost-effective operation of the M&C Division Procurement Office, May 28, 1993.

**L. L. HORTON AND T. A. NOLAN** received a Martin Marietta Energy Systems, Inc., Award for the multi-site team utilized characterization equipment by creating an inventory data base and producing a video intended to educate customers about the equipment's capabilities, May 28, 1993.

**L. L. HORTON** won the 1993 YWCA (Knoxville, Tennessee) Tribute to Women Award for Science and Technology, October 11, 1993.

**L. L. HORTON** was named a Trustee of ASM International for a term of three years, October 18, 1993.

**L. L. HORTON** was named a Fellow by ASM International, October 19, 1993.

**C. R. HUBBARD, S. A. DAVID, X.-L. WANG, AND S. SPOONER** received the Best Poster Paper Award in the Engineering Ceramics Division for "Nondestructive Residual Stress Mapping in Ceramic-to-Metal Joints" from the American Ceramic Society, January 10, 1993.

**M. A. JANNEY AND O. O. OMATETE** received the Advanced Technology Award from the Inventors Clubs of America, Inc., April 19, 1993.

**J. I. KELLY AND M. K. FERBER** received Most Value-Able Player and Team Awards for being "shining examples of Energy Systems values in action," July 14, 1993.

**M. C. LAMB** received a Martin Marietta Energy Systems, Inc., Operations and Support Award as a member of the Administrative/Office Support Team for the ANS, May 28, 1993.

**R. J. LAUF AND D. W. BIBLE** received the Excellence in Technology Transfer Award from the Federal Laboratory Consortium, April 19, 1993.

**R. J. LAUF** received a Martin Marietta Energy Systems, Inc., Inventor of the Year Award for sustained contributions in devising innovative solutions to a wide variety of technologically important problems and transferring the inventions to industry, May 28, 1993.

**R. J. LAUF** was a Finalist for the Product of the Year Award from the *Plant Engineering Magazine*, November 18, 1993.

**R. J. LAUF** received an Unlicensable and Government Use Invention Disclosure Award from Martin Marietta Energy Systems, Inc., December 13, 1993.

**R. J. LAUF AND E. H. LEE** were named Martin Marietta Corporation Jefferson Cup Winners, May 28, 1993.

**E. H. LEE** received the Martin Marietta Energy Systems, Inc., Scientist of the Year Award, May 28, 1993.

**E. H. LEE, M. B. LEWIS, AND L. K. MANSUR** received a Martin Marietta Energy Systems, Inc., R&D Accomplishment Award for the development of hard-surfaced polymers, a new class of materials having hardness and wear resistance greater than that of metals, by ion beam processing, May 28, 1993.

**GERRY M. LUDTKA** received the 1992 DOE Weapons Program Award of Excellence for significant contributions toward increased quality, productivity, and creativity in support of the nation's weapons program, January 29, 1993.

**GAIL M. LUDTKA AND GERRY M. LUDTKA** received a Martin Marietta Energy Systems, Inc., Operations and Support Award for development of reverse-blow-forming for near-net-shape forming, May 28, 1993.

**C. G. MCKAMEY, P. J. MAZIASZ, AND S. VISWANATHAN** were named First In Class, Electron Microscopy - Scanning, in the 1993 International Metallographic Contest for "Microstructure and Fracture Behavior of As-Cast Fe<sub>3</sub>Al-Based Ordered Intermetallic Alloys," September 2, 1993.

**K. L. MORE** received a Martin Marietta Energy Systems, Inc., Publication Award for significant contributions to the understanding of mechanisms of performance of silicon nitride structural ceramics via excellence in technical publication, May 28, 1993.

**D. L. MORRIS** received the 1993 M&C Division Administrative Support Award for Distinguished Achievement, September 16, 1993.

**K. F. RUSSELL** received a Martin Marietta Energy Systems, Inc., Technical Support Award for outstanding and sustained contributions to the development of the ORNL Atom-Probe Facility and the use of this facility in materials research, May 28, 1993.

**V. K. SIKKA** was named a Fellow of ASM International, October 19, 1993.

**G. M. SLAUGHTER** was awarded the 1993 Allan Ray Putnam Service Award for sustained technical contributions in promoting the goals, objectives, and ideals of ASM International, October 18, 1993.

**C. J. SPARKS** was named a Fellow of the American Physical Society, November 21, 1993.

**M. W. TERRELL** was honored as a finalist in the Volunteer Community Service category of YWCA's Tribute to Women competition, October 11, 1993.

**P. T. THORNTON** was elected a member of Pi Lambda Theta, International Honor and Professional Association in Education, Alpha Xi Chapter, April 24, 1993.

**T. N. TIEGS AND P. F. BECHER** received the Advanced Technology Award for Silicon Carbide Whisker-Zirconia Reinforced Mullite and Alumina Ceramics from the Inventors Clubs of America, Inc., May 28, 1993.

**J. D. VOUGHT** received a Martin Marietta Energy Systems, Inc., Technical Support Award, December 13, 1993.

**M. H. YOO** was named the winner of the Humboldt Research Award for his accomplishments in materials research by the Alexander Von Humboldt Foundation, March 23, 1993.

**J. R. WEIR** received a Martin Marietta Energy Systems, Inc., Technical Achievement Award for developing and successfully piloting EM-50 environmental restoration technology logic diagrams to be used as a model for the DOE complex, May 28, 1993.

## APPOINTMENTS

**D. N. BRASKI** was appointed to the Executive Board of the American Vacuum Society, Tennessee Valley Chapter for a three-year term from September 1993, to September 1996.

**N. C. COLE** was appointed a member of the Board of Advisors for the University of Tennessee College of Engineering beginning July 1988 through June 1994.

**N. C. COLE** was appointed a member-at-large of the Technical Advisory Committee of the American Welding Society beginning July 1992 through June 1995.

**G. M. GOODWIN** was appointed a member of the Awards Committee and Fellows Selection Committee of the American Welding Society, June 1, 1993, through May 31, 1996.

**L. L. HORTON** was appointed a Director for Physical Sciences by the Microscopy Society of America for a three-year term, July 1993.

**C. R. HUBBARD** was appointed to the peer Review Panel for the Materials Science Program of the Department of Energy, Office of Basic Energy Sciences, Bethesda, Maryland, May 1993.

**C. R. HUBBARD** was elected as general member, Materials Science Special Interest Group, American Crystallographic Association, May 1993.

**L. K. MANSUR** was appointed Chairman of Editors for the *Journal of Nuclear Materials*, October 1993.

**L. K. MANSUR** was appointed Chairman of the Nuclear Materials Committee, a joint committee of TMS and ASM International, for a two-year term from February 1993-1995.

**M. K. MILLER** was elected president of the International Field Emission Society for a one-year term from August 1, 1993, to July 31, 1994.

**M. K. MILLER** was elected to the Steering Committee of the International Field Emission Society for a four-year term from August 1, 1993, to July 31, 1997.

**D. F. PEDRAZA** was appointed a Professor in Residence by the University of Connecticut, April 1993.

**P. S. SKLAD** was appointed Director for Physical Sciences by the Microscopy Society of America for a three-year term from January 1, 1991, to December 31, 1993.

**C. J. SPARKS** was appointed an Adjunct Professor in the College of Engineering, Department of Materials Science and Engineering, by the University of Tennessee, May 1993.

**V. J. TENNERY** was appointed a Courtesy Professor in the Department of Materials Science and Engineering by the University of Florida for academic year 1993-94, July 28, 1993.

## **CONFERENCES**

**ASTM Symposium Committee C-28 on Advanced Ceramics, 17th Annual Conference on Composites and Advanced Ceramics, American Ceramic Society (ACerS), Cocoa Beach, Florida, January 11-13, 1993**

C. R. Brinkman, Chairman

**WATTEC '93 20th Annual Technical Conference and Exhibition on Nondestructive Testing: The Key to New Applications for Advanced Materials, Knoxville, Tennessee, February 16-19, 1993**

G. M. Slaughter, Moderator

**Organizing Committee for the Symposium on Nanocrystalline Materials, 1993 TMS Annual Meeting, Denver, Colorado, February 21-25, 1993**

P. J. Maziasz, Co-organizer

**Modeling for Welding Science Workshop, International Research Assistance Task Force, Cocoa Beach, Florida, March 16-19, 1993**

T. Zacharia, Organizer

**ASM International, Oak Ridge Chapter, Intelligent Materials Systems & Structures 1993 Educational Symposium, Oak Ridge, Tennessee, April 16, 1993**

C. G. McKamey, Organizer

**The Seventh Annual Conference on Fossil Energy Materials, Oak Ridge, Tennessee, May 11-13, 1993**

N. C. Cole, Co-organizer

R. R. Judkins, Co-organizer

**American Society for Testing and Materials Development of Fracture Mechanics Test Practices for Toughness in the Transition Temperature Range, May 18, 1993**

D. E. McCabe, Organizer

**American Crystallography Association Annual Meeting, Albuquerque, New Mexico, May 23, 1993**

C. J. Sparks, Organizer

**38th ASME International Gas Turbine & Aeroengine Congress & Exposition, Coal Utilization Committee, Cincinnati, Ohio, May 24-27, 1993**

R. R. Judkins, Chairman and Organizer

**1993 American Society of Mechanical Engineers Pressure Vessels and Piping Conference, Denver, Colorado, July 25-29, 1993**

D. E. McCabe, Chairman

**42nd Annual Denver X-ray Conference, Denver, Colorado, August 1-6, 1993**

C. R. Hubbard, Workshop Chairman and Organizer

**Editorial Board of the 40th International Field Emission Symposium, Nagoya, Japan, August 2-6, 1993**

M. K. Miller, Co-chairman

**International Conference on Silicon Nitride-Based Ceramics, Stuttgart, Germany, October 4-6, 1993**

P. F. Becher, Co-chairman

**1993 Materials Week, ASM-TMS Fall Meeting, Pittsburgh, Pennsylvania, October 17, 1993**

P. J. Maziasz, Vice-Chairman and Secretary

**International Conference on Modeling and Control of Joining Processes, Orlando, Florida, December 8-10, 1993**

T. Zacharia, Co-organizer

## **PATENTS ISSUED**

**R. J. LAUF, C. E. HOLCOMBE, JR., AND N. L. DYKES, "Process for Manufacturing Tantalum Capacitors," U.S. Patent 5,184,286, February 2, 1993.**

**R. J. LAUF AND R. L. HEESTAND, "Device and Method for Skull-Melting Depth Measurement," U.S. Patent 5,185,031, February 9, 1993.**

**A. J. MOORHEAD AND H. E. KIM**, "Process for Strengthening Silicon-Carbide Bodies, Whiskers, and Fibers," U.S. Patent H1166, April 6, 1993.

**T. N. TIEGS**, "Improved Pressureless Sintering of Whisker-Toughened Ceramic Composites," U.S. Patent 5,207,958, May 4, 1993.

**R. A. MCKEE AND F. J. WALKER**, "Process for Depositing Epitaxial Barium Oxide onto a Silicon Substrate and Novel Devices Prepared Thereby," U.S. Patent 5,225,031, July 6, 1993.

**R. J. LAUF**, "Zinc Oxide Varistors and/or Resistors," U.S. Patent 5,231,370, July 27, 1993.

**K. C. LIU**, "Apparatus for Tensile Testing Plate-Type Ceramic Specimens," U.S. Patent 5,237,876, August 24, 1993.

**V. K. SIKKA AND C. G. MCKAMEY**, "High Ductility Iron-Aluminide Alloy," U.S. Patent 5,238,645, August 24, 1993.

**R. J. LAUF, C. HAMBY, M. A. AKERMAN, AND R. D. SEALS**, "Damage Tolerant Light Absorbing Material," U.S. Patent 5,243,464, September 7, 1993.

**T. G. KOLLIE, L. H. THACKER, AND H. A. FINE**, "Improved Instrument for Measurement of Vacuum in Sealed Plastic Film Packets," U.S. Patent 5,249,454, October 5, 1993.

**T. J. HUXFORD**, "Improved Metal Atomization Spray Nozzle," U.S. Patent 5,261,611, November 16, 1993.

**H. D. KIMREY**, "Method and Apparatus for Radio Frequency Ceramic Sintering," U.S. Patent 5,266,762, November 30, 1993.

**R. J. LAUF, W. D. ARNOLD, AND W. D. BOND**, "Method of Preparing Hydrous Zinc Oxide Microspheres," U.S. Patent 5,269,972, December 14, 1993.

## Appendix D

### SEMINAR PROGRAM

*Compiled by Muriel Tate*

Because effective exchange of information is so vital to scientific and technological advance, the division sponsors and maintains an active seminar program for communication of ideas and discussion of results among researchers working in the broad field of materials science and engineering. Most of the talks deal with technical topics and are presented by invited speakers affiliated with research institutions located elsewhere in North America and abroad. The actual number of talks scheduled in any given week varies but, over the year, averages more than one per week.

The seminar program is administered by a committee appointed by division management. In function, the program achieves the desired goal of providing a forum for free exchange of information and for the passage of intellectual ideas that one can criticize, react to, and act upon. In short, these periodic exchanges aid the researcher in his or her quest for new knowledge and provide stimuli for further meaningful work that enhances basic understanding. The speakers and topics of seminars presented in the past year are as follows:

- K. J. KOZACZEK, Pennsylvania State University, University Park, "Material Microstructure Property Relationships Measurement and Modeling," January 6, 1993.
- T. L. STARR, Georgia Institute of Technology, Atlanta, "Development of Silicon Nitride Composites with Continuous Fiber Reinforcement," January 6, 1993.
- Y. WANG, Southwest Missouri State University, Springfield, Mo., "Ion Implantation and Characterization of High-Temperature and High-Performance Polymers," January 8, 1993.
- G. WELSCH, Case Western Reserve University, Cleveland, Ohio, "Ion-Implanted High-Temperature Microstructural Fences," January 14, 1993.

**M. MCNALLAN**, University of Illinois, Chicago, "Oxidation of Silicon-Based Ceramics in Environments Contaminated by Chlorine or Alkali Chlorides," January 25, 1993.

**Y. BAE**, Stevens Institute of Technology, Hoboken, N.J., "Metalorganic Chemical Vapor Deposition of Silicon-Based Dielectric Thin Films and In Situ Laser Diagnostics," January 26, 1993.

**M. S. DAW**, Sandia National Laboratories, Livermore, Calif., "A Tale of Two Topics: Epitaxial Growth and Atomistic Calculations of Large Systems," January 28, 1993.

**K. C. WALTER**, University of Wisconsin, Madison, "Plasma Source Ion Implantation of Aluminum," February 4, 1993.

**D. J. SROLOVITZ**, University of Michigan, Ann Arbor, "Grain Growth and Recrystallization in Metals," February 4, 1993.

**B. RADHAKRISHNAN**, University of Alabama, Tuscaloosa, "Modeling of Microstructural Evolution in the Weld Heat-Affected Zone," February 5, 1993.

**R. E. FULTON**, Georgia Institute of Technology, Atlanta, "High-Performance Computing in Engineering Analysis and Design," February 10, 1993.

**A. J. PAUL**, Concurrent Technologies Corporation, Johnstown, Pa., "An Integrated Approach to Modeling the Casting Process," February 11, 1993.

**E. K. OHRINER**, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tenn., "External Oxidation of Thorium in an Iridium Alloy as a Cause of Accelerated Grain Growth," February 26, 1993.

**D. W. RICHERSON**, Private Consultant, "The Role of Characterization in Product Development and Manufacturing," March 2, 1993.

**L. XIE**, University of Wisconsin, Madison, "Surface Modification and Deposition of Diamond-Like Carbon Films by Plasma Source Ion Implantation," March 11, 1993.

**A. J. GRIFFIN, JR.**, Rice University, Houston, Tex., "Physical Properties of Thin Films," March 12, 1993.

**R. O. RITCHIE**, Lawrence Berkeley Laboratory and University of California, Berkeley, "Crack Growth at or Near Bi-Material Interfaces," March 18, 1993.

**R. VISHNU**, Lulea University of Technology, Sweden, "Phenomenological Modeling of Phase Transformations in Welds," March 22, 1993.

**J. D. LIVINGSTON**, Massachusetts Institute of Technology, Cambridge, "Deformation and Defects in Laves-Phase Intermetallics," March 23, 1993.

**E. VERLEMMAN AND A. WAGEMANN**, Fraunhofer-Institut für Produktionstechnologie, Aachen, Germany, "Innovative Machining Processes," March 26, 1993.

**A. J. DEARDO**, University of Pittsburgh, Pa., "Deformation Processing of Advanced Alloys," March 31, 1993.

**X. JIANG**, Fraunhofer - Institut für Schicht-und Oberfachentechnik Hamburg, Germany, "Epitaxial Growth of Diamond on Silicon," April 15, 1993.

**R. NEWNHAM**, Pennsylvania State University, University Park, "Electrostriction and Piezoelectricity," April 15, 1993.

**D. T. WIRIG**, Modern Controls, Inc., Minneapolis, Minn., "New Developments in Measuring Gas and Water Vapor Permeability Through Barrier Films and Packages," April 15, 1993.

**R. GLASSELL**, "FARS: Teamwork in Action, Technology for the Future Battlefield," April 16, 1993.

**P. LLOYD**, "FARS: Teamwork in Action, Technology for the Future Battlefield," April 16, 1993.

**T. RAY**, "FARS: Teamwork in Action, Technology for the Future Battlefield," April 16, 1993.

**C. MCKAMEY**, Oak Ridge National Laboratory, Oak Ridge, Tenn., "Intelligent Material Systems and Structures," April 16, 1993.

**H-G BUSMANN**, Freiburger-Material Forschungszentrum (FMF), Freiburg, Germany, "STM-View on Polycrystalline Diamond Films and Scattering of  $C_{60}^+$  from Graphite, Diamond, and Epitaxial Fullerite," April 20, 1993.

**S. SIMUNOVIC**, Carnegie Mellon University, Pittsburgh, Pa., "Topics in Computational Science: Contact Mechanics, Eigenvalue Analysis, and Scientific Visualization," April 23, 1993.

**K. RAMESH**, Battelle Memorial Institute, Columbus, Ohio, "Material Issues Relating to Advanced Gas Turbine Systems," April 26, 1993.

**M. MIZUNO**, Japan Fine Ceramics Center, Nagoya, "Creep Rupture of Silicon Nitride Ceramics Related to the Ceramic Gas Turbine Project in Japan," April 27, 1993.

**S. E. BABCOCK**, University of Wisconsin, Madison, "What is the Matter With Grain Boundaries in  $YBa_2Cu_3O_{7-\delta}$ ?" April 28, 1993.

**J. ZOU**, Washington University, St. Louis, Mo., "Potential Energy Functions for Intermetallics," May 6, 1993.

**J. GOLDSTONE**, Los Alamos National Laboratory, N.M., "Measurements of Strain in Composites During Mechanical Loading," May 10, 1993.

**Z. LI**, Case Western Reserve University, Cleveland, Ohio, "Nucleation of Chemical Vapor-Deposited Diamond from Graphitic Carbon," May 12, 1993.

**D. WASSERMAN**, Waste Management and Remedial Action Division, Oak Ridge National Laboratory, Oak Ridge, Tenn., "Paper Recycling Meeting for Bldg. 4508," May 12, 1993.

**G. RILEY**, American Superconductor Corporation, Watertown, Mass., "Status of High-Temperature Superconducting Composite Wire Fabrication at American Superconductor Corporation," May 12, 1993.

**J. WOODYARD**, U.S. Bureau of Mines, Albany Research Center, Oregon, "Structure and Properties of Iron Aluminides," May 13, 1993.

**S. SASS**, Materials Science and Engineering Department, Cornell University, Ithaca, N.Y., "In Situ Formation of Metal-Ceramic Composites and Ductile-Phase Toughened Ceramics Using Partial Reduction Reactions," May 18, 1993.

**M. J. ANGWIN AND M. D. LIPSCHUTZ**, Fourth Floor Database Incorporated, Palo Alto, Calif., "Database Development for Technical Applications," May 19, 1993.

**I. ANDERSON**, Cornell University, Ithaca, N.Y., and University of Minnesota, Duluth, "Nickel Titanate Spinel," May 1993.

**W. GAWALEK**, Institute of Physical High Technology, Jena, Germany, "Melt Processing of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ ," May 24, 1993.

**Y. A. CHANG**, University of Wisconsin, Madison, "Composition Dependencies of Self-Diffusion Coefficients in  $\text{B}_2$  Intermetallic Compounds," June 14, 1993.

**E. D. SHCHUKIN**, North Carolina State University, Raleigh, "Physico-chemical Mechanics of Materials - (1) Catalytic effects in Sintering Metals and Ceramics, and (2) Electrochemical Machining of Hard Metals and Ceramics," June 21, 1993.

**D. JOSLIN**, University of Tennessee, Knoxville, "Ion Mixing in Oxide Sulfur Systems," June 28, 1993.

**S. WU**, University of Illinois, Urbana, "Effects of Lithium Doping on Phase Formation of Partial-Melt-Processed Polycrystalline  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ ," June 30, 1993.

**T. A. BURTSEVA**, The D. V. Efremov Institute of Electrophysical Apparatus, St. Petersburg, Russia, "Technological Features and Thermophysical Properties of Russian Carbon-Carbon Composites and RG-Ti Graphites for Fusion Application," and "Properties of Carbon-Based Materials Irradiated in Fission Reactors," July 7, 1993.

**J. C. M. LI**, University of Rochester, N.Y., "Dislocation Crack Interactions," July 9, 1993.

**P. K. LIAW**, University of Tennessee, Knoxville, "Nondestructive Evaluation for Metal-Matrix Composite Processing," July 12, 1993.

**J-B. LEBLOND**, University of Paris, "Mechanical Behavior of Steels During Phase Transformation," July 21, 1993.

**R. P. WAHL**, Hahn-Meitner-Institut, Berlin, "Deformation Mechanism Map for the Superalloy IN738LC Under High-Temperature Monotonic Loading," July 26, 1993.

**M. V. NATHAL**, NASA Lewis Research Center, Cleveland, Ohio, "Intermetallics Research at NASA Lewis Research Center," July 30, 1993.

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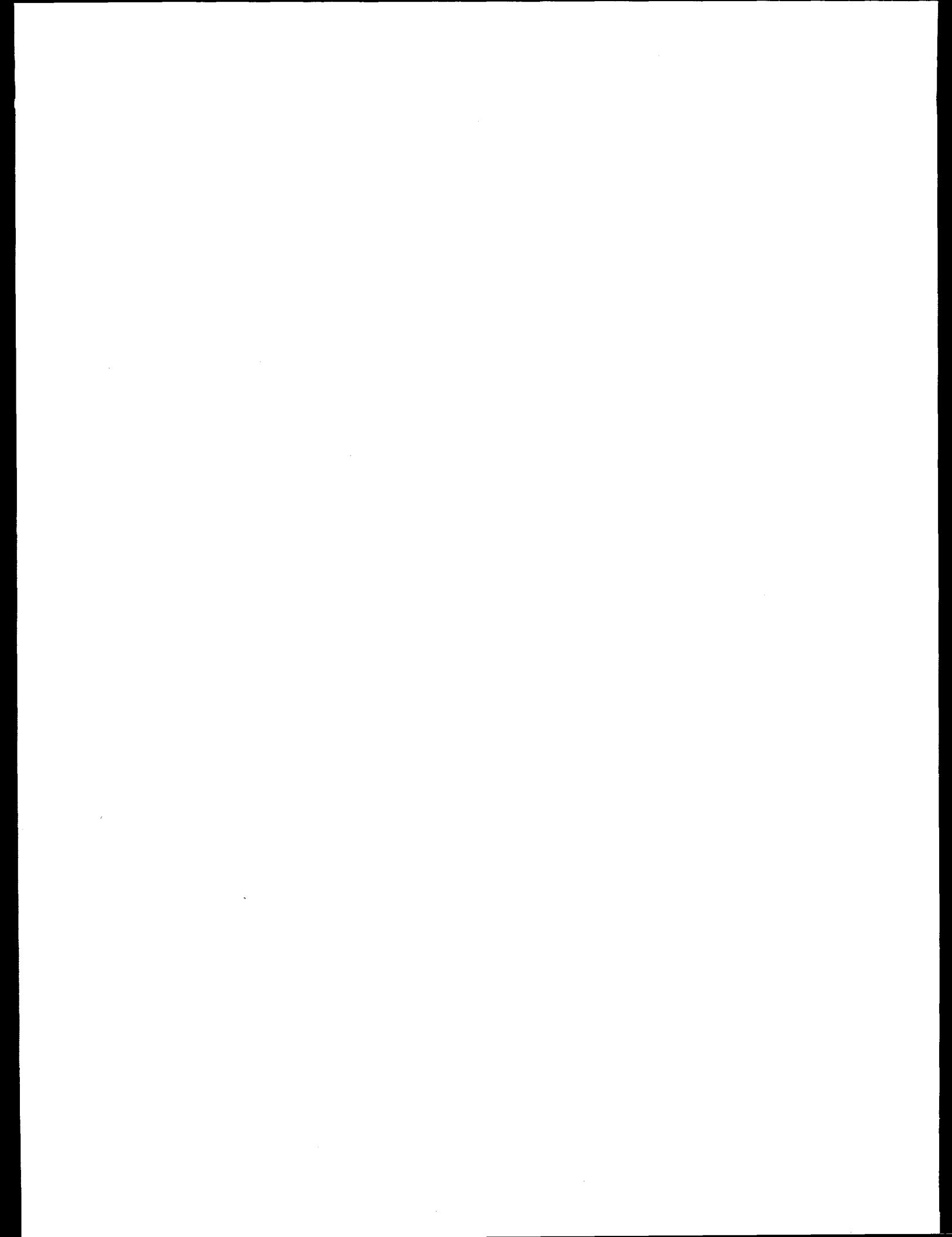
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## Appendix E

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**A. A. WERESZCZAK, K. BREDER, AND M. K. FERBER**

"Role of Oxidation in the Time-Dependent Failure Behavior of HIPed Silicon Nitride at 1370° C," *J. Am. Ceram. Soc.* 76(11), 2919-22 (November 1993).

**A. A. WERESZCZAK, M. K. FERBER, AND R. A. LOWDEN**

"Development of an Interfacial Test System for the Determination of Interfacial Properties in Fiber-Reinforced Ceramic Composites," *Ceram. Eng. Sci. Proc.* 14(7-8), 156-66 (1993).

**D. P. WHITE**

"The Effect of Ionizing and Displacive Radiation on the Thermal Conductivity of Alumina," *J. Appl. Phys.* 73(5), 2254-58 (1993).

**W. O. WINER AND R. S. COWAN**

*Development of a Theory of the Wear of Ceramics*, ORNL/Sub/93-07802, October 1993.

**K. H. WU AND C. T. LIU**

"Reactive Sintering of Ni<sub>3</sub>Al Intermetallic Alloys Under Compressive Stress," pp. 841-46 in *Proceedings of the Materials Research Society 1992 Fall Meeting, Boston, Massachusetts, November 30 - December 4, 1992*, Vol. 2, Materials Research Society, Pittsburgh, 1993.

**C. H. XU, C. L. FU, AND D. F. PEDRAZA**

"Simulations of Point Defect Properties in Graphite by a Tight-Binding Force Model," *Phys. Rev. B* 48(18), 13 273-79 (Nov. 1, 1993).

**H. YANG, J. C. SWIHART, D. M. NICHOLSON, AND R. H. BROWN**

"Calculation of the Electronic Properties of Ni-P Amorphous Alloys," *Phys. Rev. B* 47(1), 107-14 (Jan. 1, 1993).

**M. H. YOO AND C. L. FU**

"Fundamentals of Mechanical Behavior in Structural Intermetallics - A Synthesis of Atomistic and Continuum Modeling," pp. 283-92 in *Structural Intermetallics*, proceedings of symposium held at Champion, Pennsylvania, September 26-30, 1993, ed. R. Darolia, J. J. Lewandowski, C. T. Liu, P. L. Martin, D. B. Miracle, and M. V. Nathal, The Minerals, Metals & Materials Society, Warrendale, Pennsylvania, 1993.

**M. H. YOO AND C. L. FU**

"Strength of Intermetallic Compounds: TiAl", pp. 1286-93 in *Intermetallic Compounds for High-Temperature Structural Applications*, proceedings of the 3rd Japan International SAMPE Symposium held at Chiba, Japan, December 7-9, 1993, ed. M. Yamaguchi and H. Fukutomi, Society for the Advancement of Materials and Process Engineering, Azusa, California, 1993.

**M. H. YOO, S. L. SASS, C. L. FU, M. J. MILLS, D. M. DIMIDUK, AND E. P. GEORGE**

"Deformation and Fracture of Intermetallics," *Acta Metall. Mater.* **41**(4), 987-1002 (1993).

**T. ZACHARIA AND S. A. DAVID**

"Heat and Fluid Flow in Welding," pp. 3-23 in *Mathematical Modelling of Weld Phenomena*, ed. H. Cerjak and K. E. Easterling, The Institute of Materials, London, 1993.

**T. ZACHARIA, S. A. DAVID, AND J. M. VITEK**

"Understanding Heat and Fluid Flow in Linear GTA Welds," pp. 27-31 in *International Trends in Welding Science and Technology*, proceedings of conference held at Gatlinburg, Tennessee, June 1-5, 1992, ed. S. A. David and J. M. Vitek, ASM International, Materials Park, Ohio, 1993.

**L ZHAO, I. BAKER, AND E. P. GEORGE**

"Room-Temperature Fracture of FeCo," pp. 501-506 in *High-Temperature Ordered Intermetallic Alloys V*, proceedings of symposium held at Boston, Massachusetts, November 30 - December 4, 1992, Vol. 288, ed. I. Baker, R. Darolia, J. D. Wittenberger, and M. H. Yoo, Materials Research Society, Pittsburgh, 1993.

**B. ZHOU, Y. T. CHOU, AND C. T. LIU**

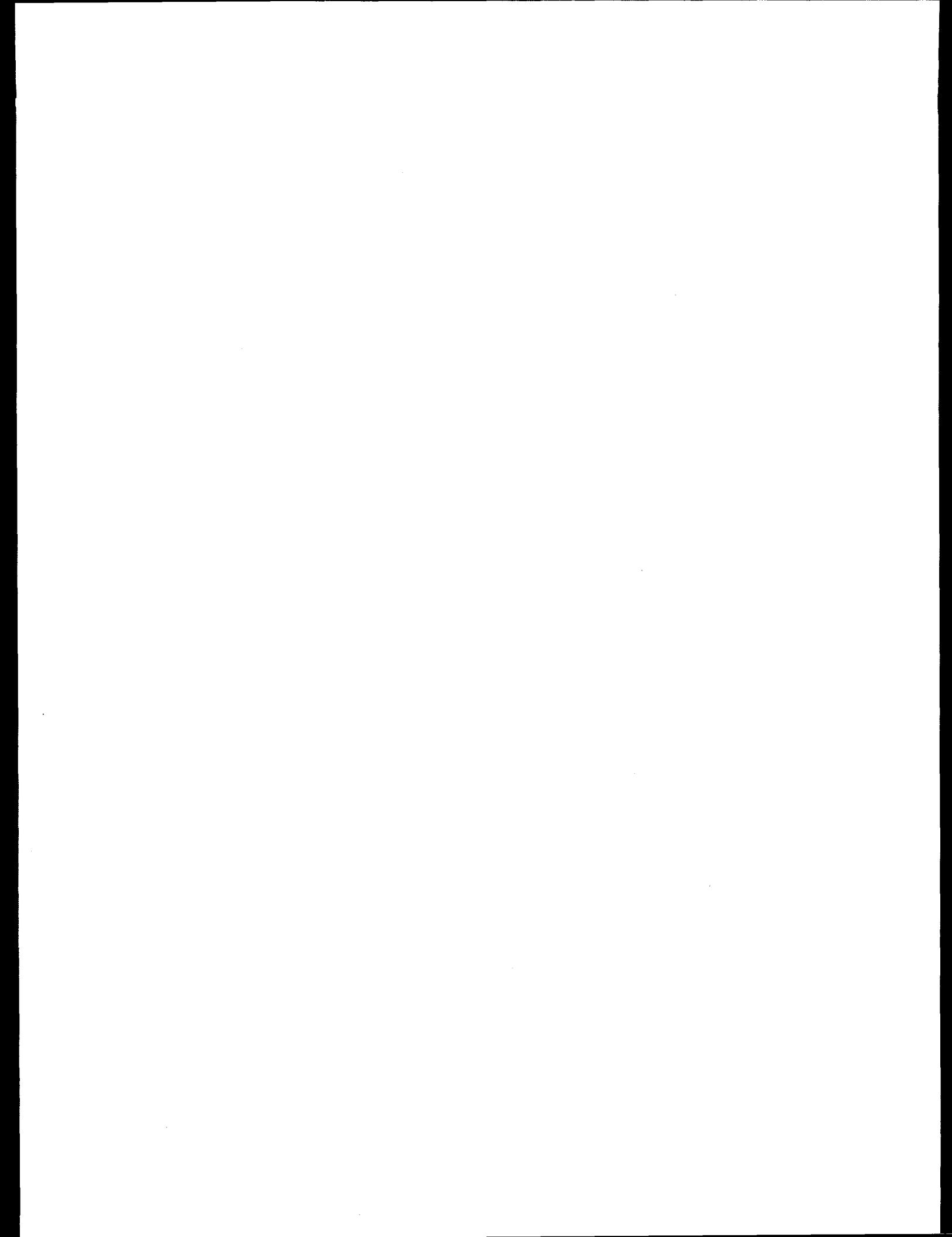
"Recrystallization and Grain Growth in Ni<sub>3</sub>Al With and Without Boron," *Intermetallics* **1**(4), 217-25 (1993).

**Y. ZHU, Z. L. WANG, AND M. SUENAGA**

"Grain Boundary Studies by the Coincident-Site Lattice Model and Electron-Energy-Loss Spectroscopy of the Oxygen K Edge in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-8</sub>," *Philos. Mag. A* **67**(1), 11-28 (1993).

**S. J. ZINKLE, P. J. MAZIASZ, AND R. E. STOLLER**

"Dose Dependence of the Microstructural Evolution in Neutron-Irradiated Austenitic Stainless Steel," *J. Nucl. Mater.* **206**, 266-86 (1993).



## Appendix F

### PRESENTATIONS AT TECHNICAL MEETINGS

*Compiled by Sherry Hempfling*

**John M. Cowley Symposium on Aspects of Electron Microscopy, Diffraction, Crystallography, and Spectroscopy, Arizona State University, Tempe, Arizona, January 5-8, 1993:**

L. F. ALLARD,\* E. VÖLKL, T. A. NOLAN, C. M. SUNG, K. J. OSTREICHER, AND B. ELMAN, "Electron Holography and High-Resolution Electron Microscopy of NiGaAs/InP Quantum Wells Grown by Gas Source MBE"

E. VÖLKL, "Density Correction of Photographic Materials"

Z. L. WANG, "Thermal Diffuse Scattering in High-Resolution Electron Holography"

**Tenth Symposium of Space Nuclear Power and Propulsion, Albuquerque, New Mexico, January 10-14, 1993:**

J. R. DISTEFANO\* AND J. W. HENDRICKS, "Oxidation/Corrosion Studies on Nb-1Zr for Space Reactor Applications"

E. K. OHRINER, "Improvements in Manufacture of Iridium Alloy Materials"

**17th Annual Conference on Composites and Advanced Ceramics, American Ceramic Society (ACerS), Cocoa Beach, Florida, January 10-15, 1993:**

S. C. BEECHER\* AND R. B. DINWIDDIE, JR., "Modeling the Thermal Conductivity of Fiber-Reinforced Ceramic Composites"

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\*Speaker

J. L. DING, K. C. LIU,\* AND C. R. BRINKMAN, "Multiaxial Deformation and Life-Prediction Model and Experimental Data for Advanced Silicon Nitride Ceramics"

R. B. DINWIDDIE, JR., "The Scanning Thermal Conductivity Microprobe: Theoretical Treatment"

M. K. FERBER,\* A. A. WERESZCZAK, AND R. A. LOWDEN, "Development of an Interfacial Test System for the Determination of Interfacial Properties in Fiber-Reinforced Ceramic Composites"

M. K. FERBER,\* A. A. WERESZCZAK, L. RIESTER, R. A. LOWDEN, AND K. K. CHAWLA, "Evaluation of the Interfacial Mechanical Properties in Fiber-Reinforced Ceramic Composites"

C. R. HUBBARD,\* S. A. DAVID, X.-L. WANG, AND S. SPOONER, "Nondestructive Residual Stress Mapping in Ceramic-to-Metal Joints"

R. A. LOWDEN,\* O. J. SCHWARZ, AND K. L. MORE, "Improved Fiber Coatings for Nicalon/SiC Composites"

K. L. MORE\* AND R. A. LOWDEN, "Evaluation of Matrix Properties in Continuous Fiber-Reinforced Ceramic Composites"

K. REIFSNIDER,\* W. STINCHCOMB, K. LIAO, L. OLEKSUK, AND D. P. STINTON, "Characterization of Ceramic Composite Materials for Gas Turbine Applications"

T. L. STARR,\* S. R. STOCK, T. M. BESMANN, AND J. C. MCLAUGHLIN, "Model Validation for Isothermal Chemical Vapor Infiltration"

D. P. STINTON,\* J. C. MCLAUGHLIN, B. FOSTER, K. REIFSNIDER, AND K. LIAO, "Characterization of Fiber-Reinforced Composites Fabricated by Forced CVI"

T. N. TIEGS,\* J. O. KIGGANS, AND K. L. PLOETZ, "Microwave Processing of  $\text{Si}_3\text{N}_4$  Ceramics"

P. F. TORTORELLI,\* J. R. KEISER, AND R. A. LOWDEN, "Influence of Fiber Coatings on the Oxidation of Fiber-Reinforced SiC Composites"

R. VAIDYANATHAN,\* J. SANKAR, A. K. KELKAR, AND D. P. STINTON, "Investigation of Mechanical Properties of Chemically Vapor Infiltrated (CVI) Ceramic Matrix Composites"

B. L. WEAVER,\* R. A. LOWDEN, J. C. MCLAUGHLIN, D. P. STINTON, T. M. BESMANN, AND O. J. SCHWARZ, "Nextel™/SiC Composites Fabricated Using Forced Chemical Vapor Infiltration"

A. A. WERESZCZAK,\* M. G. JENKINS, AND R. R. SANDERS, "Fracture Toughness ( $K_{Ic}$  &  $\Gamma_{vol}$ ) of a HIPed  $\text{Si}_3\text{N}_4$  at Elevated Temperatures"

**Wire Development Group Meeting, University of Wisconsin, Madison, Wisconsin, January 12, 1993:**

V. K. SIKKA, "Deformation Processing of High-Temperature Superconductor"

**Fusion Energy Advisory Committee Panel 6 Meeting, Dallas, Texas, January 13-15, 1993:**

E. E. BLOOM, "Development of Ferritic/Martensitic Steels for Fusion Applications"

**Seminar at the University of California, Department of Materials Science, Santa Barbara, California, January 15, 1993:**

Z. L. WANG, "Exploring the Surface and Internal Microstructures of Ceramics Using the Imaging and Spectroscopy Techniques in TEM"

**International Symposium on Advanced Electronic and Optoelectronic Materials, Los Angeles, California, January 18-23, 1993:**

H. M. PHILLIPS,\* T. FEURER, S. P. LE BLANC, D. L. CALLAHAN, R. SAUERBREY, AND J. BENTLEY, "Excimer Laser-Induced Electrical Conductivity and Mechanical Nanostructures in Polymers"

**Back to Basics, 1993 Energy Management Conference, Louisville, Kentucky, January 19, 1993:**

K. E. WILKES, "Insulation"

**Seminar at the University of Cincinnati, Cincinnati, Ohio, February 2, 1993:**

M. K. FERBER, "Evaluation of the Creep/Fatigue Behavior of a HIPed Silicon Nitride"

V. J. TENNERY, "The High Temperature Materials Laboratory Fellowship Program"

**Technical Seminar and Facilities Tour, Insituform Technologies, Insituform of North America, Memphis, Tennessee, February 3-4, 1993:**

L. J. TURNER, "Overview of LLLW Drain System, Bldg. 3025E"

**1993 Golden Gate Materials Technology Conference, San Francisco, California, February 3-5, 1993:**

A. E. PASTO, "Manufacture of High-Performance Structural Ceramics"

**Society for Photo, Electronic, and Instrumentation Engineering Conference, Albuquerque, New Mexico, February 5, 1993:**

M. J. DE VRIES, M. NASTA, J. PATEL, K. KAMDAR, R. A. LOWDEN, D. P. STINTON, S. ALLISON, J. MUHS, AND R. O. CLAUS,\* "Survivability of Optical Fiber Sensor Elements Embedded in Silicon Carbide Matrix Composites"

**International Conference on Advanced Composites - Advanced Composites '93, University of Wollongong, Australia, February 15-19, 1993:**

E. P. GEORGE,\* E. K. OHRINER, AND J. H. SCHNEIBEL, "Compatibility of Potential Composite Reinforcements with Ni<sub>3</sub>Al and Tungsten Metal Matrices"

**WATTEC '93, Knoxville, Tennessee, February 16-19, 1993:**

R. E. CLAUSING\* AND L. HEATHERLY, JR., "Advanced Synthetic Diamonds"

M. K. MILLER, "Visualization of Materials at the Atomic Level"

V. K. SIKKA, "Advanced Intermetallic Alloys: Nickel and Iron Aluminides"

**1993 TMS-AIME Annual Meeting, Denver, Colorado, February 21-25, 1993:**

R. L. HEESTAND, "A History of Tungsten- and Molybdenum-Base Alloys"

C. T. LIU, "Environmental Embrittlement of Ordered Intermetallic Alloys at Elevated Temperatures"

P. J. MAZIASZ, "Nano-Scale Information from Analytical Electron Microscopy (AEM) in Steels and Alloys"

M. K. MILLER, "Atomic-Scale Characterization of Materials with the Atom-Probe Field-Ion Microscope"

W. C. OLIVER,\* B. N. LUCAS, AND G. M. PHARR, "The Mechanical Characterization on the Submicron Scale Using Indentation Experiments"

J. H. SCHNEIBEL,\* R. DAROLIA, AND D. F. LAHRMAN, "Fracture Morphology of NiAl Single Crystals Tested in Tension"

Z. L. WANG,\* R. KONTRA, A. GOYAL, AND D. M. KROEGER, "Microstructures of Defects Near Y<sub>2</sub>BaCuO<sub>5</sub> Particles in Melt-Textured YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> and Their Possible Effects on Critical Current"

**U.S. Department of Energy/Japan Atomic Energy Research Institute (DOE/JAERI) Collaborative Program, Technical Progress Meeting, Oak Ridge, Tennessee, February 22, 1993:**

**D. J. ALEXANDER,\* J. E. PAWEL, M. L. GROSSBECK, AND A. F. ROWCLIFFE, "Fracture Toughness Testing of Miniature Disk Compact Specimens: Methodology and Initial Results"**

**Seminar at Commonwealth Scientific and Industrial Research Organization (CSIRO), Clayton, Australia, February 24, 1993:**

**E. P. GEORGE,\* C. T. LIU, AND D. P. POPE, "A Reexamination of Grain Boundary Fracture in Ni<sub>3</sub>Al: Environmental and Alloying Effects"**

**Workshop on Ti-Based Superconductors, National Renewable Energy Laboratory, Golden, Colorado, February 25-26, 1993:**

**A. GOYAL,\* Z. L. WANG, J. BRYNESTAD, AND D. M. KROEGER, "Microstructure and Processing of Thallium-Based Superconductors"**

**University of Dayton Research Institute (UDRI) Seminar Program, University of Dayton, Dayton, Ohio, February 26, 1993:**

**A. E. PASTO, "Microwave Processing of Ceramics"**

**International Symposium on Improved Technology for Fossil Power Plants—New and Retrofit Applications, Washington, D.C., March 1-3, 1993:**

**R. W. SWINDEMAN,\* C. R. BRINKMAN, AND H. D. UPTON, "Rupture Life Prediction for 9Cr-1Mo-V-Nb-Steel"**

**Non-Fuel Uses of Coal Research Team Meeting, Pittsburgh, Pennsylvania, March 5, 1993:**

**T. D. BURCHELL, "Carbon Materials R&D at ORNL, A Presentation to the Non-Fuel Uses of Coal Research Team"**

**The National Association of Corrosion Engineers (NACE) Annual Conference and Corrosion Show, New Orleans, Louisiana, March 7-10, 1993:**

**E. J. BARBER,\* M. S. TAYLOR, AND J. H. DEVAN, "Corrosion of Breached UF<sub>6</sub> Storage Cylinders"**

**G. E. C. BELL\* AND P. S. BISHOP, "Compatibility of Vanadium Alloys with Reactor-Grade Helium for Fusion Reactor Applications"**

**G. L. EDGEMON,\* D. F. WILSON, AND G. E. C. BELL, "Stability of Test Environments for Performance Evaluation of Materials for the Modular High-Temperature Gas-Cooled Reactor"**

E. L. FULLER, JR.,\* O. C. KOPP, AND A. D. UNDERWOOD, "Corrosion Kinetics and Mechanisms for Nuclear-Grade Graphites"

J. R. KEISER,\* J. I. FEDERER, J. J. WILLIAMS, AND R. A. ROSENBERG, "Evaluation of Ceramic Materials for an Advanced Steam-Methane Reformer"

R. E. PAWEL,\* D. K. FELDE, J. A. CLINARD, AND T. A. THORNTON, "The Corrosion Behavior of 8001 Al Under Heat-Transfer Conditions in an Aqueous Loop System"

P. F. TORTORELLI\* AND J. H. DEVAN, "Growth of Oxide Layers on Cr-Cr<sub>2</sub>Nb Alloys"

P. F. TORTORELLI,\* J. H. DEVAN, AND U. K. ABDALI, "Cyclic Oxidation of Iron Aluminides"

**Engineering Foundation Conference on Critical Issues in the Development of High-Temperature Structural Materials, Kona, Hawaii, March 7-12, 1993:**

C. T. LIU, "Alloy Design of Ordered Intermetallics"

M. H. YOO\* AND C. L. FU, "Critical Issues in the Computational Materials Science for Structural Materials"

**American Society for Metals (ASM) International Dinner Meeting, Nashville, Tennessee, March 9, 1993:**

P. J. BLAU, "Wear-Resistant Materials for the '90s"

**Affordable Comfort Conference, Philadelphia, Pennsylvania, March 12, 1993:**

K. E. WILKES, "Laboratory Measurements of Thermal Performance of Attic Insulations"

**10th Symposium on the R&D Project of Basic Technology for Future Industry, Fine Ceramics, Tokyo, Japan, March 16, 1993:**

D. R. JOHNSON, "Ceramic Technology for Advanced Heat Engines"

**DOE Workshop on Modeling for Welding Science, Cocoa Beach, Florida, March 16-19, 1993:**

T. ZACHARIA, "Modeling of Heat and Fluid Flow in Welds"

**Presentation at Los Alamos National Laboratory, Los Alamos, New Mexico, March 19, 1993:**

D. L. JOSLIN,\* C. J. MCHARGUE, AND C. W. WHITE, "Ion Mixing in Oxide-Sapphire Systems"

**Modeling of Casting, Welding, and Advanced Solidification Processes VI, Palm Coast, Florida, March 21-26, 1993:**

S. VISWANATHAN,\* V. K. SIKKA, AND H. D. BRODY, "The Application of Quality Criteria for the Prediction of Porosity in the Design of Casting Processes"

**American Physical Society (APS) Meeting, Seattle, Washington, March 22-26, 1993:**

R. H. BROWN AND D. M. C. NICHOLSON,\* "Electrical Resistivity of Strong-Scattering Short-Range Ordered Alloys: Explanation of the K-State Effect"

W. H. BUTLER, D. M. C. NICHOLSON, Z.-G. ZHANG, AND J. M. MACLAREN,\* "Theory of Giant Magnetoresistance Effect in Layered Transition Metal Systems"

D. M. C. NICHOLSON,\* G. M. STOCKS, W. A. SHELTON, W. M. TEMMERMAN, AND Z. SZOTEK, "Application of Local Self-Consistency to Alloy Phase Stability"

G. S. PAINTER, "Effects of Impurities and Dopants on Metallic Cohesion"

D. SEIFU,\* F. J. PINSKI, W. A. SHELTON, G. M. STOCKS, AND D. D. JOHNSON, "Total Energy of Ordered and Disordered Cu<sub>x</sub>Au<sub>1-x</sub> Alloys"

E. D. SPECHT\* AND F. J. WALKER, "Near-Edge X-Ray Fine Structure of an Interface"

**Cellular Polymers II, Heriot-Watt University, Edinburgh, United Kingdom, March 23-25, 1993:**

D. W. YARBROUGH,\* R. S. GRAVES, AND J. R. BOOTH, "Aging of Thin Slices of PIR Foams Manufactured with Alternative Blowing Agents"

**1993 American Chemical Society National Spring Meeting, Materials Chemistry in Energy R&D Symposium, Denver, Colorado, March 28 - April 3, 1993:**

R. R. JUDKINS, "The DOE Fossil Energy Advanced Research and Technology Development Materials Program"

**1993 American Society for Nondestructive Testing, Inc. (ASNT), Spring Conference, Nashville, Tennessee, March 29 - April 2, 1993:**

C. V. DODD\* AND J. R. PATE, "Advancement in Eddy-Current Test Technology for Steam Generator Tube Inspection"

**Presentation at Georgia Institute of Technology, Atlanta, Georgia, March 30, 1993:**

V. K. SIKKA, "Development and Commercialization Status of Fe<sub>3</sub>Al-Based Intermetallic Alloys"

**Navy Best Manufacturing Practices Review Team Meeting, Oak Ridge National Laboratory, Oak Ridge, Tennessee, March 30-31, 1993:**

C. R. BRINKMAN AND H. E. MCCOY,\* "Test Method Development and Mechanical Property Testing of Metals, Ceramics, and Polymers"

T. D. BURCHELL, "Processing and Characterization of Graphite and Carbon-Carbon Composite Materials"

B. L. COX, "High Temperature Materials Laboratory (HTML)"

G. M. LUDTKA,\* K. W. CHILDS, G. A. ARAMAYO, K. H. LUK, AND J. E. PARK, "Quench Simulator"

V. K. SIKKA, "Development and Manufacture of Refractory Metals for Space Power Applications"

**Pittsburgh Energy Technology Center, Pittsburgh, Pennsylvania, April 6, 1993:**

K. BREDER\* AND V. J. TENNERY, "Materials Support for HITAF"

K. BREDER\* AND V. J. TENNERY, "Materials Support for HITAF Proposed Work, FEAA291"

**Presentation at AMOCO Chemical Corporation, Oak Ridge National Laboratory, Oak Ridge, Tennessee, April 8, 1993:**

T. D. BURCHELL, "Monolithic Carbon-Fiber Composites for Hydrogen Separation"

**Tennessee Wesleyan College - Business Communications Class, Oak Ridge, Tennessee, April 8, 1993:**

P. M. HUMPHREYS\* AND M. K. FERBER, "High Temperature Materials Laboratory (HTML)"

**Materials Research Society (MRS) 1993 Spring Meeting, San Francisco, California, April 12-16, 1993:**

W. H. BUTLER,\* J. M. MACLAREN, AND X.-G. ZHANG, "Theory of the Giant Magneto-Resistance Effect"

Y. L. CHEN,\* J. BENTLEY, D. M. MAHER, C. WANG, AND G. LUCOVSKY, "Electron Microscopy Studies of Undoped and Doped Si:H and Si<sub>x</sub>C<sub>x</sub>:H Films"

B. D. FABES, W. C. OLIVER,\* R. A. MCKEE, AND F. J. WALKER, "Determination of Film Hardness from the Composite Response of Film and Substrate to Nanometer-Scale Indentations"

G. M. PHARR\* AND W. C. OLIVER, "Measurement of Thin-Film Mechanical Properties Using Nanoindentation"

G. M. PHARR\* AND W. C. OLIVER, "Studies of the Mechanical Behavior of Rocks and Ceramics Using Nanoindentation"

W. H. POISL,\* B. D. FABES, AND W. C. OLIVER, "A Quantitative Model for Interpreting Nanometer-Scale Hardness Measurements of Thin Films"

M. SLUITER,\* P. E. A. TURCHI, AND G. M. STOCKS, "Effect of Pressure on Order and Stability in Al-Ti Alloys"

X.-L. WANG,\* J. A. FERNANDEZ-BACA, C. R. HUBBARD, K. B. ALEXANDER, AND P. F. BECHER, "Transformation Behavior in  $ZrO_2$ ( $CeO_2$ )/ $Al_2O_3$  Ceramic Composites"

X.-L. WANG,\* C. R. HUBBARD, S. SPOONER, S. A. DAVID, AND T. A. DODSON, "Stress Mapping in a Zirconia/Iron Joint"

**9th International Conference on Wear of Materials, San Francisco, California, April 13-17, 1993:**

P. J. BLAU, "Friction Microprobe Investigation of Particle Layer Effects on Sliding Friction"

P. J. BLAU, "Tribology - Fact or Friction?"

G. R. RAO,\* E. H. LEE, AND L. K. MANSUR, "Structure and Dose Effects on Improved Wear Properties of Ion-Implanted Polymers"

**ACerS 95th Annual Meeting, Cincinnati, Ohio, April 18-22, 1993:**

K. B. ALEXANDER\* AND P. F. BECHER, "Grain Morphology and Evolution in Alumina/Zirconia Composites"

K. BREDER,\* A. A. WERESZCZAK, M. K. FERBER, AND M. G. JENKINS, "Comparison of Dynamic Fatigue Behavior of a PY6 Silicon Nitride in Tension and Flexure"

F. M. FOUST, "The High Temperature Materials Laboratory - A National Resource"

D. J. GRELLINGER\* AND M. A. JANNEY, "Temperature Measurement in a 2.45-GHz Microwave Furnace"

C.-H. HSUEH, "Evaluation of Interfacial Properties of Fiber-Reinforced Ceramic Composites Using a Mechanical Properties Microprobe"

M. A. JANNEY,\* M. L. JACKSON, AND H. D. KIMREY, "Microwave Sintering of  $\text{ZrO}_2$ - $\text{CeO}_2$ - $\text{Y}_2\text{O}_3$ "

J. O. KIGGANS, JR.,\* T. N. TIEGS, AND C. E. HOLCOMBE, "Production of Complex SRBSN Parts by Microwave Heating"

R. J. LAUF,\* D. W. BIBLE, S. R. MADDOX, C. A. EVERLEIGH, R. J. ESPINOSA, AND A. C. JOHNSON, "Materials Processing Using a Variable-Frequency Microwave Furnace (VFMF)"

H. T. LIN\* and P. F. BECHER, "Microstructural Aspects Contributing to the Creep Deformation of Alumina-SiC Composites"

M. S. MORROW, C. E. HOLCOMBE, H. D. KIMREY,\* AND C. T. WILSON, "Microwave Furnace for Large-Scale Ceramic Processing"

D. K. PEELER,\* T. D. TAYLOR, O. B. CAVIN, AND W. D. PORTER, "Melting Kinetics of Soda-Lime-Silica Glasses by In Situ High-Temperature X-Ray Diffraction"

W. D. PORTER,\* R. B. DINWIDDIE, JR., AND C. R. HUBBARD, "Characterization of a Composite Latent/Sensible Heat Storage Medium Using Thermal Analysis and X-Ray Diffraction"

A. W. SMITH, T. L. STARR,\* AND D. P. STINTON, "Thermal Radiation Effects in Chemical Vapor Infiltration: Modelling and Experimentation"

X.-L. WANG, J. A. FERNANDEZ-BACA,\* K. B. ALEXANDER, P. F. BECHER, AND C. R. HUBBARD, "Residual Microstresses and the t-m Phase Transition in  $\text{ZrO}_2$ ( $\text{CeO}_2$ )/ $\text{Al}_2\text{O}_3$  Ceramic Composites"

A. A. WERESZCZAK,\* K. BREDER, and M. K. FERBER, "Dynamic Fatigue Behavior of a HIPed Silicon Nitride in Air and Inert Environments at 1370°C"

Seminar at University of Illinois, Urbana-Champaign, Illinois, April 19, 1993:

C. T. LIU, "Moisture-Induced Hydrogen Embrittlement of Ordered Intermetallic Alloys"

**Workshop on Computational Modeling of Materials with Evolving Microstructures,  
University of California, San Diego, California, April 19-21, 1993:**

G. M. LUDTKA,\* K. E. CHILDS, G. A. ARAMAYO, K. H. LUK, J. E. PARK,  
A. ALINAS-RODRIGUEZ, J. H. ROOT, T. M. HOLDEN, AND S. R. MACEWEN,  
"Computer Simulation of the Effects of Heat Treatment and Uniaxial Compression  
on the Residual Stress in a Uranium-0.8 Weight Percent Titanium Alloy: Analytical  
Predictions and Experimental Verification"

**International Conference on Metallurgical Coatings and Thin Films (ICMCTF-93),  
San Diego, California, April 19-23, 1993:**

P. J. BLAU\* AND C. S. YUST, "Microfriction Studies of Model Self-Lubricating  
Surfaces"

G. M. PHARR\* AND W. C. OLIVER, "Measurement of Thin-Film Mechanical  
Properties Using Nanoindentation"

**Seminar at Lawrence Livermore National Laboratory, Livermore, California, April 20, 1993:**

W. H. BUTLER, "Giant Magnetoresistance Effect in Transition-Metal Superlattices"

**Seminar at IBM Almaden, San Jose, California, April 21, 1993:**

W. H. BUTLER, "Giant Magnetoresistance Effect in Transition-Metal Superlattices"

**Casting Expo '93, American Foundrymen's Society, Inc., Chicago, Illinois, April 23-27,  
1993:**

S. VISWANATHAN\* AND H. D. BRODY, "Microporosity in Grain-Refined Al-4.5%  
Cu Alloy Castings and Its Relation to Casting Practice"

**Korean-American Scientist Engineers Association (K-ASEA), Tennessee Chapter Meeting,  
Knoxville, Tennessee, April 24, 1993:**

W. Y. LEE, "Synthesis of High-Temperature Materials by Chemical Vapor  
Deposition"

**American Welding Society (AWS) Annual Meeting, Houston, Texas, April 25-30, 1993:**

S. A. DAVID, "Recent Advances in Welding Science"

**Wire Development Quarterly Review Meeting, Argonne National Laboratory, Argonne,  
Illinois, April 28, 1993:**

V. K. SIKKA, "Deformation Processing of High-Temperature Superconductor"

**American Society of Mechanical Engineers (ASME) Workshop on Research Guidelines for Aluminum Products Applications on Transportation and Industry, Clearwater Beach, Florida, May 3-5, 1993:**

S. VISWANATHAN, "Computer-Aided Design and Analysis of MMC Casting Processes"

**Expo '93, Oak Ridge, Tennessee, May 3-7, 1993:**

A. E. PASTO, "Ceramics for Engines"

**Seminar at Material Science Department at the Technion, Haifa, Israel, May 4-6, 1993:**

T. M. BESMANN, "Vapor-Phase Processing of Ceramic Coating and Composites"

**Environmentally Conscious Synthesis, Processing, and Use of Ceramics, DOE Office of Basic Energy/Office of Industrial Technology (OBES/OIT) Research Assistance Task Force Meeting, Princeton University, Princeton, New Jersey, May 5-7, 1993:**

S. D. NUNN, "Gelcasting with Water-Based Additives"

**Experts Meeting on Cladding and Finite Flaw Length Effects, San Diego, California, May 7, 1993:**

R. K. NANSTAD,\* F. M. HAGGAG, AND D. E. MCCABE, "Effects of Irradiation and Thermal Aging on Fracture Toughness of Stainless Steel Cladding"

**2nd International Seminar on Numerical Analysis of Weldability, Graz, Austria, May 10-11, 1993:**

T. ZACHARIA, S. A. DAVID, AND T. A. DEBROY,\* "Computational Modeling of Linear GTA Welds and Comparison to Experimental Results"

**Presentation at National Renewable Energy Laboratory, Denver, Colorado, May 11, 1993:**

R. L. BEATTY, "Materials Capabilities"

**Seventh Annual Conference on Fossil Energy Materials, Oak Ridge, Tennessee, May 11-13, 1993:**

D. J. ALEXANDER\* AND V. K. SIKKA, "Mechanical Properties of Iron-Aluminum Alloys"

J. H. DEVAN\* AND P. F. TORTORELLI, "Environmental Effects on Iron Aluminides"

G. M. GOODWIN,\* C. J. MCKAMEY, P. J. MAZIASZ, AND V. K. SIKKA, "Weldability of Iron Aluminides"

C. T. LIU, "Cr<sub>2</sub>Nb Development"

C. G. MCKAMEY\* AND V. K. SIKKA, "Development of Ductile Fe<sub>3</sub>Al-Based Aluminide - A Bit of History"

V. K. SIKKA,\* R. H. BALDWIN, AND C. R. HOWELL, "Low-Aluminum Content Iron-Aluminum Alloys"

R. W. SWINDEMAN, "Investigation of Austenitic Alloys for Advanced Heat Recovery and Hot-Gas Cleanup Systems"

P. F. TORTORELLI,\* L. J. CARSON, AND J. H. DEVAN, "Effects of Alloying and Temperature on the High-Temperature Oxidation of Cr-Cr<sub>2</sub>Nb"

S. VISWANATHAN,\* C. G. MCKAMEY, P. J. MAZIASZ, AND V. K. SIKKA, "Tensile Properties of As-Cast Iron-Aluminide Alloys"

**Presentation at The Gillette Company, Boston Research and Development, Boston, Massachusetts, May 13, 1993:**

E. H. LEE, "Improvement of Surface-Sensitive Mechanical Properties of Polymers by Ion Beam Processing"

**RES/NRR/ORNL Meeting of Reactor Pressure Vessel (RPV) Integrity, NRC Contractors Meeting on Radiation Effects, Rockville, Maryland, May 13-14, 1993:**

W. R. CORWIN, "Heavy-Section Steel Irradiation Program Summary"

K. FARRELL, "Gamma-Ray-Induced Atomic Displacement Effects in Metals - A Review"

K. FARRELL,\* S. T. MAHMOOD, R. E. STOLLER, AND L. K. MANSUR, "Evaluation of Low-Temperature Radiation Embrittlement Mechanisms in Ferritic Alloys"

R. K. NANSTAD, "Observations on Irradiation-Induced Fracture Toughness and Charpy Impact Shifts"

R. E. STOLLER, "Microstructural Analysis of Radiation Effects in Pressure Vessel Steels"

**American Society for Testing and Materials (ASTM) Workshop on User's Experience in Crack-Arrest Testing, Atlanta, Georgia, May 17, 1993:**

S. K. ISKANDER,\* W. R. CORWIN, AND R. K. NANSTAD, "ORNL's Experiences in the Preparation and Testing of Irradiated and Unirradiated Crack-Arrest Specimens"

**Annual Meeting of the Society of Tribologists and Lubrication Engineers, Alberta, Canada, May 17-20, 1993:**

C. S. YUST, "Fast Fourier Transform Analysis of Computer-Acquired Friction Data"

**Meeting with Duke/Fluor Daniel Company and Duke Power Utility, Oak Ridge National Laboratory, Oak Ridge, Tennessee, May 19, 1993:**

N. C. COLE, "Overview of the AR&TD Fossil Energy Materials Program"

**Materials Engineering Program Lecture, Auburn, Alabama, May 20, 1993:**

H.-T. LIN, "Elevated-Temperature Mechanical Behavior of Toughened Ceramics in Air"

**National Institute of Standards and Technology (NIST) Seminar, Gaithersburg, Maryland, May 21, 1993:**

Z. L. WANG,\* A. GOYAL, R. KONTRA, D. M. KROEGER, J. R. THOMPSON, AND R. K. WILLIAMS, "Microstructures of YBCO Superconductors and Ceramic Surfaces Determined by HRTEM and Associated Analytical Techniques"

**Seminar at National Aeronautics and Space Administration (NASA) Lewis Research Center, Cleveland, Ohio, May 21, 1993:**

P. F. TORTORELLI, "High-Temperature Oxidation Effects on Fiber-Reinforced SiC Composites"

**Seminar at the Advanced Research Laboratory, Hitachi Ltd., Saitama, Japan, May 21, 1993:**

J. BENTLEY,\* E. A. KENIK, N. D. EVANS, AND Z. L. WANG, "Analytical Electron Microscopy of Materials"

**American Society for Information Science (ASIS) Meeting, Knoxville, Tennessee, May 21-27, 1993:**

D. N. BRASKI,\* A. D. UNDERWOOD, L. RIESTER, AND R. L. JACKSON, "The Use of Atomic-Force Microscopy in Materials Science"

**1993 American Crystallographic Association Annual Meeting, Albuquerque, New Mexico, May 23-28, 1993:**

C. R. HUBBARD,\* T. A. DODSON, X.-L. WANG, S. SPOONER, O. B. CAVIN, AND T. R. WATKINS, "Non-Destructive Residual Stress Mapping Facilities by Neutron and X-Ray Powder Diffraction"

**E. D. SPECHT\* AND F. J. WALKER, "Anomalous Scattering from Surfaces and Interfaces"**

**38th ASME International Gas Turbine and Aeroengine Congress and Exposition Meeting, Cincinnati, Ohio, May 24-27, 1993:**

**C. R. BRINKMAN\* AND G. D. QUINN, "Development of ASTM Standards in Support of Advanced Ceramics Development"**

**D. R. JOHNSON\* AND R. B. SCHULZ, "The Ceramic Technology Project: Ten Years of Progress"**

**G. V. SRINIVASAN,\* S. K. LAU, R. S. STORM, M. K. FERBER, AND M. G. JENKINS, "Process Optimization of Hexoloy SX-SiC Towards Improved Mechanical Properties"**

**General Electric Corporation, Central Research and Development, Schenectady, New York, May 25, 1993:**

**Z. L. WANG,\* J. BENTLEY, R. E. CLAUSING, A. GOYAL, D. M. KROEGER, L. HEATHERLY, JR., L. L. HORTON, J. R. THOMPSON, AND R. K. WILLIAMS, "Microstructures of YBCO Superconductors and As-Grown CVD Diamond Films Studied by Analytical Electron Microscopy"**

**ATS Materials Planning Workshop, Cincinnati, Ohio, May 28, 1993:**

**M. A. KARNITZ, "Overview of Materials Support Planning Activities for DOE-ATS Program"**

**Presentation at the Nuclear Research Center, Rez, Czech Republic, May 31, 1993:**

**W. R. CORWIN, "Heavy-Section Steel Irradiation Program Summary"**

**International Conference on Fundamentals of Fracture (ICFF-IV), Urabandai, Japan, May 31 - June 4, 1993:**

**M. H. YOO\* AND C. L. FU, "Fundamentals of Fracture Behavior in Ordered Intermetallics"**

**Presentation at Skoda Nuclear Machinery Plant, Plzen, Czech Republic, June 1, 1993:**

**W. R. CORWIN, "Heavy-Section Steel Irradiation Program Summary"**

**Workshop on High-Temperature Superconductor (HTS) Phase Diagrams, Santa Fe, New Mexico, June 2-4, 1993:**

**R. K. WILLIAMS, "High-Pressure Studies"**

MPA, University of Stuttgart, Stuttgart, Germany, June 3, 1993:

W. R. CORWIN, "Heavy-Section Steel Irradiation Program Summary"

BAM, Berlin, Germany, June 4, 1993:

W. R. CORWIN, "Heavy-Section Steel Irradiation Program Summary"

SES-AIME-ASCE Symposium on Brittle Matrix Structural Composites, Charlottesville, Virginia, June 6-9, 1993:

C.-H. HSUEH\* AND P. F. BECHER, "Evaluation of Interfacial Properties of Fiber-Reinforced Ceramic Composites"

J. H. SCHNEIBEL\* AND K. B. ALEXANDER, "Composites Based on  $\text{Ni}_3\text{Al}$  and  $\text{Al}_2\text{O}_3$ "

Electricite de France (EDF), Renardiers, France, June 7, 1993:

W. R. CORWIN, "Heavy-Section Steel Irradiation Program Summary"

The Joint Research Committee on Intermetallic Compounds, Sendai, Japan, June 7, 1993:

M. H. YOO\* AND C. L. FU, "Strength of Ordered Intermetallics: Fundamentals"

Advanced Industrial Concepts (AIC) Materials Program Guidance and Evaluation Board Meeting, Oak Ridge, Tennessee, June 7-9, 1993:

K. B. ALEXANDER,\* J. H. SCHNEIBEL, T. N. TIEGS, H. T. LIN, AND P. F. BECHER, "Metallic and Intermetallic Bonded Ceramic Composites"

C. T. LIU,\* J. A. HORTON, AND S. H. WHANG, "Advanced  $\text{NiAl}$  and  $\text{TiAl}$  Intermetallic Alloys"

P. J. MAZIASZ\* AND G. M. GOODWIN, "Development of Weldable, Corrosion-Resistant Iron-Aluminide Alloys"

P. J. MAZIASZ,\* C. G. MCKAMEY, G. M. GOODWIN, C. R. HUBBARD, AND W. D. PORTER, "High-Temperature Precipitate-Strengthened Iron Aluminides and Other Intermetallic Alloys"

V. K. SIKKA, M. L. SANTELLA,\* D. J. ALEXANDER, AND S. VISWANATHAN, " $\text{Ni}_3\text{Al}$  Technology Transfer: Castability and Weldability of  $\text{Ni}_3\text{Al}$ "

Framatom, Paris, France, June 8, 1993:

W. R. CORWIN, "Heavy-Section Steel Irradiation Program Summary"

**Fourth International Conference on Residual Stresses, Baltimore, Maryland, June 8-10, 1993:**

C. R. HUBBARD,\* X.-L. WANG, S. SPOONER, O. B. CAVIN, AND T. R. WATKINS, "Research Opportunities and Facilities at ORNL's Residual Stress User Center"

S. SPOONER,\* S. A. DAVID, X.-L. WANG, C. R. HUBBARD, J. H. ROOT, AND T. M. HOLDEN, "Investigation of Residual Stresses in a Multi-Pass Weld in 1-inch Stainless Steel Plate"

S. SPOONER,\* S. A. DAVID, X.-L. WANG, AND C. R. HUBBARD, "Residual Stresses in a Multi-Pass Weld in Austenitic Stainless Steel Plate"

X.-L. WANG, C. R. HUBBARD,\* K. B. ALEXANDER, P. F. BECHER, J. A. FERNANDEZ-BACA, AND S. SPOONER, "Neutron Diffraction Study of the Transformation Behavior in Alumina-Zirconia Ceramic Composites"

T. R. WATKINS,\* X.-L. WANG, AND C. R. HUBBARD, "Mapping of Residual Stresses of a Ceramic-to-Metal Joint Using X-Ray Diffraction"

**13th Annual Tennessee Valley Section-American Vacuum Society (TVS-AVS) Symposium and Equipment Exhibit, Oak Ridge, Tennessee, June 9-10, 1993:**

K. L. MORE,\* R. A. LOWDEN, N. L. VAUGHN, AND O. J. SCHWARZ, "Alternative Fiber Coatings for Improved Oxidation Resistance in SiC/SiC Composites"

**University of Kentucky Workshop on Theory of Alloys, Lexington, Kentucky, June 10, 1993:**

W. H. BUTLER, "Electrical Resistivity of  $Ni_xFe_{1-x}$  Alloys"

**Harwell Laboratory, Harwell, England, June 11, 1993:**

W. R. CORWIN, "Heavy-Section Steel Irradiation Program Summary"

**21st Biennial Conference on Carbon at the State University of New York, Buffalo, New York, June 13-18, 1993:**

T. D. BURCHELL, "Neutron-Irradiation-Induced Changes in the Dimensions and Thermal Conductivity of Carbon-Carbon Composites"

T. D. BURCHELL, "The Temperature Dependence of Thermal Conductivity in NP-MHTGR Fuel Compacts"

T. D. BURCHELL\* AND W. P. EATHERLY, "Neutron-Irradiation-Induced Changes in the Dimensions and Thermal Conductivity of Carbon-Carbon Composites"

**T. D. BURCHELL\* AND J. P. STRIZAK, "Modelling the Tensile Strength of H-451 Nuclear Graphite"**

**E. L. FULLER, JR., "Coal Composition Analyses by Diffuse Reflectance Infrared Spectroscopy (DRIS)"**

**E. L. FULLER, JR., "Evaluation of Surface Area and Porosity of Carbonaceous Materials"**

**E. L. FULLER, JR.,\* O. C. KOPP, AND A. D. UNDERWOOD, "Microgravimetric Evaluation of Graphite Corrosion Kinetics and Mechanisms"**

**E. L. FULLER, JR.,\* AND K. A. THOMPSON, "Evaluation of Surface Area and Porosity of Carbonaceous Materials"**

**B. T. KELLY AND T. D. BURCHELL,\* "Irradiation Creep in Nuclear Reactor Graphite"**

**D. F. PEDRAZA,\* B. K. ANNIS, AND S. P. WITHROW, "The Topography of Ion-Irradiated Graphite"**

**J. P. STRIZAK, "Tensile Strength Variability in H-451 Nuclear-Grade Graphite"**

**North American Member Management Symposium XIII, Knoxville, Tennessee, June 16, 1993:**

**T. ZACHARIA, "High-Performance Computing for Automotive Applications"**

**Engineering Section Advisory Committee Meeting, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee, June 17 & 30, 1993:**

**V. K. SIKKA, "Materials Processing Activities at the Oak Ridge National Laboratory"**

**1993 Annual Meeting of the American Nuclear Society, San Diego, California, June 20-24, 1993:**

**E. E. BLOOM\* AND A. F. ROWCLIFFE, "Development of Radiation-Resistant Materials"**

**ASTM Seminar on Advanced Ceramics, Atlanta, Georgia, June 23, 1993:**

**A. E. PASTO, "Fabrication of High-Performance Structural Ceramics"**

**International Atomic Energy Agency Coordinated Research Program (CRP2), Vienna, Austria, June 23-25, 1993:**

**B. F. MYERS, "Experiment HFR-B1: A Preliminary Analysis of the Water-Vapor Injection Experiments in Capsule 3"**

B. F. MYERS,\* F. C. MONTGOMERY, AND R. N. MORRIS, "The Distribution Sphere Model"

**Materials Issues for Severe Environments Workshop, University of California, San Diego, California, June 23-25, 1993:**

M. L. GROSSBECK\* AND L. K. MANSUR, "Irradiation Creep in Structural Materials at ITER Operating Conditions"

**Tribology and Surface Engineering Symposium, Massachusetts Institute of Technology, Cambridge, Massachusetts, June 29-30, 1993:**

P. J. BLAU, "Friction: A Multidimensional Phenomenon"

**27th Microbeam Analysis Society (MAS) 1993 Annual Meeting, Los Angeles, California, July 11-16, 1993:**

I. M. ANDERSON,\* J. BENTLEY, AND C. B. CARTER, "Correction of Secondary Fluorescence in the AEM for Common Specimen Geometries"

J. BENTLEY,\* S. MCKERNAN, C. B. CARTER, AND A. REVCOLEVSCHI, "Microanalysis of Directionally Solidified Cobalt Oxide - Zirconia Eutectic"

N. D. EVANS,\* D. L. JOSLIN, AND C. J. MCHARGUE, "Ion Mixing of Thin  $ZrO_2$  Films on Sapphire"

J. JAYARAM\* AND M. K. MILLER, "Applications of the Atom-Probe Field-Ion Microscope to the Atomic Level Characterization of Metals and Alloys"

**28th Microwave Power Symposium, Montreal, Canada, July 12-14, 1993:**

R. J. LAUF,\* F. L. PAULAUSKAS, AND A. C. JOHNSON, "Polymer Curing in a Variable-Frequency Microwave Oven"

**Seminar at the Army Research Laboratory and the Spire Company, Boston, Massachusetts, July 14-15, 1993:**

E. H. LEE, "Super-Hard-Surfaced Polymers by Ion Beam Processing"

**NATO Advanced Research Workshop on Metallic Alloys: Experimental and Theoretical Perspectives, Boca Raton, Florida, July 16-21, 1993:**

C. J. SPARKS,\* G. E. ICE, L. B. SHAFFER, AND J. L. ROBERTSON, "Pair Correlations and Displacements in Crystalline Solid Solutions"

**26th Annual Technical Program of the International Metallographic Society Meeting,  
Charleston, South Carolina, July 18-21, 1993:**

C. G. MCKAMEY, P. J. MAZIASZ, AND S. VISWANATHAN,\* "Microstructure and Fracture Behavior of As-Cast Fe<sub>3</sub>Al-Based Ordered Intermetallic Alloys"

J. M. VITEK,\* S. A. DAVID, AND D. J. ALEXANDER, "Microscopic Evaluation of Low-Temperature Embrittlement in Type 308 Stainless Steel Weldments"

**Gordon Research Conference on Corrosion, New London, New Hampshire, July 19-23, 1993:**

C. G. MCKAMEY AND P. F. TORTORELLI,\* "Pest Oxidation in Polycrystalline MoSi<sub>2</sub>"

P. F. TORTORELLI\* AND K. B. ALEXANDER, "Mechanical Properties of Oxide Scales"

**ASME Pressure Vessels and Piping Conference, Denver, Colorado, July 25-29, 1993:**

R. W. SWINDEMAN, "Overview of Some Design and Materials Issues in Combined-Cycle Applications"

**U.S. DOE Office of Advanced Utility Concepts - Superconducting Technology Program Annual Review Meeting, Washington, D.C., July 27-29, 1993:**

D. M. KROEGER, "Critical Currents and Microstructure"

**Conference on Application of Nanoscale Technologies to Materials Science, Osaka, Japan, July 30, 1993:**

M. K. MILLER,\* R. JAYARAM, AND K. F. RUSSELL, "Atomic-Scale Characterization of Materials with the ORNL Atom Probe"

**ASM 3rd International Conference on Trends in Welding Research on Measurement of Radiation-Induced Grain Boundary Segregation and Influence of Particle Type and Dose, San Diego, California, August 1, 1993:**

G. S. WAS, D. DAMCOTT, D. CARTER, E. A. KENIK, G. E. C. BELL,\* S. M. BRUEMMER, J. S. VETRANO, AND L. A. CHARLOT, "Measurement of Radiation-Induced Grain Boundary Segregation and Influence of Particle Type and Dose"

**6th International Symposium on Environmental Degradation of Materials in Nuclear Power System-Water Reactors, San Diego, California, August 1-5, 1993:**

R. D. CARTER, D. L. DAMCOTT, M. ATZMON, G. S. WAS,\* S. M. BRUEMMER, AND E. A. KENIK, "Capabilities and Limitations of Analytical Methods Used to Measure Radiation-Induced Grain Boundary Segregation"

M. K. MILLER, R. JAYARAM,\* P. J. OTHEN, AND G. BRAUER, "Atom-Probe Field-Ion Microscopy Characterizations of VVER Steels"

R. E. PAWEL,\* D. K. FELDE, AND M. T. MCFEE, "Corrosion Studies on Aluminum Alloy Fuel Cladding for the Advanced Neutron Source"

R. E. STOLLER, "The Role of Point Defect Clusters in Reactor Pressure Vessel Embrittlement"

**40th International Field Emission Symposium, Nagoya, Japan, August 1-6, 1993:**

K. O. BOWMAN, M. K. MILLER,\* AND L. R. SHENTON, "The Evaluation of Moments Methods for Fitting Multiparameter Sinusoidal Models"

A. J. DUNCAN, M. J. KAUFMAN, C. T. LIU, AND M. K. MILLER,\* "Segregation of Interstitial Impurities in Single-Crystal NiAl"

A. J. DUNCAN, M. J. KAUFMAN, C. T. LIU, AND M. K. MILLER,\* "Site Occupation of Iron in Intermetallic NiAl"

J. M. HYDE,\* A. CEREZO, M. K. MILLER, AND G. D. W. SMITH, "Modelling and Analysis of Spinodal Decomposition in the Binary Fe-Cr System"

R. JAYARAM AND M. K. MILLER,\* "An Atom-Probe Field-Ion Microscopy Study of Ball-Milled, Nanocrystalline Ni<sub>3</sub>Al Powders"

M. K. MILLER, "Contributions of the Atom-Probe Technique to the Understanding of Materials Used in Nuclear Applications"

M. K. MILLER,\* R. JAYARAM, P. J. OTHEN, AND G. BRAUER, "APFIM Characterization of 15Kh2MFA Cr-Mo-V- and 15Kh2NMFA Ni-Cr-Mo-V-Type Steels"

M. K. MILLER,\* L. S. LIN, AND A. D. CETEL, "APFIM Characterization of Single-Crystal PWA 1480 Nickel-base Superalloy"

**51st Annual Meeting of the Microscopy Society of America (MSA), Cincinnati, Ohio, August 1-6, 1993:**

J. BENTLEY, "High-Resolution Microscopy of Ceramic Surfaces"

J. BENTLEY,\* H. M. PHILLIPS, D. L. CALLAHAN, AND R. SAUERBREY, "Electron Energy-Loss Spectrometry of Laser-Irradiated Kapton Polyimide"

M. G. BURKE\* AND E. A. KENIK, "Intergranular Segregation in Pressure Vessel Steels"

M. G. BURKE\* AND M. K. MILLER, "Applications of Atom-Probe Field-Ion Microscopy to the Study of Interfaces"

N. D. EVANS,\* J. BENTLEY, AND A. T. FISHER, "A Vacuum Modification for the Philips CM30 Electron Microscope"

N. D. EVANS,\* D. L. JOSLIN, AND C. J. MCHARGUE, "Ion Mixing of Thin  $ZrO_2$  Films on  $\alpha\text{-Al}_2\text{O}_3$  by  $Cr^+$  or  $Kr^+$  Ions"

E. A. KENIK,\* L. F. ALLARD, AND J. BENTLEY, "Hole Count and Secondary Excitation in Several Analytical Electron Microscopes"

E. A. KENIK,\* D. F. PEDRAZA, AND S. P. WITHROW, "Radiation Damage in Ion-Irradiated Pyrolytic Graphite"

M. P. MALLAMACI,\* J. BENTLEY, AND C. B. CARTER, "In Situ Crystallization of Silicate Glass Films Deposited on  $\alpha\text{-Al}_2\text{O}_3$ "

M. P. MALLAMACI,\* J. BENTLEY, AND C. B. CARTER, "Microanalysis of Silicate Glass Films Grown on  $\alpha\text{-Al}_2\text{O}_3$  by Pulsed-Laser Deposition"

Z. L. WANG, "Dislocation Contrast in High-Angle, Hollow-Cone Dark-Field TEM"

Z. L. WANG,\* J. BENTLEY, R. E. CLAUSING, L. HEATHERLY, JR., AND L. L. HORTON, "Reflection Electron Microscopy of As-Grown Diamond Surfaces"

Z. L. WANG,\* J. BRYNESTAD, D. M. KROEGER, Y. R. SUN, J. R. THOMPSON, AND R. K. WILLIAMS, "Nano-Probe Microanalysis of Grain Boundary Chemistry in  $YBa_2Cu_4O_8$  and Its Relationship to Weak-Link Behavior"

Z. L. WANG,\* R. KONTRA, A. GOYAL, D. M. KROEGER, AND L. F. ALLARD, "Flux-Pinning-Related Defect Structures in Melt-Processed  $YBa_2Cu_3O_{7-x}$ "

International Summer School on the Fundamentals of Radiation Damage, Champaign/Urbana, Illinois, August 1-12, 1993:

L. K. MANSUR, "Theory of Dimensional Changes Caused by Defect Reactions in Irradiated Alloys"

**42nd Annual Denver X-Ray Conference, Denver, Colorado, August 2-6, 1993:**

C. R. HUBBARD,\* X.-L. WANG, S. SPOONER, AND J. JO, "Comparison of Residual Stresses within HY-100 Weldments Prepared With and Without Vibratory Stress Relief"

T. R. WATKINS,\* C. R. HUBBARD, AND J. L. BJERKE, "Measurement of an Elastic Constant in Plasma-Sprayed Zirconia Coatings Using X-Ray Diffraction"

J. S. WOLF AND O. B. CAVIN,\* "The Effective Thermal Expansion of Nickel and Nickel Oxide During High-Temperature Oxidation"

**Advanced Turbine Systems, Heat Engines, and Fuel Cells Review Meeting, Morgantown Energy Technology Center, Morgantown, West Virginia, August 3-5, 1993:**

M. A. KARNITZ,\* J. H. DEVAN, R. W. HARRISON, M. K. FERBER, AND R. S. HOLCOMB, "Materials/Manufacturing Element of the Advanced Turbine Systems Program"

**8th International Meeting of Ferroelectrics - NIST, Gaithersburg, Maryland, August 8-13, 1993:**

X. ZHANG,\* D. C. JOY, L. F. ALLARD, AND T. A. NOLAN, "Study of Ferroelectric Domain Wall Structures Using Electron Holographic Techniques"

**Conference on Atom-Probe Applications to Materials Science, Sendai, Japan, August 9-10, 1993:**

M. K. MILLER,\* R. JAYARAM, AND K. F. RUSSELL, "Applications of the ORNL Atom Probe to Materials Characterization"

**1993 American Institute of Chemical Engineers (AIChE) Summer National Meeting, Seattle, Washington, August 15-18, 1993:**

E. L. FULLER, JR., "Evaluation of Surface Area and Porosity of Carbonaceous Materials from Inert Gas Adsorption Isotherms"

E. L. FULLER, JR., "Methodology for Evaluation of Surface Area and Porosity of Carbon Materials from Inert Gas Adsorption Isotherms"

**International Thermonuclear Experimental Reactor Steering Committee United States-United States Technical Advisory Committee (ISCUS/U.S.-TAC) Meeting, UCLA, Los Angeles, California, August 16-17, 1993:**

E. E. BLOOM,\* R. L. KLUEH, A. F. ROWCLIFFE, AND D. S. GELLES, "Status of Development, Major Issues, and Required R&D for the Use of Ferritic/Martensitic Steels for ITER"

**XVI International Congress and General Assembly International Union of Crystallography,  
Beijing, China, August 21-29, 1993:**

C. J. SPARKS,\* G. E. ICE, P. ZSCHACK, L. ROBERTSON, AND L. SHAFFER,  
"Separation of Displacement Pair Correlations in Crystalline Solid Solutions by  
Anomalous Diffuse X-Ray Scattering"

**Office of Basic Energy/Office of Fusion Energy (OBES/OFE) Workshop on the Evaluation  
and Development of Interatomic Potentials for Large-Scale Simulations of Non-Equilibrium  
Phenomena in SiC, Santa Barbara, California, August 23-25, 1993:**

R. E. STOLLER\* AND L. L. SNEAD, "Microstructural and Mechanical Property  
Changes in Irradiated SiC"

**Fine Particle Society Meeting, Chicago, Illinois, August 23-27, 1993:**

A. BLEIER, "Rheology of Concentrated Ceramic Suspensions"

**Central Iron and Steel Research Institute, Beijing, China, August 25, 1993:**

Z. L. WANG,\* J. BENTLEY, R. E. CLAUSING, A. GOYAL, D. M. KROEGER,  
L. HEATHERLY, JR., L. L. HORTON, J. R. THOMPSON, AND R. K. WILLIAMS,  
"Microstructures of YBCO Superconductors and As-Grown CVD Diamond Films  
Studied by Analytical Electron Microscopy"

**The Residual Stress Workshop, Oak Ridge National Laboratory, Oak Ridge, Tennessee,  
August 26-27, 1993:**

C. R. HUBBARD, "X-Ray Safety Instruction - Resources, Methods, and  
Responsibilities with a Government Laboratory Perspective"

C. R. HUBBARD,\* X.-L. WANG, T. A. DODSON, T. R. WATKINS, O. B. CAVIN, AND  
S. SPOONER, "Nondestructive Residual Stress Mapping by X-Ray and Neutron  
Diffraction"

**Beijing Laboratory of Electron Microscopy, Beijing, China, August 27, 1993:**

Z. L. WANG,\* J. BENTLEY, R. E. CLAUSING, A. GOYAL, D. M. KROEGER,  
L. HEATHERLY, JR., L. L. HORTON, J. R. THOMPSON, AND R. K. WILLIAMS,  
"Microstructures of YBCO Superconductors and As-Grown CVD Diamond Films  
Studied by Analytical Electron Microscopy"

**46th Annual Assembly 1993, The International Institute of Welding (IIM), Glasgow,  
Scotland, August 28 - September 4, 1993:**

S. A. DAVID,\* J. M. VITEK, T. ZACHARIA AND T. DEBROY, "Weld Pool  
Phenomena"

**Environmentally Conscious Manufacturing (ECM) '93, Arlington, Virginia, August 29 - September 1, 1993:**

H. W. HAYDEN,\* M. J. STEPHENSON, J. K. BAILEY, J. R. WEIR, JR., AND W. C. GILBERT, "Contaminated Scrap Metal Management on the Oak Ridge Reservation"

**Eighth American Society for Testing and Materials-European Atomic Energy Community (ASTM-EURATOM) Symposium on Reactor Dosimetry, Vail, Colorado, August 29 - September 3, 1993:**

F. B. KAM,\* K. FARRELL, C. A. BALDWIN, R. W. HOBBS, L. P. PUGH, R. W. STALLMAN, L. ROBINSON, F. F. DYER, F. M. HAGGAG, J. B. PACE, III, B. M. OLIVER, L. R. GREENWOOD, AND E. D. MCGARRY, "Neutron Dosimetry Data from the HFIR Pressure Vessel"

**U.S. DOE/JAERI HTGR Collaboration Agreement Annex 3 Graphite Development Testing Coordinating Committee Meeting, JAERI Oarai Research Establishment, Oarai, Japan, August 31, 1993:**

T. D. BURCHELL, "Update on the USDOE MHTGR Program"

**Powder Diffraction, Satellite Meeting of the XVIth IUCr Congress, Hangzhou, China, August 31 - September 3, 1993:**

C. J. SPARKS,\* S. KHOSLA, E. SPECHT, G. E. ICE, AND P. ZSCHACK, "Recovery of Site Occupation Parameters in Ternary Alloys with Anomalous (Resonance) X-Ray Scattering"

**3rd International Union of Materials Research Society (IUMRS) Conference on Advanced Materials, Tokyo, Japan, August 31 - September 4, 1993:**

D. S. EASTON, C. T. LIU,\* E. P. GEORGE, J. J. CAMPBELL, AND C. A. CARMICHAEL, "Processing and Characterization of Ni-Al-Fe-B Shape-Memory Alloy Wires Produced by Rapid Solidification"

E. P. GEORGE,\* C. T. LIU, J. A. HORTON, C. J. SPARKS, H. KUNSMANN, AND T. KING, "Mechanical Behavior and Phase Stability of NiAl-Based Shape-Memory Alloys"

C. T. LIU, "Recent Advances in Ordered Intermetallics"

C. J. MCHARGUE,\* D. L. JOSLIN, J. M. WILLIAMS, AND M. E. O'HERN, "Surface Modification of Sapphire for Enhanced Infrared Window Performance"

T. N. TIEGS,\* S. D. NUNN, C. A. WALLS, D. BARKER, C. DAVISSON, AND P. J. JONES, "Effect of Powder Characteristics on Gas-Pressure Sintering of  $\text{Si}_3\text{N}_4$  With Rare-Earth Additives"

**Continuous Fiber Ceramic Composites (CFCC) Working Group Meeting, Knoxville, Tennessee, September 1, 1993:**

D. J. MCGUIRE, "CFCC Test Methods - 2.3.3b Nondestructive Characterization"

**U.S. DOE/JAERI HTGR Collaboration Agreement Annex 3 Graphite Development Testing Coordinating Committee Meeting, JAERI Tokai Research Establishment, Tokai, Japan, September 1, 1993:**

T. D. BURCHELL, "Recent MHTGR Graphite Technology Development Activities at ORNL"

**Beijing Laboratory of Electron Microscopy, Beijing, China, September 2, 1993:**

Z. L. WANG,\* A. GOYAL, R. KONTRA, D. M. KROEGER, J. R. THOMPSON, AND R. K. WILLIAMS, "Physical Property Related Microstructures of YBCO Superconductors and Ceramic Surfaces Determined by HRTEM and Associated Analytical Techniques"

**Presentation at Howmedica, Rutherford, New Jersey, September 2, 1993:**

E. H. LEE, "Hard-Surfaced Polymers by Ion Beam Processing"

**Seminar at Ibaraki University, Katsuta, Japan, September 2, 1993:**

T. D. BURCHELL, "Neutron Irradiation Damage in Carbon-Carbon Composites"

**Vermont Department of Economic Development, Montpelier, Vermont, September 3, 1993:**

J. R. WEIR, JR., "The Federal Laboratories and Their Capabilities in Technological Assistance to the United States Business Community"

**7th International Conference of Radiation Effects of Insulators (REI-7), Nagoya, Japan, September 5-10, 1993:**

D. L. JOSLIN, C. J. MCHARGUE,\* AND C. W. WHITE, "Ion Mixing in Oxide-Sapphire Systems"

D. L. JOSLIN, C. J. MCHARGUE,\* C. W. WHITE, AND N. D. EVANS, "Amorphization of Sapphire During Ion Beam Mixing"

C. J. MCHARGUE,\* D. L. JOSLIN, AND C. W. WHITE, "Ion Beam Mixing in Insulator Substrates"

S. J. ZINKLE, "Microstructure of Ion-Irradiated Oxide Ceramics"

**Heart of Texas Chapter of ASM, Austin, Texas, September 8, 1993:**

L. L. HORTON, "Materials Science and Engineering R&D at Oak Ridge National Laboratory - From Basic Science to Industrial Collaborations"

**North American Conference on Molecular Beam Epitaxy, Stanford University, Stanford, California, September 13-15, 1993:**

R. A. MCKEE,\* F. J. WALKER, AND K. B. ALEXANDER, "The MBE Growth and Optical Quality of  $\text{BaTiO}_3$  and  $\text{SrTiO}_3$  Thin Films on  $\text{MgO}$ "

**Surface Modification of Metals by Ion Beams '93, Kanazawa, Japan, September 13-17, 1993:**

C. J. MCHARGUE,\* D. L. JOSLIN, J. E. PAWEL, AND L. ROMANA, "Design of Metal/Sapphire Interfaces for Enhanced Adhesion"

**Electron Microscopy and Analysis Group (EMAG) '93, British Institute of Physics, Liverpool, England, September 14-17, 1993:**

J. BENTLEY,\* S. MCKERNAN, C. B. CARTER, AND A. REVCOLEVSCHI, "Microanalysis of an Oxidized Cobalt Oxide-Zirconia Eutectic"

J. BENTLEY,\* S. TANAKA, AND R. F. DAVIS, "Electron Microscopy of  $\text{AlN-SiC}$  Interfaces and Solid Solutions"

**ASM International Oak Ridge Chapter Meeting, Oak Ridge, Tennessee, September 16, 1993:**

D. F. CRAIG, "Observations on the Future of Materials Science and Engineering at Oak Ridge"

**Seminar at University of Glasgow, Glasgow, Scotland, September 20, 1993:**

J. BENTLEY, "Materials Characterization by Electron Energy-Loss Spectrometry"

**Diamond Films '93, Algarve, Portugal, September 20-24, 1993:**

D. J. KESTER, K. S. AILEY, R. F. DAVIS,\* AND K. L. MORE, "Ion-Beam-Assisted MBE and Phase Evolution of BN Thin Films"

T. J. KREUTZ,\* K. WANDELT, R. E. CLAUSING, AND L. HEATHERLY, JR., "Diamond(111): Growth Mechanisms and Defects Identified by Means of Scanning Tunneling Microscopy"

**High-Temperature Ceramic Matrix Composites International Conference, Bordeaux, France, September 20-24, 1993:**

T. M. BESMANN,\* D. P. STINTON, AND R. A. LOWDEN, "Overview of Chemical Vapor Infiltration"

**Space Nuclear Power and Propulsion Technologies - Materials and Fuels, Podolsk, Moscow, September 21-24, 1993:**

J. A. HORAK\* AND L. K. EGNER, "Studies to Improve the Strength of Nb-1Zr for Space Nuclear Power"

J. A. HORAK\* AND M. L. GROSSBECK, "Effects of Irradiation on the Tensile Properties of Nb-1 wt % Zr"

**Seminar at University of Birmingham, Birmingham, England, September 22, 1993:**

J. BENTLEY, "Materials Characterization by Electron Energy Loss Spectrometry"

**Joint UT/ORNL Ultra-High Resolution Scanning Electron Microscopy (SEM) Workshop, Knoxville, Tennessee, September 23-24, 1993:**

K. B. ALEXANDER, "Low-Voltage Image Contrast Mechanisms in Ceramic Composites"

L. F. ALLARD, E. VÖLKL,\* AND A. K. DATYE, "High-Resolution SEM Imaging of Model Catalytic Materials"

**Seminar at Max-Planck Institute, Stuttgart, Germany, September 26, 1993:**

G. M. STOCKS,\* W. A. SHELTON, D. M. NICHOLSON, Y. WANG, Z. SZOTEK, AND W. M. TEMMERMAN, "LDA on Parallel Machines"

**III Congreso Nacional de Ciencia de Materiales, Cancun, Mexico, September 26-30, 1993:**

W. C. OLIVER,\* B. N. LUCAS, AND G. M. PHARR, "The Mechanical Characterization on the Submicron Scale Using Indentation Experiments"

**International Symposium on Structural Intermetallics, Champion, Pennsylvania, September 26-30, 1993:**

C. L. FU AND M. H. YOO\*, "Fundamentals of Mechanical Behavior in Structural Intermetallics - A Synthesis of Atomistic and Continuum Modeling"

E. P. GEORGE,\* C. T. LIU, AND D. P. POPE, "A Reexamination of Grain Boundary Fracture in Ni<sub>3</sub>Al: Environmental and Alloying Effects"

N. L. PETOUHOFF,\* A. J. ARDELL, W. C. OLIVER, AND B. N. LUCAS, "Miniaturized Disk-Bend Testing, Nano-Indentation and the Microstructure of Ion-Irradiated Titanium Aluminides"

V. K. SIKKA\* AND C. G. MCKAMEY, "Development and Commercialization Status of Fe<sub>3</sub>Al-Based Intermetallic Alloys"

Third International Conference on Near-Net-Shape Manufacturing, Pittsburgh, Pennsylvania, September 27, 1993:

S. VISWANATHAN, "The Prediction of Porosity in the Computer-Aided-Design and Analysis of Casting Processes"

6th International Conference on Fusion Reactor Materials, Stresa, Italy, September 27 - October 1, 1993:

J. E. PAWEL,\* D. J. ALEXANDER, M. L. GROSSBECK, A. F. ROWCLIFFE, AND K. SHIBA, "Fracture Toughness of Candidate Materials for ITER First Wall/Blanket Structures"

V. R. BARABASH, S. A. FABRITSIEV, A. S. POKROVSKY, S. J. ZINKLE,\* AND F. A. GARNER, "Description of a Cu Alloy Spectral-Tailoring Experiment in a Mixed-Spectrum Reactor"

M. I. DE VRIES AND R. L. KLUEH,\* "Tensile Properties of Low-Temperature Neutron-Irradiated Ferritic-Martensitic Steels"

M. L. GROSSBECK,\* B. E. NELSON, AND B. A. CHIN, "Considerations for Repair Welding in Tokamak Fusion Reactors"

S. JITSKUAWA, K. SHIBA, AND J. E. PAWEL,\* "On Post-Irradiation Ductility of Austenitic Stainless Steels"

Y. KATOH,\* R. E. STOLLER, AND A. KOHYAMA, "A Composite Model of Point Defect Agglomeration and Extended Defect Evolution in Irradiated Austenitics"

E. A. KENIK\* AND G. E. C. BELL, "Irradiation-Assisted Stress Corrosion Cracking"

R. L. KLUEH\* AND D. J. ALEXANDER, "Charpy Impact Behavior of Irradiated Reduced-Activation Steels"

R. L. KLUEH\* AND D. J. ALEXANDER, "Effects of Vanadium and Titanium on Chromium-Tungsten Steels"

R. L. KLUEH\* AND E. A. KENIK, "Thermal Stability of Manganese-Stabilized Stainless Steels"

K. SHIBA, S. JITSKUWA, J. E. PAWEL,\* AND A. F. ROWCLIFFE, "Tensile Properties of Base and Weld Metal Specimens of Austenitic Stainless Steels Irradiated in the High Flux Isotope Reactor"

L. L. SNEAD\* AND T. D. BURCHELL, "Degradation of High-Thermal-Conductivity Graphite Materials"

L. L. SNEAD\* AND M. C. OSBORNE, "Irradiation Effects on Continuous Silicon Carbide Fibers"

L. L. SNEAD,\* D. P. WHITE, AND S. J. ZINKLE, "In-Core Measurement of DC Electrical Conductivity of Ceramics"

D. P. WHITE, "The Effect of Ionizing and Displacive Radiation on the Thermal Conductivity of Alumina at Low Temperature"

S. J. ZINKLE, "Gas-Assisted Cavity Formation in  $\text{Al}_2\text{O}_3$  and  $\text{MgAl}_2\text{O}_4$ "

S. J. ZINKLE, "Microstructural Evidence for Ionization-Enhanced Diffusion in Oxide Ceramics"

S. J. ZINKLE\* AND Y. KATANO, "Threshold Dose for Observable Defect Cluster Formation in Ion-Irradiated  $\text{Al}_2\text{O}_3$ "

S. J. ZINKLE,\* A. HORSEWELL, B. N. SINGH, AND W. F. SOMMER, "Defect Microstructure in Copper Alloys Irradiated with 750-MeV Protons"

**Ad Hoc Workshop on Ceramics for Li/FeS<sub>2</sub> Batteries, Oak Brook, Illinois, September 29, 1993:**

D. F. WILSON,\* R. J. LAUF, AND J. H. DEVAN, "Compatibility of Ceramic Insulators with Lithium"

**Hoescht AG Materials Research, Frankfurt, Germany, September 30, 1993:**

V. J. TENNERY, "Research and Development Activities in the High Temperature Materials Laboratory"

**Ceramics Afternoon Seminar, Cornell University, Ithaca, New York, October 1, 1993:**

J. H. SCHNEIBEL, "Processing Considerations and Mechanical Properties of Intermetallic/Ceramic Composites"

**Silicon Nitride '93 Interaction Conference, Stuttgart, Germany, October 4-6, 1993:**

P. F. BECHER,\* S.-L. HWANG, AND H. T. LIN, "Microstructural Contributions to the Fracture Resistance of Silicon Nitride Ceramics"

**Third International Workshop on Long-Term Thermal Performance of Cellular Plastics,  
Toronto, Canada, October 4-6, 1993:**

**R. S. GRAVES\* AND D. W. YARBROUGH, "The Use of an Array of Heat Flux  
Transducers to Study Thermal Property Variations in Rectangular Test Specimens"**

**D. W. YARBROUGH,\* R. S. GRAVES, AND M. T. BOMBERG, "A Comparison of  
Time-Average R-Values for Foamboard Insulation Predicted by the IRC Model with  
Measured Values from ORNL"**

**U.S. DOE Plasma Facing Materials and Components Task Group Meeting, Livermore,  
California, October 5-6, 1993:**

**T. D. BURCHELL, "CFC Development and Radiation Damage: Loss of Thermal  
Conductivity"**

**First Annual Symposium of the East Tennessee Section of the MRS, Oak Ridge,  
Tennessee, October 7-8, 1993:**

**G. M. LUDTKA, "Superplasticity, Superplastic Forming (SPF), and Supercomputer  
Modeling of SPF"**

**184th High-Temperature Materials Meeting, New Orleans, Louisiana, October 10-15, 1993:**

**A. A. WERESZCZAK,\* K. BREDER, M. K. FERBER, T. P. KIRKLAND, AND  
P. KHANDELWAL, "Environmental Effects on the Flexure Strength of HIPed Silicon  
Nitride at Elevated Temperatures"**

**Pressure Vessel Research Committee (PVRC) Meeting, New York, New York, October 11,  
1993:**

**D. E. MCCABE, "New Concepts in Transition Temperature Definition"**

**R. K. NANSTAD, J. A. KEENEY, AND D. E. MCCABE,\* "Preliminary Review of the  
Bases for the  $K_{Ic}$  and  $K_{IR}$  Curves in the ASME Code"**

**Graduate Seminar Program Presentation, University of Cincinnati, Cincinnati, Ohio,  
October 15, 1993:**

**V. K. SIKKA, "Development of Intermetallic Compounds"**

**1993 ASM-TMS Fall Meeting, Pittsburgh, Pennsylvania, October 17-21, 1993:**

**D. J. ALEXANDER, "High-Temperature Fracture of Nickel Aluminide Alloys"**

**D. J. ALEXANDER\* AND V. K. SIKKA, "Fracture Behavior of Iron-Aluminum Alloys"**

K. B. ALEXANDER AND L. L. HORTON,\* "Education Programs in Materials Science"

K. B. ALEXANDER,\* H. T. LIN, J. H. SCHNEIBEL, AND P. F. BECHER, "Fabrication and Properties of Alumina Matrix Composites Containing Nickel-Aluminide Reinforcements"

S. CAO,\* C. R. BROOKS, AND L. F. ALLARD, "Effect of Plastic Deformation on the Ordering Reaction in  $Ni_4Mo$ "

A. E. CARLSSON, J. ZOU, AND R. PHILLIPS,\* "Consequences of Oscillatory Potentials and Angular Forces in Transition Metals and Their Aluminides"

T. M. CHEEK, P. DEWO, M. J. KAUFMAN,\* AND N. D. EVANS, "Microstructural Evolution and Thermal Stability of Rapidly Solidified Al-Fe Based Alloys"

C. L. FU,\* Y.-Y. YE, AND M. H. YOO, "Point Defects in Alloys from First-Principles Calculations"

C. L. FU AND M. H. YOO,\* "Bulk and Defect Properties of Ordered Intermetallics"

A. N. GUBBI,\* E. P. GEORGE, E. H. LEE, E. K. OHRINER, AND R. H. ZEE, "Effects of Cerium on High-Temperature Impact Ductility and Fracture of Iridium Alloys"

J. HYDE, A. CEREZO, G. D. W. SMITH, AND M. K. MILLER,\* "Three-Dimensional Atomic-Scale Microanalysis and Computer Modelling of Spinodal Decomposition in Iron-Chromium Alloys"

G. E. ICE,\* C. J. SPARKS, L. SHAFFER, AND P. ZSCHACK, "Pair Correlations in Crystalline Solid Solutions"

R. JAYARAM,\* C. T. LIU, AND M. K. MILLER, "An Atom-Probe Investigation of the Influence of Trace Impurities on the Mechanical Behavior of NiAl"

R. G. JORDAN,\* S. L. QIU, Y. LIU, E. L. HINES, AND G. M. STOCKS, "The Origin and Stabilization of Long-Period Superlattices in Ag-Mg Alloys"

D. M. KROEGER,\* A. GOYAL, AND Z. L. WANG, "Microstructures and Critical Current Densities in High- $T_c$  Oxides"

Y. LIN AND E. P. GEORGE,\* "Effect of Environment and Alloy Stoichiometry on Mechanical Behavior and Grain Boundary Chemistry of B-Doped FeAl"

C. T. LIU,\* J. A. HORTON, AND E. P. GEORGE, "Alloy Design of Polycrystalline NiAl Alloys"

M. K. MILLER\* AND R. JAYARAM, "Microstructural Characterization of Nickel Aluminides with the Atom-Probe Field-Ion Microscope"

J. R. MORRIS,\* Y. Y. YE, K. M. HO, C. T. CHAN, AND M. H. YOO, "First-Principles and Molecular Dynamics Studies of Twin Boundaries in hcp Zirconium"

E. K. OHRINER, "Processing of High-Purity Iridium Alloys"

J. H. SCHNEIBEL\* AND C. T. LIU, "Structure, Stability, and Mechanical Properties of Intermetallic Phases"

J. H. SCHNEIBEL\* AND P. J. MAZIASZ, "Fracture Toughness and Fracture Mode of FeAl Intermetallics"

V. K. SIKKA, "Casting and Properties of Nickel-Aluminide Alloys"

V. K. SIKKA, "Nickel and Iron Aluminides for High-Temperature Aggressive Environments"

C. J. SPARKS, S. KHOSLA, J. H. SCHNEIBEL,\* E. D. SPECHT, G. E. ICE, AND P. ZSCHACK, "Site Substitution and Phase Stability for Third-Element Additions to Binary Intermetallics"

R. W. SWINDEMAN, "Evaluation of Structural Materials for Use in Second-Generation Combined-Cycle Applications"

P. E. A. TURCHI,\* M. SLUITER, AND G. M. STOCKS, "Effect of Pressure on Stability and Order in Substitutional Alloys"

S. VISWANATHAN,\* V. K. SIKKA, J. K. WRIGHT, AND R. N. WRIGHT, "Effect of Heat Treatment on the Microstructure and Room-Temperature Tensile Properties of an Fe<sub>3</sub>Al-Based Alloy"

M. H. YOO,\* C. L. FU, AND J. K. LEE, "Elastic Properties of Twin Dislocations in Titanium Aluminides"

1993 Automotive Technology Development Contractor's Coordination Meeting (ATD/CCM), Dearborn, Michigan, October 18, 1993:

P. J. BLAU, "Cost-Effective Ceramic Machining Project Update"

ACerS Symposium on Design of Experiment, Worcester, Massachusetts, October 20, 1993:

A. E. PASTO, "Structural Ceramics Processing Improvements via Taguchi Methods"

**Society of Tribologist and Lubrication Engineers-American Society of Mechanical Engineers (STLE-ASME) Tribology Conference, New Orleans, Louisiana, October 24-27, 1993:**

P. J. BLAU, "On the Origins and Modeling of Frictional Behavior in Interfacially Complex Sliding Contacts"

**JAERI Management Team, Oak Ridge National Laboratory, Oak Ridge, Tennessee, October 25, 1993:**

T. D. BURCHELL, "Past and Present Collaboration Under the U.S. DOE/JAERI/MHTGR Agreement: Graphite Technology Activities"

**University of Connecticut Seminar, Storrs, Connecticut, October 25, 1993:**

J. M. VITEK, "Basic Energy Sciences Welding Research at Oak Ridge National Laboratory"

**Vermont Department of Economic Development, Burlington, Vermont, October 25, 1993:**

J. R. WEIR, JR., "The Federal Laboratories and Their Capabilities in Technological Assistance to the United States Business Community"

**21st Water Reactor Safety Information Meeting, Bethesda, Maryland, October 25-27, 1993:**

W. R. CORWIN,\* R. K. NANSTAD, AND D. E. MCCABE, "Heavy-Section Steel Irradiation Program Progress - Recent Results from Midland Low Upper-Shelf Weld Studies"

**Joint Coordinating Committee on Civilian Nuclear Reactor Safety (JCCCNRS) Working Group 3, Rockville, Maryland, October 25-29, 1993:**

S. K. ISKANDER,\* M. A. SOKOLOV, R. K. NANSTAD, AND W. R. CORWIN, "Heavy-Section Steel Irradiation Program Annealing and Reembrittlement Studies at the Oak Ridge National Laboratory"

D. E. MCCABE, "An ASTM 'Proposed Test Practice' for Fracture Mechanics Testing in the Ductile-Brittle Transition Temperature Range"

M. K. MILLER, "Atom-Probe Field-Ion Microscopy Studies of U.S. and Russian Pressure Vessel Steels"

R. K. NANSTAD\* AND J. A. JOYCE, "Preliminary Test Results for Working Group 3 Round Robin on J-R Curve"

M. A. SOKOLOV,\* S. K. ISKANDER, AND R. K. NANSTAD, "Heavy-Section Steel Irradiation Program Annealing and Reembrittlement Studies with Previously Irradiated Materials and Working Group 3 Activities"

M. A. SOKOLOV\* AND R. K. NANSTAD, "Specimen Size Effects from Charpy Impact Tests and Preliminary Results for HSST Plate 02"

**Science and Technology Alliance Materials Conference '93, North Carolina AT&T State University, Greensboro, North Carolina, October 27-29, 1993:**

D. R. JOHNSON\* AND R. B. SCHULZ, "Ceramic Technology for Advanced Heat Engines"

K. C. LIU,\* C. O. STEVENS, AND C. R. BRINKMAN, "Enhancement of Creep Resistance of a Sintered Silicon Nitride Ceramic by Microwave Annealing"

D. P. STINTON,\* J. C. MCLAUGHLIN, B. FOSTER, K. REIFSNIDER, W. STINCHCOMB, AND K. LIAO, "Fabrication of Tubular SiC/SiC Composites by Chemical Vapor Infiltration"

**Oklahoma Center for the Advancement of Science and Technology, Edmond, Oklahoma, October 29, 1993:**

J. R. WEIR, JR., "Technology Transfer from Large Federal Labs and the Economic Development Consequences"

**35th APS Annual Meeting, St. Louis, Missouri, November 1-5, 1993:**

S. J. ZINKLE,\* R. H. GOULDING, AND R. E. STOLLER, "In-Reactor Measurements of the Effects of Neutron and Gamma Radiation on the RF Dielectric Characteristics of Ceramic Insulators"

**Japan Fine Ceramics Center Seminar, Nagoya, Japan, November 2, 1993:**

A. A. WERESZCZAK, "The Effect of Oxidation on Yttrium Silicate Grain Boundaries in HIPed Silicon Nitride and Its Role in Dictating Creep Lifetime"

**University of Tennessee Space Institute Seminar, Knoxville, Tennessee, November 2, 1993:**

V. J. TENNERY\* AND K. BREDER,\* "Research at ORNL Related to High-Performance Materials in Coal Conversion/Utilization"

**National Educators Workshop, NASA Langley Research Center, Hampton, Virginia, November 3-5, 1993:**

T. D. BURCHELL, "Developments in Carbon Materials"

**Oak Ridge Center for Manufacturing Techology Meeting, Y-12 Plant, Oak Ridge, Tennessee, November 5, 1993:**

**T. D. BURCHELL, "Carbon-Fiber Composite Molecular Sieves: An Opportunity for a Unique ORNL/Y-12/Industry Collaboration"**

**22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993:**

**S. C. BEECHER,\* R. B. DINWIDDIE, JR., AND A. M. ABEEL, "An Automated, Guarded Longitudinal Heat Flow Instrument to Measure Thermal Conductivity in the Temperature Range 85 to 500 K"**

**S. C. BEECHER,\* R. B. DINWIDDIE, JR., A. M. ABEEL, AND R. A. LOWDEN, "The Thermal Conductivity of Silicon Nitride with Molybdenum Disilicide Additions"**

**S. C. BEECHER,\* R. B. DINWIDDIE, JR., AND R. A. LOWDEN, "The Thermal Conductivity of Carbon-Coated Silicon Carbide Fibers Embedded in a Silicon Carbide Matrix"**

**R. B. DINWIDDIE, JR., "Microscopic Thermal Conductivity Measurement"**

**R. B. DINWIDDIE, JR.,\* S. C. BEECHER, AND P. ARYA, "The Thermal Conductivity of Carbon-Fiber-Reinforced Metal Matrix Composites"**

**R. B. DINWIDDIE, JR.,\* H. EATON, AND J. LINSEY, "The Effect of Thermal Aging on the Thermal Conductivity of Plasma-Sprayed Fully Stabilized Zirconia"**

**R. S. GRAVES, K. E. WILKES,\* AND D. L. MCELROY, "Thermal Resistance Decreases in Attic Loose-Fill Insulations Under Simulated Winter Conditions"**

**R. S. GRAVES\* AND T. G. KOLLIE, "Interlaboratory Comparison Measurements of the Thermal Conductivity of Powder-Filled Evacuated Panel Superinsulation"**

**T. E. WHITAKER,\* R. S. GRAVES, D. L. MCELROY, AND D. R. SMITH, "Interlaboratory Comparison of the Horizontal Pipe Insulation Test Apparatus Up to 350°C"**

**D. W. YARBROUGH,\* R. K. WILLIAMS, AND D. R. SHOCKLEY, "Thermal Conductivities, Electrical Resistivities, and Seebeck Coefficients of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  Superconductors from 80 to 300 K"**

**First Pacific Rim International Conference on Processing Materials for Properties, Honolulu, Hawaii, November 7-11, 1993:**

**P. F. BECHER,\* C.-H. HSUEH, AND K. B. ALEXANDER, "The Role of Microstructure in Toughened Ceramics"**

G. CARRASQUILLO,\* S. C. DANFORTH, L. F. ALLARD, T. A. NOLAN, W. CHEN, S. J. DAPKUNAS, G. J. PIERMARINI, S. G. MALGHAN, AND A. PECHENIK, "High-Pressure Consolidation of Laser-Derived Nanosized Silicon Nitride Powder"

J. A. HORAK, "Effect of Processing History on the Creep Strength of Nb-1Zr"

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M. G. JENKINS,\* K. BREDER, J. A. SALEM, AND V. J. TENNERY, "Elevated-Temperature Macro-Flaw Fracture and Tensile Creep/Creep Rupture Behaviours of a Self-Reinforced Silicon Nitride"

**1993 AIChE Annual Meeting, St. Louis, Missouri, November 7-12, 1993:**

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**GT-MHR Graphite Technical Review Meeting, General Atomics, San Diego, California, November 9, 1993:**

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T. D. BURCHELL, "Overview of Recent Graphite Studies for the NP-MHTGR"

**Association of Research Directors Meeting, Newark, New Jersey, November 11, 1993:**

J. R. WEIR, JR., "The Federal Laboratories and Technology Transfer: An Evolution Taking Form"

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G. M. STOCKS, "Towards Quantum Mechanically Aided Materials Design"

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**3rd Japan International Society for the Advancement of Materials and Process Engineering (SAMPE) Metals Conference, Chiba, Japan, December 6-10, 1993:**

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**International Conference on Modeling and Control of Joining Processes, Orlando, Florida, December 8-10, 1993:**

W. B. DRESS,\* T. ZACHARIA, AND B. RADHAKRISHNAN, "Cellular Automata Modeling of Solidification Processes"

B. RADHAKRISHNAN\* AND T. ZACHARIA, "On the Prediction of HAZ Grain Size Using Monte Carlo Simulation"

S. SPOONER,\* S. A. DAVID, X.-L. WANG, C. R. HUBBARD, T. M. HOLDEN, AND J. H. ROOT, "Residual Stresses in Weldments - Effect of Vibration on Stress Relief"

S. SPOONER,\* S. A. DAVID, X.-L. WANG, C. R. HUBBARD, T. M. HOLDEN, AND J. H. ROOT, "Effect of Vibratory Stress Relief During Welding of Thick Stainless Steel Plate"

T. ZACHARIA\* AND G. A. ARAMAYO, "Modeling of Thermal Stresses in Welds"

**Advisory Group Meeting for Waveguide Technology, Oak Ridge National Laboratory, Oak Ridge, Tennessee, December 9, 1993:**

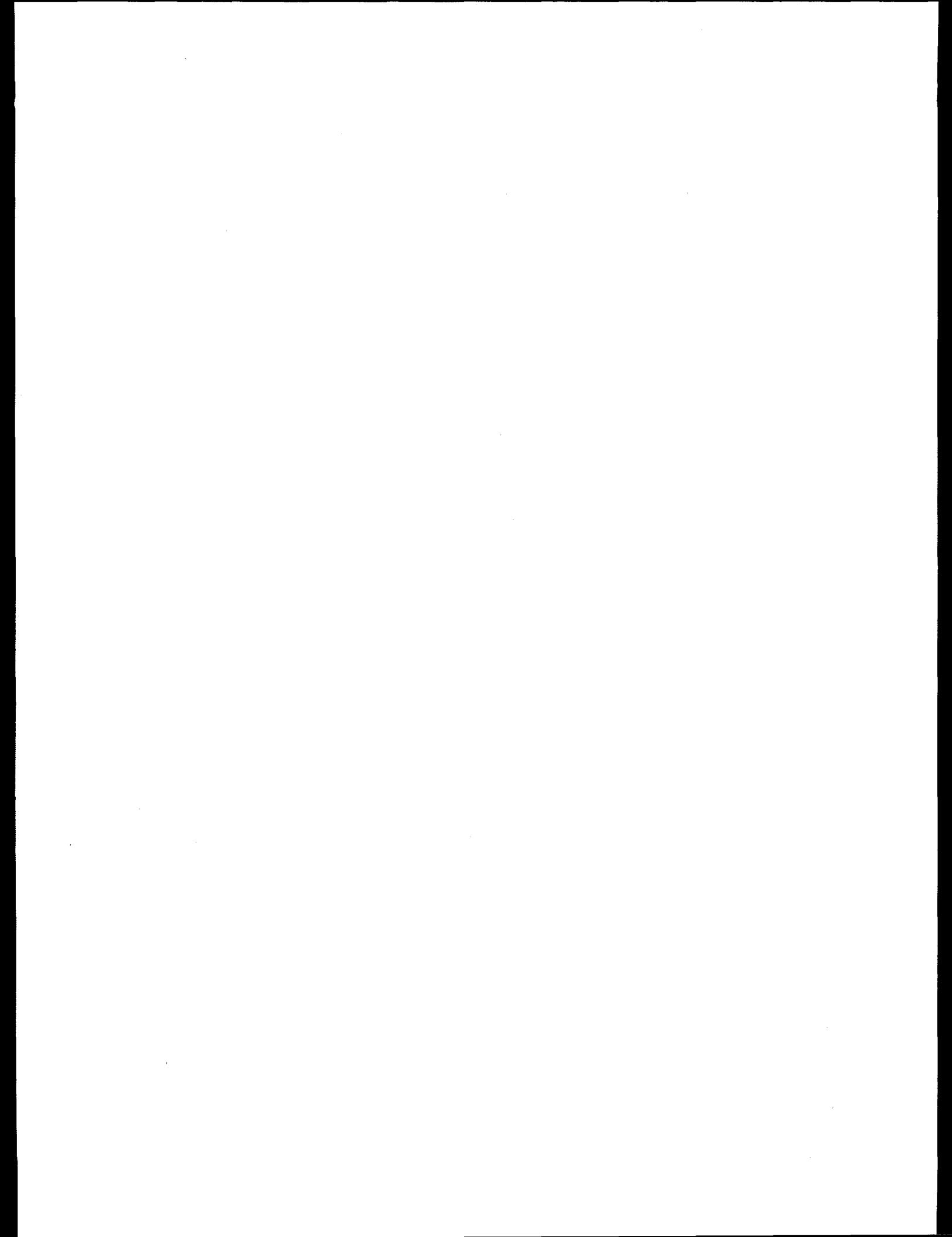
R. A. MCKEE, "Thin-Film Ferroelectrics, MBE Growth, and Waveguide Properties"

**ASM Chapter Presentation, Chicago, Illinois, December 14, 1993:**

L. L. HORTON, "Materials Science and Engineering R&D at Oak Ridge National Laboratory in the 1990s: The New Missions"

**Technical Seminar at Dupont Company, Wilmington, Delaware, December 14, 1993:**

**L. F. ALLARD\* AND T. A. NOLAN, "Electron Holography in Materials Science Research at the High Temperature Materials Laboratory"**



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