METALS AND CERAMICS DIVISION PROGRESS REPORT FOR
PERIOD ENDING SEPTEMBER 30, 1991

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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 fiscal year (FY) 1991. 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; and Sect. 7 summarizes external interactions including cooperative research and development programs, educational activities, and technology transfer functions. 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.

Several organizational changes were made during the year. The New Production Reactor (NPR) Project Office headed by Dave Moses was transferred from Engineering Technology to M&C because the focus of the Oak Ridge National Laboratory (ORNL) effort is on materials issues. The Enrichment Materials Group was transferred to the Atomic Vapor Laser Isotope Separation (AVLIS) Division located at the K-25 site. The Ceramic Technology and Ceramic Processing Development Groups were combined to form the Ceramic Processing Group under the leadership of Ron Beatty. Carbon and graphite activities were spun off as the Carbon Materials Technology Group led by Tim Burchell. Nancy Cole joined the division to assume responsibility for the Fossil Energy Materials Program. Linda Horton, who had been leader of the Structure and Properties of Surfaces and Interfaces Group, moved to lead the Microscopy and Microanalytical Sciences Group. Members of the Structure and Properties of Surfaces and Interfaces Group were moved to the X-ray Research and Applications Group. Tom Kollie became leader of the Building Materials Group, Jim Parks took over leadership of the Fuel Materials Testing Group, and Ruth Hengstler assumed responsibility for the Administrative and Engineering Services Group. Patricia Miller became Division Secretary.

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.

Several members of the division received major awards during the year. R&D 100 Awards were received by Bob Lauf and Barbara Hoffheins (Instrumentation and Controls Division) for their work on gas sensors and by Igor Alexeff, Dave Hobson, and Vinod Sikka for
development of magnetohydrodynamics (MHD) technology for metal atomization. The 1991 Materials Sciences Research Competition Awards were won by C. L. Fu and M. H. Yoo for theoretical work on iron aluminide and by Cullie Sparks and Gene Ice for sustained accomplishments in the development and application of synchrotron radiation. Gene Goodwin was given the William Irngang Award by the American Welding Society, and Malcolm Stocks was a coreipient of the 1990 Gordon Bell Prize awarded by the Institute of Electrical and Electronic Engineers. Paul Becher received an Alexander von Humboldt Research Fellowship for study at the Max-Planck-Institut in Stuttgart.

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

G. M. Slaughter

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 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 six different laboratories, which bear the functional names Corrosion Science and Technology, Fracture Mechanics, Materials Joining, Mechanical Properties, Metalworking Theory and Practice (MTP), and Nondestructive Testing (NDT). Additionally, divisional 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 are presented.

1.1 HEAVY-SECTION STEEL IRRADIATION PROGRAM — W. R. Conwin

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 that 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 (NRC) 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 RPV integrity. Effects of specimen size, material chemistry, product form and microstructure, irradiation fluence, flux, temperature and spectrum, and post-irradiation annealing are being examined on a wide range of fracture properties, including fracture toughness (\(K_{\text{IC}}\) and \(J_{\text{IC}}\)), crack-arrest toughness (\(K_{\text{A}}\)), ductile tearing resistance (\(\Delta J/\Delta a\)), Charpy V-notch (CVN) impact energy, drop-weight nil-ductility temperature, and tensile properties. Models based on observations of radiation-induced microstructural changes using the field ion microscope 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 post-irradiation properties are most frequently limiting in the continued safe operation of commercial RPVs. In addition, a limited effort focuses on stainless steel weld overlay
cladding, typical of that used on the inner surface of RPVs, since its post-irradiation fracture properties have the potential for strongly affecting the extension of small surface flaws during overcooling transients.

Of particular interest are the results of the past year concerning the shifts in fracture toughness and crack-arrest toughness in high-copper welds, the unirradiated examination of a low upper-shelf (LUS) weld from the Midland reactor, and the continued investigation into the causes of accelerated low-temperature embrittlement recently observed in RPV support steels. In the Fifth and Sixth Irradiation Series, designed to examine the shifts and possible changes in shape in the American Society of Mechanical Engineers (ASME) $K_{IC}$ and $K_{IC}$ curves for two irradiated high-copper welds, it was seen that both the lower bound and mean fracture toughness shifts were greater than those of the associated Charpy impact energies, whereas the shifts in crack-arrest toughness were comparable. The irradiation-shifted fracture toughness data fell slightly below the appropriately indexed ASME $K_{IC}$ curve, even when it was shifted according to Rev. 2 of Regulatory Guide 1.99 including its margins. The fact that the fracture toughness has shifted upward in temperature more than the crack-arrest toughness has also apparently contributed to the increased propensity of crack pop-in behavior for the irradiated material. This has the potential for reducing the likelihood of vessel fracture, if the brittle flaws that may initiate have a better chance of arresting prior to significant extension, and will be examined further in the upcoming year. The bettline weld, which was removed from the Midland reactor, fabricated by Babcock and Wilcox (B&W) using Linde 80 flux, is being examined in the Tenth Irradiation Series to establish the effects of irradiation on a commercial LUS weld. A wide variation in the unirradiated fracture properties of the Midland weld was measured with the 95% confidence levels of the Charpy impact 41- and 66-J indices ranging from -35.5 to 19.5°C and 32.7 to 217.6°C, respectively. The $RT_{NOT}$ values, which are governed by the Charpy energy in the LUS welds, ranged from -23 to 23°C. In addition, a wide range of copper content from 0.21 to 0.46 wt % was found with significantly different mean values of the two different weld seams, which had been fabricated with nominally identical welding consumables. This has raised serious questions regarding the appropriate levels and ranges of embrittling impurities to use in vessel integrity analyses. The remainder of the unirradiated fracture testing and preparations for the irradiations of this material are currently in progress. A theoretical examination of the detailed irradiation mechanics that exist in low-temperature irradiations, such as those which produced the accelerated embrittlement of the HFIR pressure vessel, has led to the tentative conclusion that the cause of the acceleration is not the effect of very low-energy thermal neutrons but rather the low rate at which the fluence was accumulated. This conclusion is being investigated in two experiments. Specimens are being irradiated at low temperatures and high-fluence rates in spectra with and without large components of thermal neutrons. Other specimens are being exposed in the cavity of a pressurized water reactor (PWR) at low temperatures and low fluence rates. As a result of the two experiments, it should be possible to establish the mechanism primarily responsible for the accelerated low-temperature embrittlement.

Results from the HSSI studies will be integrated to aid in resolving major regulatory issues facing the NRC 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 light-water RPVs.
1.2 CORROSION SCIENCE AND TECHNOLOGY — J. R. DiStefano

The Corrosion Science and Technology Group conducts tasks that involve evaluations of materials in aqueous, liquid metal, and gaseous environments. Although all corrosion processes have some common features, there are fundamental differences in the processes involved for these different environments. The fields of electrochemical, liquid metal, and high-temperature gaseous corrosion are large and complex, all requiring special knowledge, equipment, and experience for effective study.

The characterization of corrosion processes typical of 6061 Al fuel cladding in the core of the Advanced Neutron Source (ANS) was continued. In the past year, we conducted additional experiments in the ANS Corrosion Test Loop to extend our database describing the film growth kinetics. Improved correlations for film growth were developed that appear much less sensitive to coolant velocity, per se, than our previous "preliminary correlation," and, therefore, we can vary this factor in order to enlarge the range of other important thermal-hydraulic parameters experimentally available in the present apparatus.

Two group staff members have been conducting corrosion studies in support of the Atomic Vapor Laser Isotope Separation (AVLIS) Program. Preliminary studies, conducted jointly with the Chemical Technology Division, have demonstrated initial compatibility of carbon/carbon composite materials in molten UCl₄-UO₂ mixtures in the presence of Cl₂ gas at 750°C.

Three research tasks, as well as a plant support effort, were continued for the Fossil Energy Materials Program. Oxidation and oxidation/sulfidation studies of Fe₇Al were conducted to characterize the effects of alloying additions to Fe₇Al on scale morphologies and oxidation/sulfidation kinetics. Chromium additions to Fe₇Al resulted in higher initial rates of oxidation at 800 and 900°C; however, the ability to rapidly form an oxide layer (particularly α-Al₂O₃) appeared to limit damage caused by any loss of scale during thermal cycling. Preoxidation reduced the corrosion of Fe₇Al-5%Cr in the oxidizing/sulfidizing gas, but preliminary results with specimens coated by a pack cementation process indicated that aluminizing to form an FeAl surface layer significantly improved the resistance to sulfidizing environments for Fe₇Al alloys containing greater than 2% Cr.

Hardness, Young’s modulus, and plasticity of protective oxide scales and bulk oxides were measured by depth-sensing submicron indentation testing to better understand the factors that contribute to the corrosion resistance of high-temperature alloys. Results for scales on oxidized iron aluminate and chromium were well within the range of values obtained by other methods. However, because the hardness and modulus of the scales did not always match those of the corresponding bulk oxide, it remains problematical whether scale behavior can always be modeled using bulk properties.

An eight-year study of the mechanisms of erosion was also completed during the year. New experimental results at very high velocities were obtained and evaluated in the context of all the findings. Hardening behavior and morphological development under various impact conditions could not be explained solely on the basis of mechanical properties. It was concluded that models based on a combination of mechanical and thermal effects are more suited to the description and prediction of the overall erosion resistance of engineering alloys.
Studies for the Fusion Energy Program continued to investigate the susceptibility of candidate structural alloys to irradiation-assisted stress corrosion cracking (IASCC). In collaboration with the Japan Atomic Energy Research Institute (JAERI), slow strain rate tensile (SSRT) tests were conducted in high-purity water on fusion spectrally-tailored specimens using the hot cell facilities of JAERI. The results confirmed earlier electrochemical results that showed only specimens irradiated at 400°C (compared with lower temperatures) were susceptible to IASCC. These results indicate that materials irradiated under fusion spectrally tailored conditions may be less susceptible to IASCC than materials irradiated in commercial power reactors.

The steam generator materials corrosion task for the Modular High-Temperature Gas-Cooled Reactor (MHTGR) New Production Reactor (NPR) continued to evaluate the susceptibility of candidate structural materials and bi-metallic welds (BMW) to stress corrosion cracking (SCC) in high-temperature aqueous environments. In general, BMWs of ferritic steels to austenitic steels showed a tendency to catastrophic failure parallel to the fusion line on the ferritic side of the nickel filler material. High-carbon versions of Alloy 800 (Alloy 800H) sensitized easily during heat treatment and were severely susceptible to SCC failure in oxygen-containing aqueous environments, while low-carbon versions of Alloy 800 (Nicrofer 3220) were not sensitized and, therefore, not susceptible to SCC for the same heat treatments. Experiments exposing these alloys to the MHTGR primary gas (helium containing small quantities of gaseous impurities) are also presently under way.

Heat pump systems offering opportunities for significant energy savings are presently being developed for industrial applications supported by the Conservation Program. The interaction between container materials and salt/ammonia complexes under thermal cyclic conditions was assessed for one such system. Several aluminum alloys and the carbon steel A214 were tested against the possible heat pump working media SrCl₂/NH₃, CaBr₂/NH₃, and CaCl₂/NH₃. None of the containment materials showed susceptibility to SCC. All the materials had excellent general corrosion resistance to SrCl₂/NH₃, but only A214 displayed good corrosion resistance to CaCl₂/NH₃. It was found that the complex CaBr₂/NH₃ was subject to thermal cyclic instability and should not be used as a heat pump working medium.

In a cooperative effort with the Ceramic Processing Group, we provided support for a Conservation Program task to assist three organizations developing high-temperature ceramic heat exchangers for industrial applications. Material loss rates have been measured and the surface reaction layer characterized for ceramic samples exposed to simulated steam reformer environments. The effect of exposure of ceramic tubes to the environment in hazardous waste incinerators has also been assessed.

Present designs of lithium-cooled space reactors utilize Nb-1Zr as the principal structural material. It is known that Nb-1Zr must be protected from oxidation to avoid corrosive attack by lithium, and the SP-100 Program has established a +300 ppm oxygen limit during testing of component systems to avoid this problem. However, experiments conducted elsewhere have indicated that exposure of Nb-1Zr to an oxidizing environment under certain low-temperature conditions raises the ductile-to-brittle transition temperature (DBTT) of the material, and high concentrations of O₂ were found on fractured grain-boundary surfaces. Testing under a variety of low-temperature, low P₂O₅ conditions has thus far failed to confirm any unusual grain-boundary oxidation/diffusion effects and has supported the +300 ppm O₂ limit as a critical level before susceptibility to attack by lithium will occur.
Advanced space power systems that use solar energy and Brayton or Stirling heat engines require thermal energy storage systems to operate continuously through periods of sunlight and shade. Receiver storage units are designed to use the latent heat of fusion of phase change materials. The compatibility of heat storage mediums with the containment materials graphite, pyrolytic carbon, boron nitride, and silicon carbide has been evaluated for applications at 800 to 1100°C. Germanium demonstrated good compatibility with the above containment materials; however, silicon-based alloys continue to present significant compatibility problems.

In tasks supporting other Martin Marietta Energy Systems, Inc. (MMES) facilities, one of our staff members has continued to direct a failure analysis for MMES Uranium Enrichment Operations involving two depleted UF₆ storage cylinders at the Portsmouth Gaseous Diffusion Plant. The cause of failure of both cylinders was traced to mechanical damage that occurred during stacking of the cylinders.

A study of the effectiveness of low-temperature filtration in reducing the Li₂O content of lithium was undertaken in support of a Y-12 Plant Development Division project. A small transfer system was designed and assembled to flow molten lithium containing a relatively high concentration of oxygen through filters of different mesh sizes (200, 40, and 2 μm). Samples of lithium were taken at the exit points of each filter for oxygen analysis to determine the purification potential of filtration.

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 NRC and the U.S. Department of Energy (DOE). We are currently emphasizing the materials property needs for the MHTGR under the NPR Program; HSST and HSSI programs, respectively; ANS; Magnetic Fusion Energy (MFE); and Advanced Industrial Concepts Materials (AIC) 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. Subsize tensile specimens of reactor vessel steel irradiated at relatively low temperatures were tested and confirmed previously determined correlations between irradiation-induced changes in yield strength and Charpy transition temperature, as well as validating the use of subsize sheet tensile specimens for this project. Irradiations of four additional capsules at the University of Michigan’s Ford Nuclear Reactor were completed and the specimens delivered to ORNL for testing. A specification for irradiation of specially designed capsules incorporating tailoring of the neutron spectrum was developed. For the high-temperature fracture mechanics studies, test methods and a special extensometer were developed, along with detailed plans and procurement of additional equipment such as high-temperature environmental chambers. A computer-controlled fatigue-crack growth system was procured and installed on an existing servohydraulic machine; laboratory personnel have been trained and have successfully conducted testing with the system.
For the HSST Program, the task regarding margin assessments during startup and shutdown operations in commercial light-water reactors (LWRs) continued. The results of the studies will be used by the NRC 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. For our continuing investigation of the role of specimen size effects in elastic-plastic fracture mechanics, analyses have shown limitations of the Weibull method and provided information sufficient for initial development of a standard test method under the jurisdiction of the American Society for Testing and Materials (ASTM). A draft standard was developed and has been submitted to the appropriate ASTM E24 task groups for review. The experiments at the University of Maryland resulted in some cleavage pop-ins for the arrested precracks, which occurred at lower K values than those from the cleavage fractures in the fatigue-precracked specimens, but those observations are preliminary because of confounding aspects of plastic yielding.

The task on effects of thermal aging of the stainless steel cladding on the inside surface of PWRs continued. Aging studies were conducted at 288° C for the time equal to that used in the cladding irradiation project (1605 h) and are under way at 288 and 343° C for times up to 50,000 h. The results show that aging at 288° C for 1605 h caused an appreciable decrease in the Charpy impact upper-shelf energy (USE), but the effect on transition temperature was insignificant. The effects on tensile properties were very small. Fracture toughness tests of the thermally aged cladding showed modest reductions in $J_{uc}$ at some temperatures and no changes at others.

For the HSSI Program, studies continued on the Fifth, Sixth, and Tenth Series. The Fifth and Sixth Series examine the upward temperature shifts and shapes of the ASME Code $K_{eq}$ and $K_{irr}$ curves using two high-copper welds. Statistical analyses of the $K_{eq}$ results show that the irradiation-induced shifts of the mean fracture toughness curves and curves fit to bound the data are greater than those of the Charpy impact energy curves, and the slopes of the mean curves have decreased somewhat; the lower boundary curve for the highest copper weld exhibits a significant decrease in the slope. This means that current procedures, which assume that fracture toughness changes are the same as Charpy impact changes, are nonconservative. For the Sixth Series on irradiated crack-arrest toughness, preliminary analyses of the result's indicate good comparison between the irradiation-induced crack-arrest temperature shifts and the Charpy impact shifts. The Tenth Series involves irradiation of the LUS weld material removed from the Midland Unit 1 reactor vessel that was installed but never operated. The weld metal from that reactor vessel is identical to that identified as the controlling material (for pressurized thermal shock analyses) in at least five operating nuclear reactors. A characterization study has been completed that included an investigation of the variations in drop-weight nil-ductility transition temperatures, Charpy impact properties, chemical composition through the thickness of the weld as well as around the belttline and nozzle course weld. The results show a very wide variation in copper content and different distributions of copper content in the belttline and nozzle course welds that will require us to consider the welds as two different materials within the irradiation studies. The design of large capsules for irradiations in the Ford Nuclear Reactor at the University of Michigan is under way. A new task has been initiated regarding the use of thermal annealing to recover radiation embrittlement.
Fracture toughness tests were continued with advanced nickel aluminides for the AIM Program. For the IC-21BZr (low-zirconium) alloy, room-temperature tests showed good strength, ductility, and fracture toughness for the cold-worked/annealed material but low ductility and fracture toughness for the cold-worked material. At 650°C the yield strengths of these materials remained high, but the fracture toughness for both materials decreased dramatically, likely due to oxygen-induced intergranular fracture. The as-cast alloy had good toughness at room temperature but lower toughness at 650°C. This testing program has demonstrated the necessity of evaluating the fracture toughness properties of all of these materials proposed for structural applications.

For the ANS Program, mechanical property tests were conducted with the 6061-T651 aluminum plate procured for the irradiation effects project. Irradiation of the first group of specimens up to $1 \times 10^{22}$ neutrons/cm² (thermal) in the HFIR was completed. The results from the irradiation experiments will be used to make judgments regarding replacement schedules for the core pressure boundary tube in the reactor.

For the Fusion Energy Program, thick-section multipass welds in 21-6-9 or 316LN stainless steel base plates were tested; a variety of filler metals and welding processes were used. Mechanical properties and fracture toughness tests were conducted at room temperature and at 77 K. The tests showed that the presence of ferrite in the weld metals caused low toughness at 77 K, and although the fully austenitic welds had much higher toughness at 77 K, their strengths were lower.

1.4 MATERIALS JOINING — S. A. David

The Materials Joining Group continues to conduct research and development for Basic Energy Sciences (BES), Fossil Energy, AIC Materials, Space Nuclear Power, Fusion Energy, and NPR. In addition, the group provides a wide variety of joining-related services to the Y-12 Plant and to other divisions at ORNL.

At the request of the DOE, ORNL coordinates the activities of the National BES Welding Science programs in the United States. This includes publication of an annual newsletter.

The in-house BES fundamental welding science program investigates the physical metallurgy of weldments and, with the use of process modeling, endeavors to develop the capability to predict weld metal microstructure and properties. The program has as its objectives the following: (1) to understand the solidification behavior of weld metal and its relation to hot-cracking phenomena, (2) to develop and verify mathematical models describing transport phenomena in weld processes, (3) to develop correlations between welding conditions and weld metal structure and properties by means of modeling, (4) to obtain an insight into the fundamentals of phase stability in stainless steel weldments, and (5) to extend the fundamental principles of welding metallurgy to industrial practice in order to promote productivity and reduce costs.

The development of weld metal microstructure is being examined with respect to its effect on hot-cracking behavior of weldments. The influence of solidification substructure, grain structure, and solute redistribution is being investigated. The study relies heavily on the
extensive use of single crystals in order to provide a fundamental understanding of the solidification behavior of the weld pool. Existing mathematical models of transport phenomena have been extended to include a moving heat source, as well as realistic welding processes, such as a multipass weld. These models have been experimentally verified. Phase transformation and mechanical behavior studies of steel weldments will continue with emphasis on the heat-affected zone (HAZ) of the weldment. A range of welding processes is utilized, including high-power beam techniques. The microstructural character and behavior of welds is evaluated using optical microscopy, analytical electron microscopy (AEM), indentation mechanical properties testing, instrumented Charpy impact testing, and thermomechanical simulation. The cooperative research programs with industries and universities will be continued.

Large, single crystals of an Fe-15Ni-15Cr alloy have been utilized in order to increase our basic understanding of the factors that influence the development of weld microstructures. Oriented single crystals were used to make electron beam (EB) welds along various principal directions lying in different principal crystallographic planes. By using oriented single crystals, it was possible to obtain crucial microstructural information that is ordinarily lost when welds are made on normal polycrystalline specimens.

A new, three-dimensional (3-D), geometrical analytical method has been developed in order to interpret the microstructural information available in the single crystal weld. This analytical method establishes a direct correlation between the 3-D weld-pool shape and the dendritic microstructures that are observed in two-dimensional transverse micrographs and can be used to reconstruct the 3-D weld-pool shape.

Single-crystal multipass and single-pass bicrystal welds have also been examined. Overlapping multipass single-crystal welds showed remarkable reproducibility from pass to pass and replicated the microstructural patterns observed in single-pass welds. The microstructure of butt welds joining two single crystals with different orientations showed a one-to-one correspondence with that associated with each individual crystallographic orientation, and the microstructure essentially represented a composite of two single-pass microstructures corresponding to the individual crystal orientations.

A systematic study was carried out to verify the predictions of a transient, multidimensional, computational model by comparing the numerical results with the results of an experimental study. The welding parameters were chosen such that the predictions of the model could be correlated with the results of an experimental investigation of the weld-pool surface temperatures during spot gas-tungsten-arc welding (GTAW) of type 304 stainless steel. This study represents the first time that such a comprehensive attempt has been made to experimentally verify the predictions of a numerical study of weld-pool fluid flow and heat flow.

The microstructures of thick-plate EB welds were also evaluated. The study was carried out on a 3Cr-1.5Mo-0.1V steel. Weld simulations were used to aid in the study of the HAZ microstructure. Such simulations allowed for a more reliable and detailed evaluation of the variation in microstructure with distance from the fusion line.
For the Radioisotope Thermoelectric Generator (RTG) Space Program, test procedures were developed to assess the relative weldability of iridium alloys on a batch-to-batch basis. A database is being established that will permit ranking of individual batches of material in terms of their resistance to hot cracking. To date, 21 production batches have been evaluated, showing a wide range of Sigmajig threshold cracking stress, a measure of hot-cracking sensitivity. We have also completed determination of the effect of oxygen, water vapor, and magnetic-arc oscillation on hot-cracking response.

The MFE Program has involved development of welding procedures and parameters to be used in fabrication of the Burning Plasma Experiment (BPX), emphasizing the highly stressed toroidal field magnet cases. Weldment properties, including yield strength, tensile strength, toughness, and fatigue-crack growth rate, have been measured at 77 K and room temperature for two candidate alloys, types 21-6-9 and 316LN stainless steels.

In collaboration with the Engineering Technology Division (ETD), we procured equipment and developed procedures for two- and three-wire submerged-arc cladding of pressure vessel steel with types 308L and 309L stainless steel. Prototypic clad specimens were prepared for testing by ETD.

The technical lead for developing the welding technology and procedures, which are in strict compliance with applicable U.S. Military Standards for submarine fabrication, was requested of the Materials Joining Group for the SEAWOLF Propulsion Project at the Y-12 Plant. This effort included the following: (1) developing welding procedures for various similar and dissimilar metal alloys' joints, (2) planning and conducting the qualification testing required, (3) writing the procedures and obtaining NAVSEA approval, and (4) specifying the welder performance qualification requirements. Additionally, support to the project is given by providing technical guidance for the fabrication, welding, and inspection of the various alloys.

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

Equipment was procured and procedures were developed for cladding a nickel-base alloy on A533B pressure vessel steel for the KAPL crack-arrest program. The gas-metal-arc welding process was used for this work. Specimens have been produced, and experiments are being performed to evaluate the influence of cladding on vessels containing flaws.

Refractory alloy joining support was provided to the SP-100 Project. Capsules containing lithium are fabricated from refractory alloys under various conditions for compatibility testing at ORNL.

The emphasis of the ceramic brazing activities on the ceramic technology for the Advanced Heat Engines Program during fiscal year (FY) 1991 was on high-temperature brazing of silicon nitride and the correlation of braze joint microstructures with strength data to identify factors controlling joint strength. Joints of Ti-vapor-coated Si₃N₄ were brazed with Au-25Ni-25Pd 65% filler metal at 1130°C and tested at temperatures up to 800°C. Joint strength was not limited by the strength of the braze layer up to 700°C. Above 700°C, joint strength decreased rapidly due to liquation in the braze layer, which appears to result from
decomposition of the Si₃N₄ during brazing, and solution of silicon in the braze filler metal. Several alloys with melting temperatures above 1130°C were also evaluated as possible braze filler metals for Si₃N₄. Initial results indicated that decomposition of Si₃N₄ materials was prevalent above about 1200°C and a major problem for joining by brazing. Thermal analysis experiments are under way to study, in more detail, the reaction of Si₃N₄ with metals at high temperatures. Another activity in support of this program involves monitoring the technical progress of industrial subcontracts in the ceramics joining area. The emphasis of the subcontracts is on optimizing joining materials and processing using computer-aided stress analysis and on predicting joint behavior under generalized loading conditions. The cost of these subcontracts exceeded $3M through FY 1991.

The AIC Materials Program concentrated on improving the weldability of Ni₃Al alloys and on the development of weldable FeAl-type alloys. Certain Ni₃Al alloys, e.g., IC396M and IC221M, have good potential for commercialization and are being considered for a variety of applications that depend on the ability to weld them. The current versions of these alloys inherently have poor resistance to hot cracking, and, therefore, they have poor weldability. Crack-free welds can only be made on them with difficulty, and the reproducibility of crack-free welds is poor. Metallographic examination and microchemical analysis of welds of IC221M and IC396M helped identify features associated with the hot cracks and provided the information necessary for modifying the compositions to improve weldability. Several castings with modified compositions were made. These castings, denoted by IC396W and IC221W, were then welded by the GTA and EB processes. The new alloys had relatively high resistance to weld hot cracking. They were also formed into filler metals and used to make crack-free weld deposits. All welds were made under normal conditions with no special gas shielding, preheating, or post-weld heating conditions being used. The weldability of IC396W and IC221W is considered to be very good. The new alloys are ductile and appear to have strength equal to existing versions of IC396M and IC221M. The new IC396W and IC221W alloys appear to represent a significant improvement over the existing nickel-aluminide alloys in terms of resistance to hot cracking and improved weldability. A patent application is being prepared for these new alloys. This work is part of the Ni₃Al Technology Transfer Task of the AIC Materials Program.

One of the major considerations in developing FeAl-type alloys for structural applications is weldability (i.e., the ease with which these alloys can be welded or joined). Initial GTAW results indicated that the alloys containing only zirconium and/or both zirconium and boron are prone to severe fusion zone (FZ) hot cracking. In addition, an alloy containing boron only was also found to crack severely during welding. Of the various alloys investigated, alloys without boron but containing carbon showed great promise. Carbon in these alloys appears to improve weldability. Successful welds free of hot cracks were produced in these three alloys. Based on these initial results, two alloys, namely FA-372 and FA-388, were selected for further weldability and properties studies.

The Fossil Energy Program concentrated on the weldability of advanced austenitic alloys for elevated-temperature applications. The weldability of an FA-129-based Fe₇Al alloy was evaluated. Of significant concern is the hot-cracking susceptibility of the material. A Sigma fatigue hot-cracking test identified B and Zr as detrimental to weldability. The test also identified FA-129-based Fe₇Al as having reasonable hot-cracking resistance compared to conventional, fully austenitic stainless steel. Delayed cold cracking in the FZ and adjacent
HAZ was identified as a potential problem for these alloys. The cold-cracking problem can be overcome by proper choice of pre- and post-weld heat treatments and careful welding practices. Welding techniques were developed to produce crack-free welds on up to 0.5-in.-thick plate material. Additionally, screening tests were performed in support of alloy development efforts to identify new alloys with a potential for welding applications.

In support of the U.S. Army Strategic Command, a feasibility study was done on the attachment of sapphire windows to metal support rings by brazing. It was demonstrated that by judicious choice of the support ring material (in this case niobium) and by controlled processing, joints of high strength and integrity could be produced. Two window assemblies were produced, and both withstood loading under simulated service conditions. Optical measurements showed that the transmission characteristics of both windows were preserved after brazing. The feasibility of using computer stress analysis to aid in joint design and materials selection was also demonstrated.

1.5 MECHANICAL PROPERTIES -- C. R. Brinkman

The Mechanical Properties Group develops and analyzes data for both metals and ceramics, qualifies new materials, and provides materials engineering support for ongoing energy- and defense-related programs. During FY 1991, we received support from the following programs: development of mechanical properties on modified 9Cr-1Mo steel in support of Advanced Liquid Metal Reactors for the Japan Atomic Power Company (JAPC), 10%; NPR, 20%; Fossil, 10%; Conservation, 15%; Space Nuclear Power, 10%; and miscellaneous service, 35%. 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 present it in a form useful to engineers working on various projects or code developers for design. We serve on several important ASME and ASTM committees developing design rules and test method standards.

Data development and analysis for the materials technology to design and to 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 2-1/4Cr-1Mo (including aged and decarburized material), A533 grade B steel, and Alloy 800. Material was procured and baseline data collection continued to support the heavy-water reactor (HWR)-NPR Program, as well.

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 started for 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. Evaluation was completed of materials and design for tube sheets in hot-gas cleanup systems.
Creep and tensile testing of refractory metal alloys continued in support of the Space Reactor (SP-100) Program. The alloy under test was Nb-1Zr; 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.

Critical to the development of advanced automotive technology using ceramic components (e.g., gas turbines) is the development of a mechanical properties database for candidate structural ceramic materials. During the year, 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 data were generated on silicon nitride and models developed for predicting creep and rupture behavior.

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.

A facility was used for the tensile testing of advanced superalloys in various forms (i.e., coated, directionally solidified, and single crystals) in hydrogen. The work was in support of National Aeronautics and Space Administration (NASA) activities and involved tensile testing of various alloys in high-pressure hydrogen at room temperature. A facility for room-temperature fatigue testing of metal samples in high-pressure hydrogen was developed. We expect to be testing Alloy 718 in this facility and to continue as round-robin monitor for other laboratories doing similar work.

1.5 METALWORKING THEORY AND PRACTICE — V. K. Sikka

The Metalworking Theory and Practice (MTP) Group deals primarily with the development of novel methods for casting, powder making, metal-matrix composites and near-net shapes, cleaning of liquid metals, and materials processing. The development of these methods is supplemented with process models. The MTP 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. In addition, it has the responsibility for transferring to U.S. industry the processing technology for nickel-aluminide and iron-aluminide alloys and other novel fabrication processes being developed.

The MTP 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 managed by MMES.
Processing work is also carried out for other national laboratories, universities, and industries. Specific projects worked on and key accomplishments for FY 1991 are listed below:

1. Magnetohydrodynamic (MHD) Atomization and Spray Nozzle Development. A liquid metal atomization process based on electromagnetic forces was developed in FY 1986. The atomization process requires no gas for atomization and is often termed as a gasless atomization process. A patent for this process was granted in April 1990. Continued development of the nozzle is currently funded under the Steel Initiative Program. The objective of this program is to develop the MHD nozzle for spray depositing 0.50 to 5 by 100-mm low-carbon steel sheet and strip. The significant achievement during FY 1989 was the successful modeling of the electric field distribution in the nozzle. The significant achievement during FY 1990 was the construction of a large chamber for the atomization and spray forming of steel. The system consists of an induction-melting chamber and an atomization chamber. The steel is melted and delivered from a crucible bottom through a tundish to the MHD nozzle. The atomized stream can be spray deposited on a moving substrate. The other significant achievement in this project during FY 1990 was the development of a new, unique design of the nozzle. A patent application has been filed for this new nozzle design.

Experiments continued to atomize and spray steel using the MHD nozzle. The corrosion of nozzle and electrode material by liquid steel continued to be a problem. Tribacore™ was found to be the most compatible material with liquid steel. After using Tribacore™, we were able to spray steel; however, the steel sprays were not adequate to study the thermomechanical processing efforts.

2. 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 (≤ 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 Fe₃Al-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 Fe₃Al alloy won the R&D 100 Award for 1990.

During FY 1991, iron-aluminide alloys were scaled up to several heats of sizes ranging from 50 to 250 kg. The scaleup was carried out at Haynes International, CarTech, and Special Metals Corporation. The large heats were characterized for their microstructure, processing response, and mechanical properties. Tensile tests on these alloys have been completed in the temperature range of room temperature to 800°C. Creep tests have been completed in the temperature range of 450 to 750°C. Several creep tests have reached test times of over 5000 h.
During FY 1991, iron-aluminide compositions have been scaled up to large round and slab ingots. The round ingot was 406 mm diam and weighed 2041 kg. The slab ingot was 203 mm thick by 965 mm wide and weighed 3266 kg. Slabs from the round ingot were processed into 0.76-mm-thick sheet and tested for mechanical properties. The tensile properties were completed from room temperature to 800°C. The results of these tests showed the 406-mm-diam ingot to have the same properties as those observed for 0.454-, 7-, and 136-kg heats.

The iron aluminides were licensed to three companies: Ametek Specialty Metals Division, Hoskins Manufacturing Company, and Harrison Alloys.

3. **MHD Inclusion Removal Device.** A device based on MHD forces to separate the nonmetallic inclusions from liquid steels was developed. A patent for this device was issued in 1988. This device won an R&D 100 Award in 1990.

4. **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 1991, the casting alloy composition was further optimized. This composition is known as IC-221M. The casting temperature for this composition was optimized by casting three sets of specimens and two sets of turbochargers. Castings were prepared under commercial conditions at Precision Castparts Corporation. The cast test bars were characterized for microstructure, tensile properties, and fatigue properties at ORNL. Data from these castings were transferred on a timely basis to Cummins Engine Company, an exclusive licensee, for use of nickel aluminide for turbochargers in diesel engines.

Another application of nickel aluminide will require the use of centrifugal-cast pipes. In support of this application, ORNL provided the chemistry standards and the technical expertise to the vendor performing the work.

Gleeble data were generated on another nickel-aluminide alloy, IC-218LZr, to determine its hot workability. Test temperatures used were 1000, 1100, 1150, and 1200°C; strain rates ranged from 0.2 to 60/min. These data are also being transmitted to potential material producers.

5. **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 of iridium, two major pieces of equipment were installed and qualified for iridium production during FY 1991.

Two technical papers and reports were prepared from this work.
6. **Superconductor Fabrication.** The effort on the fabrication of long lengths of high-temperature superconductor material continued during 1991. The primary emphasis was on process development to yield very high-current density. A new process has been identified to produce high-current density ribbons. A patent application for the process is currently being filed.

7. **Technology Transfer.** Significant effort was spent in the transfer of various technologies to industry. 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. Three licenses on the production of iron aluminide resulted from this effort.

8. **Work for Others (WFO).** Work was carried out in the MTP group for Wright Patterson Air Force Base (through Universal Energy Systems), Pratt & Whitney, Cummins Engine Company, AMAX, NASA, Fansteel Metals, and several universities. The effort grew in FY 1991 to include Southwest Research Institute, American Superconductor Corporation, Idaho National Engineering Laboratory, and several universities.

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

The NDT Group develops new and improved methods and equipment for nondestructive examination (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, thermal imaging, dye penetrants, eddy currents, and penetrating radiation. Applications of NDT methods are of interest to a number of sponsoring agencies, including the DOE and NRC. Technical development and support services have also been provided in cooperation with other ORNL divisions and for outside agencies through the WFO Program.

We continued development and application of ultrasonics and computerized tomography (CT) techniques for structural ceramics for the Ceramic Technology for Advanced Heat Engines (CTAHE) Project. We have digitally radiographed and CT scanned a number of tensile test bars and ceramic turbine rotors from various sources in search of internal flaw indications. We have also worked with industrial interests in developing CT and radiographic reference and calibration standards for detection of small flaws in ceramic components. An ultrasonic scanner has been modified and reprogrammed for investigation of synthetic aperture focused transducer (SAFT) techniques. This specialized signal-processing technique should offer the improved signal-to-noise figures necessary to detect small flaws at greater depths in ceramic materials. Also, our high-frequency ultrasonic system has been modified for greater resolution and interfaced to a computerized image processing system. Work continues in applications of focused transducers and surface waves.
For the DOE Office of New Production Reactors (ONPR), we have developed eddy-current characterization techniques for conductivity in graphite core material. Extensions of this work will be used for material flaw detection and for surface oxidation erosion measurements. We have also done ultrasonic characterization of ceramic core support materials for the MHTGR. Before work was curtailed this year on our work for the HWR, we did studies for ultrasonic inspectability of heavy-section austentic welds and coordinated the work among teams at ORNL, Savannah River, and the reactor contractor.

We continued component inspection support for the RTG Program for the DOE Office of Special Applications. Inspection of iridium blanks and carbon composite insulator sleeves continues. An automated evaluation method for radiographs of carbon composite components is being developed. Confirmatory ultrasonic inspections for a new design for multicouple components were made.

For the NRC, our program of improved in-service inspection of steam generator tubing continues with improved computer analysis codes and a new, 16-coil, non-rotating probe design. We are testing our eddy-current systems by participation in an international round robin, PISC. Our activities in commercial reactor inspection consultation for the Regulatory Office have increased this year with evaluation of a number of reactor inspections and special studies on tube plugging criteria.

A number of other activities have received NDT support through WFO or other laboratory programs. We continue development of neural network analysis of eddy-current signals under U.S. Air Force (USAF) sponsorship. CT imaging of aggregate distribution in asphaltic concrete continues, also for the USAF. We have characterized, by ultrasonic modulus measurements, a number of ceramic armor samples for a U.S. Army program. Flaw detection studies were performed by an ORNL-designed, pulsed, eddy-current system on tubing samples submitted by Bettis Atomic Laboratory—commercialization of our instrument design may result from this work. Development of our instrument for magnetic signature analysis of mechanical properties of steels under the Advanced Diagnostics Engineering R&D Center seed money project is nearly complete. We sponsored work by a summer visiting professor for the ultrasonic characterization of iron-aluminide alloys.
2. HIGH TEMPERATURE MATERIALS

V. J. Tennery

A major objective of the research in the High Temperature Materials Section is to characterize the microstructure of materials and to understand how the microstructure controls physical and mechanical properties. Another major function is to provide one of the primary interfaces between the M&C Division and materials researchers outside ORNL, who visit the Laboratory as users. Research achievements of this past year clearly exemplify these objectives.


2.1 HIGH TEMPERATURE MATERIALS LABORATORY USER PROGRAM — F. M. Foust

The HTML User Program became operational in FY 1988. This User Program is the focal point at the ORNL for interaction between researchers from industry, universities, and ORNL in their pursuit of developing advanced high-temperature ceramics.

During FY 1991, instruments were purchased to equip two new user centers (X-ray Residual Stress and Ceramic Specimen Preparation) that were approved in FY 1990. These two centers will be staffed and fully operational by mid-FY 1992.

Instruments located in the four original user centers of the HTML (Materials Analysis, X-ray Diffraction, Physical Properties, and Mechanical Properties) were utilized by 88 researchers from 46 institutions (26 industries, 19 universities, and 1 other government facility). These outside users accrued a total of 3875 user days during the year. There were 90 researchers from MMES, who accounted for an additional 5309 user days during this time period.

A description of the research capabilities available in the HTML User Program is given in the High Temperature Materials Laboratory, Metals and Ceramics Division, Fourth Annual Report (October 1990 through September 1991) by Victor J. Tennery and Felicia M. Foust, ORNL/TM-12023 (December 1991). This report also summarizes the nonproprietary research projects conducted at the HTML during FY 1991 by outside users.

Formal user agreements were executed with 27 universities, 23 industries, and 1 other government facility during FY 1990. To date, 149 user agreements have been executed (64 universities, 80 industry, and 5 other government facilities). These totals include 28 proprietary agreements.

The HTML User Program is funded by the Office of Transportation Technologies, Conservation and Renewable Energy, U.S. DOE.
2.1.1 Materials Analysis User Center (MAUC) — T. A. Nolan

The MAUC 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 materials performance. During the past year, user activities with researchers from universities and industrial companies have continued at about the same level as during 1990. Use of the surface chemistry facilities by ORNL researchers has increased significantly during the past year.

Several important new instruments are in the process of being added to the suite of user center instruments. A scanning probe microscope (SPM) incorporating both scanning tunneling and atomic force microscopy modes is being purchased. This instrument will have interchangeable heads that will allow the surfaces of large specimens to be imaged with near-atomic resolution. Another major, new addition will be a field emission gun (FEG) transmission electron microscope (TEM). The contract for a Hitachi HF-2000 200-kV FEG-TEM was placed in September 1991, and delivery is planned for June 1992. This instrument adds two 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. The field emission source illuminates the specimen coherently, thus allowing electron holography to be performed. Electron holograms preserve image phase information (lost in conventional TEM techniques). Utilizing the additional phase information, lens aberration corrections can be made that should result in greatly improved resolution (possibly reaching the 0.1-nm level). Also, magnetic flux quanta can be imaged, and specimen thickness variations of less than 0.05 nm can be determined. A Director's Fund initiative to develop electron holography and demonstrate it for solving materials characterization problems was approved, and work is under way.

Both internal and user research projects have produced significant results during the past year. Analytical and high-resolution TEM have been used to determine mechanisms of creep and fatigue in Si₃N₄ structural ceramics. These studies contributed to the reformulation of a manufacturer's Si₃N₄ ceramic, which greatly improved the high-temperature creep resistance and other properties. TEM techniques were utilized to identify pure edge vacancy loops and antiphase domain boundaries as the primary crystal defects in α-Si₃N₄ grown by chemical vapor deposition (CVD). Imaging secondary ion mass spectroscopy (SIMS) was used to unequivocally detect intergranular lithium attack in Nb-Zr alloy (used in the SP-100 Program) where none had been detected by conventional techniques. In association with the University of Tennessee and Pennsylvania State University, electron holography has been used for the first time to characterize details of ferroelectric domain walls in barium titanate.

2.1.2 Mechanical Properties User Center (MPUC) — M. K. Ferber

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. The major research facilities include: (1) a Flexure Test Facility (FTF) comprising six high-temperature flexure load frames; (2) a Tensile Test Facility (TTF) consisting of eight...
high-temperature tensile testing load frames, a fiber test machine, a composites test machine, and servo-hydraulic universal test machine equipped with tension/compression grips; (3) a
general-purpose testing lab comprising two universal test machines and a room-temperature
tensile tester; and (4) a mechanical properties microprobe (MPM) [Nanoindenter]. In the
following, detailed descriptions of the tension, flexure, and indentation research facilities are
provided, along with appropriate examples of data generated by these facilities.

2.1.2.1 Flexure Test Facility

During FY 1991, extensive flexure strength research was conducted using both the FTF and
a universal test machine (UTM) equipped with a high-temperature furnace and ceramic retort.
Studies involving flexure strength measurements have focused upon (1) the correlation of
high-temperature strength of sialon ceramics with variations in the processing parameters,
(2) the effect of microwave annealing of silicon nitride upon the creep and fatigue resistance,
(3) the relationship between fracture toughness of whisker-reinforced alumina and
crack/whisker orientation, (4) the evaluation of the stress intensity factor threshold for
high-temperature crack growth in silicon nitride, (5) the effect of environment upon the fatigue
resistance of silicon nitride, and (6) the correlation of flexural creep data with tensile creep
data generated for a high-performance silicon nitride.

2.1.2.2 Tensile Strength Research

During FY 1991, extensive studies of the strength, creep, and fatigue behavior of silicon
nitride button-head tensile specimens were conducted at temperatures in the range of 900 to
1400°C. 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 this research was that when failure was
controlled by creep damage generation and accumulation, the fatigue life was uniquely
determined by the steady-state creep rate. A second objective was to verify the expected
improvements in creep and fatigue resistance of a hot isostatically pressed (HIPed) silicon
nitride, which resulted from processing modifications to the intergranular phase. Much of the
research activity in the TTF focused on the measurement of the tensile fatigue and creep
properties of both NT 154, a mature HIPed silicon nitride, and modified NT 154 material
designated as NTX 164. Baseline data generated for the NT 154 at 1260 and 1370°C
indicated that fatigue was controlled by the growth and coalescence of creep damage in the
form of lenticular cavities formed between two-grain faces oriented at angles of about ±30°
of the tensile axis. In this temperature regime, the fatigue life was uniquely determined by the
steady-state creep rate. These cavities were absent in the NTX 164 material.

At a given stress and temperature, the creep rate was a factor of 2 to 4 lower than for the
NT 154 silicon nitride, while the fatigue life was a factor of 2 to 5 greater. These results have
been instrumental to the introduction of the NTX 164 silicon nitride as a commercial material.

2.1.2.3 Mechanical Properties Microprobe

The MPM (the Nanoindenter) in the MPUC is a special microhardness measuring system
capable of operating at loads in the microgram range (0 to 20 mN). A high load range (0 to
120 mN) is also available. Unlike conventional hardness measuring instruments, it is not
necessary to determine the area of an indent optically in order to calculate hardness. Instead, the height of the indenter relative to the surface of the specimen is constantly monitored with a sensitive capacitance gage, thus allowing the depth of an indent to be determined. The unique feature of the Nanoindenter is its ability to measure indent depths to ±0.2 nm. The area of the indent is then calculated from a knowledge of the geometry of the tip of the diamond indenter. The load is also constantly monitored with the result that hardness is reported as a function of displacement. Measurements of sample stiffness from unloading data permit a separation of the plastic and elastic components of displacement, and the projected areas for indents can be calculated on the basis of the plastic depth of the indents. The elastic moduli of samples can also be estimated from stiffness data. During FY 1991, the Nanoindenter was used to (1) evaluate the plastic and elastic properties of thin films, ion-implanted surfaces, and laser-annealed surfaces; (2) generate load-displacement curves for silicon microbeams; and (3) measure the interfacial properties of fiber-reinforced ceramic-ceramic composites.

2.1.3 Ceramic Specimen Preparation User Center (CSPUC) — M. K. Ferber

The CSPUC provides basic facilities for (1) investigating material removal processes associated with the machining of high-performance ceramics, (2) fabrication of new specimen test geometries required to evaluate the mechanical performance of structural ceramics, (3) dimensional inspection of machined specimens, (4) measurement of surface roughness and form, and (5) application of strain gages. Specific equipment includes a slicer/grinder with open-loop control, a computer numerically controlled (CNC) slicer/grinder, a CNC four-axis grinder, an optical comparator, and a computer-controlled profilometer. Facilities for the application of strain gages are also available. Gage outputs are monitored during testing using a dedicated computer and data acquisition system (DAS).

During FY 1991, CSPUC played a major role in a number of in-house programs. For example, research activities in the International Energy Agency (IEA) Annex II Subtask 5 agreement required extensive utilization of both the optical comparator and strain-gage facilities. The IEA Subtask 5 effort is aimed at comparing flexure and tensile strengths of a silicon nitride ceramic. These strength measurements were conducted at ten participating U.S. laboratories. A total of 450 flexure and 150 button-head tensile specimens were fractured as part of the program during this year. Because dimensional consistency is critical to the successful measurement of the strength of button-head tensile specimens, each specimen was inspected with the optical comparator. This inspection process resulted in the identification of machining problems in the shank-to-button transitions of several specimens. By utilizing the inspection data, the vendor responsible for machining the specimens was able to correct the problem. Properly machined specimens were critical to the success of this international IEA research project.

For the IEA flexure strength measurements, a gaged flexure specimen was used to evaluate each participant's room- and high-temperature flexure fixtures. The strain measurements were made with the strain-gage DAS. The resulting data revealed some serious design problems with many high-temperature fixtures used in U.S. laboratories and point to the serious need for a standard in this area. The strain-gage DAS was also used to measure the strain at various locations in the gage section of each button-head tensile specimen during actual loading to fracture. These data were then used to calculate the percent bending as a
function of applied load. Several special machining procedures were also developed by CSPUC personnel. One procedure involved the fabrication of C- and O-ring specimens from both 2.5- and 5-in.-diam ceramic composite tubes. Originally, a minimum of 8 h was required to machine one specimen from the 2.5-in.-diam tube while the preparation of specimens from the 5-in. tube was unsuccessful. Effective utilization of the CNC 4-axis grinder reduced the grinding time to a maximum of 15 min/specimen (after setup) for both tube sizes. This work was instrumental for the timely completion of the mechanical testing tasks in the ORNL Advanced Industrial Heat Exchangers Program.

A second example involving the CNC 4-axis grinder concerned the development of a procedure for machining 0.157- and 0.490-in.-diam disks of alumina from square blocks (0.20 x 0.20 x 0.062 in. for the 0.157-in.-diam disks and 0.55 x 0.55 x 0.062 in. for the 0.490-in.-diam disks). This procedure necessitated fabrication of a special ceramic arbor for holding the square stock in the machine. The circular disks were required for physical properties measurements, which were part of a DOE-OR milestone for the MHTGR-NPR Program.

2.1.4 X-ray Diffraction (XRD) and Physical Properties User Centers (PPUC) — C. R. Hubbard

The facilities of these two User Centers provide both XRD and thermophysical property measurement techniques to characterize structure, phase content, stability, reactions, and thermal properties of advanced structural materials. The temperature range of the facilities is from room temperature to 1500°C and above. The knowledge obtained is used to improve synthesis, processing, and utilization of advanced materials and to develop models relating microstructure, phase content, and defect concentration to properties and performance. During the past year, overall user activity, both external and ORNL, increased steadily. Details of the numerous user research activities have been published in the HTML annual report. A couple of examples are given below, along with a brief description of facility enhancements.

The capabilities of the high-temperature X-ray diffractometer were extended with the addition of computer control of the temperature controller, the installation of capability to use hydrogen gas for reduction and hydrogen embrittlement studies, and the modification of the furnace system to permit studies well above 1500°C in 1 atm nitrogen. The latter capability will be important in pressureless sintering studies of nitrogen ceramics and was first used by Dow Chemical Company in a study of the temperature and compositional dependence of Al₂O₃-Y₂O₃ intergranular phases in AlN. The completion of the automated xenon flash instrument for room-temperature thermal diffusivity measurement complements the high-temperature laser flash system, enabling rapid and more accurate measurements at room temperature. Work has commenced to develop a longitudinal bar thermal conductivity cryostat to provide thermal transport measurements from about 90 to 500 K, needed for modeling the contribution of microstructure and defects on thermal conductivity.

With United Technologies Research Center (UTRC), the thermal aging of plasma spray thermal barrier coatings was studied by laser flash thermal diffusivity measurements in combination with scanning electron microscopy (SEM) and density determinations. Together, models to predict the thermal conductivity and density as a function of aging time and temperature were developed. Simultaneous thermal analysis (STA) and high-temperature X-ray diffraction (HTXRD) of polymer precursors for oxide ceramic synthesis were studied
with a user from Cornell University. This study has led to a detailed understanding of the chemical processes and crystallization of ceramics involved in burnout and consolidation of the ceramic. The method holds promise for development of high-purity ceramics with unusually well-controlled chemical composition. STA and HTXRD were also used to characterize the conversion of ZrO₂ and (Zr,Hf)O₂ gels prepared at the University of Kentucky Center for Applied Energy Research. The observation that either tetragonal or monoclinic zirconia could be formed by simply changing the rate of gel formation was clarified by determination that, in all cases, tetragonal zirconia forms upon heating above 400°C, and that only on cooling to below 200°C, does the monoclinic form of zirconia evolve in the rapidly precipitated gels. The differences were determined not to be due to differences in crystallite or agglomerate size. Utilizing studies with sulfate anions, we determined that the transformation to monoclinic is likely due to differences in anionic defects on the surfaces of the particles. The HTXRD study with Dow Chemical Company demonstrated furnace operation to above 2000°C and the ability, when combined with a position-sensitive detector, to study intergranular phase reactions and transformations in aluminum nitride ceramics at these extremely high temperatures.

2.1.4.1 Residual Stress User Center — C. R. Hubbard

The Residual Stress User Center is currently being developed and will initially consist of an XRD system for mapping macro residual stresses on the surface of ceramic and alloy materials. The procurement of the instrument has been completed, and delivery is expected in early FY 1992. The system consists of a state-of-the-art stress and texture goniometer equipped with a Peltier-cooled, solid-state detector and an 18-kW rotating anode generator. The system provides for complete flexibility in sample tilt and provides either divergent or parallel beam operation. Recruitment for a professional staff member is currently under way. User research is planned to begin in early 1992.

To develop competence in measurement of residual stresses within a component, a Director's Fund R&D project, "Development and Demonstration of Neutron and X-ray Residual Stress Mapping," was proposed and funded. This project is being conducted in collaboration with the Neutron Scattering Group of the Solid State Division and utilizes an existing triple-axis spectrometer. Attachments for sample positioning, data collection, and automation have been developed and successfully tested on the spectrometer. The first demonstration study was a comprehensive mapping of the residual stresses within a 12- x 2- x 1/2-in. ferritic plate containing a multipass weld. The results will be used to develop and verify models for stresses in multipass weldments. Studies of macro stresses in ceramic and micro stresses in composites are also to be conducted.

2.2 METALLOGRAPHY AND TECHNICAL PHOTOGRAPHY — J. R. Mayotte and J. W. Nave

These groups provided state-of-the-art metallography, light microscopy, technical photography, and image analysis support to research programs originating within M&C Division; research and development activities at other MMES sites; and research efforts involving industrial companies and universities through user program activities.
2.2.1 Metallography — J. R. Mayotte

The Metallography Group performs research in optical microscopy, microanalysis, and photography of both alloy and ceramic materials. Metallographic examination is performed in collaboration with materials researchers within the M&C Division, ORNL, other federal laboratories and agencies, and industrial firms. During this reporting period, the group prepared 1764 metallographic specimens. In microanalysis, 442 specimens were analyzed.

As renovation of facilities within the metallurgy laboratory continues, environmental, safety, and health (ES&H) deficiencies are being corrected.

The Datamat Automated Rotary System continues to be a valuable tool for improving polishing techniques for both alloy and ceramic materials. Metallographic preparation of cross-sectioned materials for materials characterization continued as a major effort. A computerized cataloging system for metallographic specimens was developed and put into use, which has the capabilities for storing and retrieving information pertaining to each metallographic specimen submitted to the group. Y-numbers assigned to photomicrograph negatives were discarded, and a new numbering system produced by the specimen number plus extensions for negatives was developed and is being used.

2.2.2 Field Metallography and Failure Analysis — J. R. Mayotte and W. S. Eatherly

Field metallography and failure analysis for ORNL and other DOE installations remains a function of our group. At specified intervals, work continued on nondestructive in situ metallographic examinations of test specimens to follow changes in microstructure. These are thermal-shock samples from the ETD.

2.2.3 Technical Photography — J. W. Nave

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

Our video capabilities have been expanded by the addition of two Panasctic cameras and light systems and two Panasonic editing systems. We produced two 30-min films for public television, which can be used in the public school systems.

In addition to research photography, the group is involved in public relations photography for ORNL. Technical Photography is responsible for the large (30- by 40-in. and 40- by 60-in.) photographs that are on display in our hallways and the south canteen.

Identification and correction of ES&H deficiencies are ongoing concerns. For example, we are beginning a recycle program with Eastman Kodak and other vendors. Also, we are beginning to regenerate some of the photographic chemicals to conserve handling and waste.

This past year, the Technical Photography staff members completed 1066 photographic work orders, which included 23,446 units of photographs, photomicrographs, slides, and viewgraphs.
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 (Defect Mechanisms Group), and alloy design (Alloy 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 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 (FWB) 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) [Structure and Properties of Surfaces and Interfaces Group]. 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

Most research in the Theory Group is based on the principle of understanding properties of materials in terms of the underlying electronic structure. The electronic structure of periodic systems is treated using first-principles pseudopotential theory, the full-potential linearized augmented plane wave (FLAPW) method, and the Korringa Kohn Rostoker (KKR) method. The properties of substitutional disordered alloys are treated using the KKR-Coherent Potential Approximation (CPA), and microchemical interactions in materials are investigated using an Augmented Gaussian Basis (AGB) interacting atomic cluster technique.
Among the noteworthy results from projects in this reporting period, we summarize the following:

1. **Basic theory and technique.** We have generalized Multiple Scattering Theory, the basic theory upon which our KKR codes are based, so that it can be used to treat general potentials. We have also devised a new technique for electronic structure calculations, which promises great improvements in calculational speed and accuracy, especially when used on a parallel processing computer.

2. **Phase stability of alloys.** We have used our new complex lattice KKR-CPA codes to study the total energy of NiAl alloys as a function of the long-range-order parameter. We have calculated the electronic structure of Martensitic B2 alloy Ni$_{50}$Al$_{50}$ and observed that the parts of the Fermi surface thought to be responsible for the Martensitic transformation survive disordering.

3. **Mechanical properties of alloys.** We have performed first-principles calculations of the fundamental properties of the B2 aluminides, NiAl and FeAl, and have used these to better understand their mechanical properties. First-principles calculations of elastic constants, anti-phase boundary (APB) energies, and cleavage energies were performed, and the fracture mode (brittle versus ductile) was explained in terms of these properties.

4. **Defects in alloys.** We have investigated the structure and energetics of vacancies and anti-site defects in B2 NiAl. We have used both FLAPW and AGB cluster methods to further investigate the electronic structure effects responsible for the strengthening effect of boron on the grain boundaries of Ni$_5$Al. We also used these approaches to investigate the embrittling effects of hydrogen in FeAl.

5. **Modeling of dislocations in alloys.** We have used molecular dynamics and the semi-empirical embedded atom technique to model the structure of dislocations in B2 NiAl and to observe how dislocations move in response to an applied stress.

6. **High-performance computing.** We modified our relativistic multi-sublattice KKR-CPA codes so that they can run quickly on a parallel processing computer. G. M. Stocks won the Gordon Bell prize for this work.

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

We are applying laboratory and synchrotron X-ray sources to the study of the structures of CVD diamond films, interfaces, intermetallic alloys, high-$T_c$ superconductors, and antiferromagnetic alloys and to the measurement of short-range order in Fe-Ni-Cr binary alloy combinations.

Diffuse X-ray scattering measurements from a nitrogen-implanted niobium film reveal that the elastic displacement fields arise mainly from dislocation loops on (211) planes. A smaller contribution is from single interstitial nitrogen in octahedral sites. An interface between a GaAs film and the silicon substrate was shown to contain a grid of misfit dislocations. They gave rise to a smaller strain (perpendicular to the interface) than expected from the
van der Merwe model. As the GaAs-Si interface was found to be rough with atoms being out of position by $2.9 \pm 1\text{Å}$, the strain could be accommodated by this roughness or by relaxation. Intermetallic alloys often have ternary metal additions, which can alter their phase stability and change their mechanical properties. In a study of Al$_4$Ti with an 8 at. % iron addition, we found that iron substituted proportionally to the two sites: 3/4 to the aluminum sites and 1/4 to the titanium sites. Of interest was the disorder among the aluminum and titanium atoms caused by the iron addition. About 15% of the titanium sites were now occupied by aluminum, and 5% of the aluminum sites were occupied by titanium. The disorder caused by ternary additions may be of practical importance in ductilizing some brittle intermetallics. A completed study of the local atomic arrangements (near-neighbor chemical preference and atomic displacements between atom pairs) in an Fe$_{80}$Cr$_{47}$ alloy quenched from above the miscibility gap gave the individual pair displacements between the Fe-Fe, Fe-Cr, and Cr-Cr atom pairs for the seven nearest neighbors. These distortions and local atomic arrangements have a major effect on the physical properties of solid-solution alloys. Measurements have been completed on Ni-Cr alloys as we build toward a study to understand the mechanical behavior of the Fe-Ni-Cr stainless steels.

3.3 MICROSCOPY AND MICROANALYTICAL SCIENCES — J. Bentley

The research of the Microscopy and Microanalytical Sciences Group involves microstructural characterization of metallic and ceramic materials by TEM or AEM and atom-probe field-ion microscopy (APFIM) with an emphasis on structure-property correlations. The group is responsible for maintaining and developing the AEM and APFIM facilities of the Materials Science Section. These facilities are the focus of the group’s activities and are also used by personnel from many other groups in the division and by collaborators on the ORNL/Oak Ridge Associated Universities (ORAU) Shared Research Equipment (SHaRE) Program (see Sect. 7.1). The main source of funding is the BES Division of Materials Sciences of DOE.

For the last few years, the main instrumentation development effort has been the design and construction of a position-sensitive atom probe. This year the optical atom probe (OAP), which employs a video-based detector system, was completed. Work continued on the more sophisticated mapping atom probe (MAP). Further enhancements to the atom-probe control software were made, and commercial licensing was completed. There was progress in techniques to visualize 3-D atom-probe data, and the effects of preferential evaporation on composition determinations by APFIM were studied. The various sophisticated methods for determining composition amplitudes from APFIM composition profiles were compared, and a fractal analysis of the α-α' interface in spinodally decomposed Fe-Cr alloys was completed. Work on irradiated pressure vessel steels and model alloys was suspended because of the lack of specimen preparation facilities for low-level irradiated specimens, but related research (with M. G. Burke, Westinghouse) on 17/4 PH steels progressed. Solute partitioning in a creep-tested Ni-Al-Mo-Ta superalloy was characterized by combined APFIM and AEM. A new class of precipitation-hardened intermetallics was proposed following APFIM characterization of grain boundaries and precipitates in boron-doped NiAl. Low-temperature phase transformations in thermally aged iron-nickel alloys were studied by APFIM in collaborative SHaRE Program research with J. Zhang (Lehigh University). Related work on meteorites over the last few years continued this year with examination of a weathered sample of the Santa Catharina Fe-Ni meteorite.
Electron microscopy work supported by the BES Microscopy and Microanalysis Task covered topics from fundamental materials science to technique development. Further theoretical work on inelastic electron scattering included treatments of multiple inelastic scattering, modeling the effects of valence or plasmon losses on high-resolution imaging, and use of the new dynamical diffraction approach, developed last year, to produce the first ever computer-simulated diffraction patterns correctly showing thermal diffuse scattering streaks. Reflection electron microscopy (REM) dynamic (video) recording of in-situ heating experiments at up to 1670 K allowed studies of the migration of atomic-height surface ledges and the formation of new phases on oxide ceramics. A method proposed last year for quantitative microanalysis by reflection electron energy-loss spectrometry (REELS) was verified with work on MgO. Further studies were made of the effects of hole count and secondary fluorescence in X-ray microanalysis. A simple but novel specimen preparation technique allowed a unique characterization of diamond films made by CVD, including direct evidence for a correlation of internal microtwins with the crystallography of growth facets. The sublattice occupancy of nickel in Cu_{50}Au_{50},Ni_{x} alloys was determined by the atom location by channeling-enhanced microanalysis (ALCHEMI) method (in collaboration with T. Shiraiishi, Kyushu University). High-spatial-resolution AEM was used to continue studies of segregation. Radiation-induced segregation (RIS) profiles at grain boundaries and dislocation loops were measured by AEM in neutron and ion-irradiated stainless steels (work jointly supported by BES and the Fusion Energy Materials Program) primarily to help understand IASCC. Parallel-detection electron energy-loss spectrometry (PEELS) was also used for segregation measurements with advantages for highly radioactive specimens. SHARe Program collaborative research began on AEM measurements of grain-boundary segregation in thermally and irradiation-sensitized type 304 stainless steel (with M. G. Burke, Westinghouse) and on equilibrium segregation in Al-Mg alloys (with P. Deymier, University of Arizona). Plasmon losses were used in PEELS research to measure the distribution of metallic aluminum, as differentiated from Al^{3+} ions, in MgAl_{2}O_{4} spinel implanted with aluminum. Other SHARe Program collaborative AEM research included further work on the growth of Si_{1-x}Ge_{x} on <001> silicon substrates (D. C. Paine, Brown University); further work on B2 intermetallic alloys (J. Baker, Dartmouth College); new work on aluminum-silver alloys (J. Todd, IITRI); and a new project on TEM Z-contrast imaging of aluminum-lithium alloys (A. Fox, Naval Postgraduate School).

Collaborative applications of AEM in support of other groups and tasks within the division received less emphasis than in recent years. Work on ion-implanted ceramics (see Sect. 3.4) was limited to the characterization of SiC ion implanted with iron. Collaborative research on structural ceramics (see Sect. 4.4) continued for part of the year but then ceased due to personnel reassignments. With funding primarily from the DOE Conservation Program through the Superconductivity Pilot Center, electron microscopy of high-temperature superconductors (HTSC) continued with an emphasis on bismuth-based materials (see Sect. 3.8). Related collaborative SHARe Program research on PEELS measurements of 1-2-3 material began with Y. Zhu (Brookhaven National Laboratory). In support of the ordered intermetallic alloy development efforts (see Sect. 3.5), shape-memory nickel-aluminum alloys were examined by AEM, and progress was made in efforts to determine charge-density difference maps (i.e., degree of directional bonding) of intermetallic alloys by determining extremely accurate electron structure factors from intensity profiles of convergent beam electron diffraction patterns. Finally, the BES welding task (see Sect. 1.4) received continued electron microscopy support but at a reduced funding level.
3.4 STRUCTURE AND PROPERTIES OF SURFACES AND INTERFACES — L. L. Horton

The focus of the research in this group is the modification and characterization of surfaces and interfaces of materials. There are three subtasks in the group: (1) ion beam modification of ceramic surfaces, (2) growth of thin-film structures with MBE, and (3) growth of diamond and diamond-like films. The ion beam modification research is a collaborative effort with the Solid State Division and the University of Tennessee; it is funded by BES and the CTAHE Program (two doctoral students). Support for the MBE research is provided by the Air Force Office of Scientific Research and Wright Research and Development Center and by the Army Strategic Defense Initiative and an ORNL Exploratory Funds Director's R&D project for the diamond film research. As of FY 1992, this group was dissolved and the research tasks assigned to other groups. The MBE and diamond research were merged with the X-ray Group, and the ion beam research was incorporated into the BES Radiation Effects Task.

3.4.1 Ion Beam Modification of Ceramics

In this program, ceramic surfaces are implanted with various ion species and characterized with a number of techniques including Rutherford back-scattering (RBS), AEM, and conversion electron Mössbauer spectroscopy (CEMS). Adhesion studies focus on metal and ceramic films on ceramic substrates and utilize standard pull tests and scratch tests. For FY 1992, the funding for this effort was significantly reduced.

Studies of ion implantation of α-SiC have continued. Electron microscopy was performed with both cross-sectional and back-thinned specimens implanted with $6 \times 10^{16}$ Fe-ions/cm$^2$ (160 keV, room temperature). The depth of the peak iron concentration measured with energy dispersive X-ray spectroscopy (EDS) and with RBS was in approximate agreement with the value calculated by the TRIM code, assuming a specimen density equal to crystalline silicon carbide. The AEM examinations did not reveal the presence of any precipitates, suggesting that the iron had not coalesced into precipitates (observation limit $= 2$ nm). Inspection of the electron energy loss fine structure for iron suggests that the iron is not metallographically bonded; this result supports the conclusion from CEMS that the iron is covalently bonded.

A guest in the group, Prof. Paul Thevenard from the Université Claude Bernard Lyon I, France, brought new insight to our continuing efforts to characterize ion implantation in Al$_2$O$_3$. Chemical interactions and microstructural evolution due to 150-keV niobium implantation (room temperature, $10^{16}$ to $2 \times 10^{17}$ ions/cm$^2$) were studied. Above $5 \times 10^{16}$ ions/cm$^2$, an amorphous phase containing small, 1-nm av diam, metallic niobium precipitates was observed. Electrical conductivity measurements showed that a hopping mechanism was likely for this system. In addition, a new study of the effects of shock waves on precipitate dissolution in MgO was initiated. This study investigated the effects of ultrasonic cavitation, ion bombardment, and laser excitation on potassium aggregates formed by thermal annealing of MgO specimens implanted with $5 \times 10^{16}$ K$^+$-ions/cm$^2$.

Studies to evaluate the effects of implantation of different ion species on the adhesion of 100-nm-thick iron films on Al$_2$O$_3$ were concluded. These studies were designed to ensure that the ion concentration profiles, damage profiles, and recoil distributions were the same for each ion species. For a fluence of $1 \times 10^{15}$ ions/cm$^2$, implantation of Cr (300 keV) and
Fe (320 keV) increased the bond strength, whereas implantation of Ni (340 keV) did not. The effect is thought to be due to changes in the interfacial energy resulting from the presence of the ion species at the interface. Only a narrow zone is affected; the mixing at the interface is less than 10 nm.

### 3.4.2 Diamond Film Research

This program focuses on the growth and characterization of diamond films grown with hot filament-activated CVD processes. The goal of the research is to develop an understanding of the growth processes and use this knowledge to improve the properties of the films for specific technological needs. The approach is to characterize and correlate the growth conditions, film structures, and film properties. Characterization techniques include SEM and AEM, Raman spectroscopy, and XRD. Mechanical, tribological, optical, and electrical properties are evaluated. The substrate temperature, filament temperature, and molecular species present in the exhaust gases are monitored. Collaborative work has been performed with North Carolina State University and the University of Florida.

Highlighting the past year was the development of insight into the growth mechanisms for diamond films. It was determined that the texture of diamond films could be predicted by the Van der Drift model. The relative $<100>$ and $<111>$ growth rates were determined from the shapes of individual crystallites. The relative growth rates were correlated with specific growth conditions. This correlation was used with the Van der Drift model to grow films with selected textures. The relationship between the orientation of the surface facets and the underlying defect concentration was established; this effort was greatly facilitated by a new specimen preparation technique for diamond films developed by the BES microscopy task. It was shown that the internal twins and stacking faults observed in diamond films were confined to the regions resulting from the growth of $\{111\}$ facets; regions directly beneath $\{100\}$ facets contained only line and point defects.

Determination of the molecular species in various regions of the reactor is the focus of a Director's R&D program. Combining the expertise in laser spectroscopy from the Analytical Chemistry Division with the materials expertise in M&C Division, a hot filament growth chamber was constructed for laser spectroscopic analyses of the bulk gas, of the region immediately above the growing surface, and at the actual surface. In the M&C component of this investigation, the nanostructure of growth surfaces has been characterized with high-resolution SEM, atomic force microscopy, grazing incidence REM, and TEM. Sufficient data have been gathered and correlated to allow the growth of films with a specific texture and crystallographic faceting. The next stages of this study will correlate the molecular spectroscopy data with the structural characterization.

### 3.4.3 Molecular Beam Epitaxy Thin-Film Growth Studies

In this program, thin-film and multilayered oxides are grown with MBE ultrahigh-vacuum processing technology. The primary emphasis is the fundamental growth mechanisms for oxide ceramic materials. However, several practical technological advancements can be made with high-quality epitaxial oxide thin-film structures; i.e., optical waveguides and lubricious oxide surfaces. Among the areas under study are homoepitaxy and heteroepitaxy in the alkaline earth oxides, metal silicide precursor phases as oxide template structures for new substrate materials, and layer-by-layer growth mechanisms in perovskite structures.
The epitaxial structural development and surface reactions of barium metal with silicon have been studied as a function of surface coverage. BaSi$_2$ is orthorhombic and grows epitaxially on Si(001) and Si(111) for surface coverages greater than 1 monolayer (ML). From 0 to 1 ML a series of ordered surface structures develops that suggests a cubic Ba-Si compound. Reflection high-energy electron diffraction (RHEED), Auger, and X-ray photoelectron spectroscopy (XPS) data have been obtained that support a chemical reaction and compound formation between barium and silicon for all barium coverages. In bulk form, at high pressures, BaSi$_2$ can be stabilized into the cubic SrSi$_2$ structure. Epitaxial strain at the silicon surface may provide a mechanism for stabilizing the low-coverage precursors of the orthorhombic BaSi$_2$.

Thin-film, multilayer, epitaxial structures of BaSi$_2$, BaO and BaTiO$_3$ have been grown on the (001) face of silicon. The epitaxy is BaTiO$_3$(001)BaO(001)Si(001) and BaTiO$_3$<110> BaO<100> Si<100>. The epitaxial growth of BaO is accomplished without silica formation at the BaO/Si interface by stabilizing BaSi$_2$ as a submonolayer template structure. Source shuttering for the metal species, coordinated with cyclic oxygen arrival at the growing oxide surfaces, significantly improved the film quality. In situ ellipsometric measurements of the indices of refraction for BaO and for BaTiO$_3$ in the multilayer yielded 1.96 for BaO and 2.2 for the BaTiO$_3$, within 10% of the values for bulk materials. These values suggest that this structure can be developed as an optical waveguide.

Anomalous XRD methods were used to measure the contribution of an epitaxial copper overlayer on silicon to the crystal truncation rods (CTRs). The intensity of the CTRs was analyzed to determine interface structure; the epitaxy of a large-misfit system, copper on Si(111), was described in relationship to this structure. Anomalous scattering observed at the CuK edge along an Si CTR demonstrated that the copper atoms were in registry with the Si(111) surface. The copper atom registry at the interface was modeled based on the known bulk η-Cu$_3$Si structure and observed epitaxy.

### 3.5 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 five major tasks: (1) determination of physical metallurgy and mechanical behavior of ordered intermetallics, (2) design of intermetallic alloys for industrial and energy-related use, (3) design and characterization of iridium alloys for space power systems, (4) processing of lithium hydride by rapid solidification, and (5) design and construction of a high-temperature mechanical properties microprobe (HTMPM). The first two tasks are closely related, with emphasis on design of new high-temperature materials through control of alloy composition, crystal structure, atomic bonding, microstructure, and processing techniques.

We have recently made significant advances in our understanding of the physical mechanisms responsible for the yield and flow strengths and the fracture toughness and ductility of ordered intermetallics. Ground-state elastic constants, point-defect self-energies, various fault energies, and cleavage energies have been determined from first-principles
total-energy calculations. From the results of these calculations, continuum modeling of dislocation and fracture mechanics behavior was performed to predict the intrinsic properties of yield strength and fracture toughness in transition-metal aluminides (e.g., Ni$_3$Al, Pt$_3$Al, Al$_3$Ti, Al$_6$Sc, NiAl, FeAl, and TiAl). This interdisciplinary effort by first-principles quantum theory and applied continuum mechanics clearly demonstrates the predictive capability for intrinsic mechanical properties of crystalline solids. A comprehensive understanding of the bonding mechanisms, point-defect structure, yield strength anomaly, and brittle fracture behavior forms the basis for a better assessment of extrinsic effects on the strength and ductility of ordered intermetallics, such as solid-solution strengthening and hydrogen embrittlement.

In particular, for NiAl, we find that the primary reason for $<$100$>$ slip is the combined effect of a high APB energy and a weak repulsive elastic force between superpartials. The active slip system of $\{112\}$$<$111$>$ in FeAl at low temperatures is predicted on the basis of the anisotropic coupling effect of non-glide stresses. The calculated ideal cleavage energies are consistent with the reported (110) habit plane of NiAl but not with the (100) plane of Fe-40% Al. The crack-tip shear stresses calculated for the $\{011\}$$<$100$>$ and $\{112\}$$<$111$>$ slip systems (crack-blunting types) in NiAl and Fe-40% Al, respectively, enable us to explain the reported cleavage habit planes and fracture toughness values reasonably well. The effect of resolved normal stresses on the modified Plesier stress is found to suppress the $\{112\}$$ [$111$]$ slip more at the (100) Mode-I crack tip than at the (110) crack tip.

During FY 1991, we extended our study of grain-boundary chemistry and fracture to include NiAl alloys, which were investigated as functions of both boron doping and alloy stoichiometry. Our results indicate considerable similarity between NiAl and Ni$_3$Al: in both cases, boron suppresses grain-boundary fracture in Ni-rich alloys but not in Al-rich alloys. Also, in both alloy systems, the boron concentration at the grain boundaries increases with increasing nickel and boron concentration in the bulk. Finally, there is no strong Ni-B cosegregation in either alloy.

As in the case of Ni$_3$Al, there may be at least two possible explanations for why boron suppresses intergranular fracture in NiAl: boron enhances Gaussian Basis (GB) cohesion and/or it facilitates slip transfer. We have investigated the latter possibility by studying the Hall-Petch behavior of high-purity, arc-melted, NiAl alloys as a function of boron doping and alloy stoichiometric. The results indicate that the Hall-Petch slope ($k_y$) is nearly zero for stoichiometric NiAl (with or without boron) and also for Ni-rich NiAl (51% Ni). In contrast, $k_y$ is mildly positive in Al-rich NiAl (51% Al). Since all three alloys fracture intergranularly and boron suppresses intergranular fracture without significantly changing $k_y$, our results indicate that there is no simple correlation between the magnitude of the Hall-Petch slope (and, therefore, the ease of slip transfer) and propensity for intergranular fracture.

Environmental effects on mechanical properties and fracture behavior of $L1_2$-ordered (Co$_{50}$Fe$_{50}$)$_3$V alloys with compositions (Co$_{50}$Fe$_{50}$)$_3$V and (Co$_{50}$Fe$_{50}$)$_3$V were studied by tensile test in the strain rate range of $3.3 \times 10^{-5}$ to $3.3 \times 10^{-1}$ s$^{-1}$ at ambient temperatures. The (Co$_{50}$Fe$_{50}$)$_3$V alloys were found to be susceptible to environmental embrittlement. The yield and flow stresses were insensitive to the test environment, while the ductility and ultimate tensile strength decreased according to the sequence of dry oxygen, vacuum, air, and distilled water. The ductility loss was closely associated with intergranular fracture, and the propensity for intergranular fracture increased in the same environmental sequence. Lower strain rate
resulted in more intergranular fracture and hence lower ductility. A beneficial effect of grain refinement was observed. All these results suggest that the embrittlement was caused by moisture-induced hydrogen, which diffused to grain boundaries, resulting in reduced grain-boundary cohesion and increased intergranular fracture.

Our study of environmental effects on tensile properties of Ni$_3$Si indicates that the tensile ductility is very sensitive to test environment at room temperature. The silicide showed no appreciable plastic deformation when tested in air and an elongation of 7.5% when tested in dry oxygen. These results clearly demonstrate that Ni$_3$Si is prone to environmental embrittlement at room temperature. Thus, an extrinsic factor—environmental embrittlement—is a major cause of low ductility and brittle grain-boundary fracture in Ni$_3$Si. It should be noted that the environmental effect appears not to be the sole source of grain-boundary brittleness in Ni$_3$Si because elimination of the environmental effect by testing in dry oxygen does not lead to extensive ductility (e.g., ~ 30%) and complete suppression of intergranular fracture. Further studies will address the effect of boron additions on environmental embrittlement in Ni$_3$Si at ambient temperatures.

Continuous-fiber reinforced metal-matrix composites consisting of Ni$_3$Al alloys and Saphikon AI$_2$O$_3$ single-crystal fibers were fabricated by hot pressing of fiber-foil layups. Two matrix compositions were employed, namely, IC50 (Ni-22.5Al-0.5Zr-0.1B, at. %) and IC396M (Ni-15.9Al-8.0Cr-0.5Zr-1.7Mo-0.02B, at. %). Etching of the foils in aqueous FeCl$_3$ solution prior to layup and hot pressing tended to improve fiber-matrix bonding and the density-normalized, room-temperature yield stress. Whereas strength improvements for the IC50 matrix were only moderate, significant improvements were found for an IC396M composite reinforced with 10 vol % of Saphikon fibers.

The intermetallic B2 compound Fe-40Al (at. %) was mechanically alloyed with fine Y$_2$O$_3$ particles in argon in a Turbula T2C mixer. Process parameters such as average energy per collision, powder quantity, additions of elemental powders, and process control agents were explored. The milling process was followed by a variety of techniques including X-ray peak broadening. Creep tests were performed with HIPed Fe-40 at. % Al containing 2 vol % Y$_2$O$_3$. At 1200 K the creep strength was improved by up to a factor of four, and a high-stress exponent characteristic of oxide dispersion-strengthened materials was found.

Recent alloy development efforts have shown that Fe$_3$Al-based alloys can have room-temperature tensile ductilities of 10 to 20% and yield strengths of 500 MPa at temperatures to 600°C. Binary Fe$_3$Al alloys have low creep resistance, but the addition of 1 to 2 at. % Mo or Nb improves the creep life and reduces the minimum creep rate, with niobium being the most effective ternary addition. The improvement in creep life of the Fe$_3$Al + 1Nb is the result of a combination of factors, which include grain-boundary strengthening, resistance to dynamic recrystallization during stressing, precipitation strengthening, and changes in the formation and mobility of the dislocation network. Correlation of optical, scanning electron, and TEM data suggests that the addition of niobium results in a strengthening of the grain boundaries by solid-solution effects and formation of fine-matrix MC precipitates, which pin dislocations and thereby strengthen the matrix.

Macroalloying process has been used to improve the mechanical properties of NiAl-base alloys. Several macroalloyed NiAl alloys were prepared and fabricated into bar stock by hot
extrusion at 900 to 1050°C. We have found that the mechanical properties of NiAl can be dramatically improved by small amounts (< 2 at. %) of alloy additions. These alloys showed excellent strength and creep resistance at elevated temperatures. Alloy additions in both solid-solution form and second-phase precipitates extend the creep-rupture life at 760°C and 138 MPa by more than five orders of magnitude. The NiAl alloys also showed excellent oxidation resistance at 1000°C.

Ir-0.3-W alloys containing nominally 60 W ppm thorium have been developed at ORNL for cladding plutonium oxide fuel in RTGs. The thorium is added to provide adequate high-temperature ductility for post-impact containment of the fuel in case of an accident. Without thorium, the iridium alloy fails by brittle grain-boundary separation at high temperatures and strain rates. The beneficial effect of thorium is believed to come from both its enrichment at the grain boundaries of iridium, resulting in improved grain-boundary cohesion, and also its precipitation as Ir₅Th intermetallic particles, which pin the grain boundaries and refine the grain size. Our current research utilizes these basic ideas to find suitable substitutes for thorium in iridium. Among the factors we consider are the size misfit of the dopant element with the iridium matrix (which drives both segregation and precipitation), likelihood that the dopant element is chemically similar to thorium, and potential for forming low-melting eutectics (to minimize hot-cracking tendency during welding of components made from the alloy). Using such simple concepts, we have already identified rare-earth elements, which segregate strongly to the grain boundaries in iridium and also refine grain size as well as or better than thorium. Preliminary tests indicate that these desirable properties are not obtained at the expense of weldability (at least in some of the alloys).

In addition to the alloy development effort, our group also conducted Ir alloy qualification and characterization studies as part of the Space Power Program. Included in this task were high-temperature tensile impact testing, grain growth studies in vacuum and low-pressure oxygen, Sigmaijig weldability testing, and construction of a new high-temperature tensile impact tester capable of testing iridium alloys at 1000°C and extension rates of 60 m/s.

The Melt Spinning Process Prototype (MSPP), funded by the Development Division at Y-12, met two MMES Awards Milestones in 1991 to produce certain quantities of LKH flakes by melt spinning. Powder compacts (prepared by the Special Materials Processing Group, Y-12) from these flakes had densities of > 99% of the theoretical density. This method has now been shown to be competitive with other LKH powder preparation techniques. The semi-continuous operation produces large quantities of stoichiometric, fine-grained flakes or powder directly from molten lithium hydride. The morphology of the flakes can be controlled by changes in melt temperature, wheel speed, and ejection pressure. Additional funding was obtained from Y-12 for the study of powders produced by rotary atomization. The initial program is to examine the feasibility of powder production by this method. A basic experimental apparatus has been put into operation and has produced powder particles of metallic tin in the 2-mm size range.

Significant progress has been achieved on several of the important subsystems of the HTMPM. Foremost among these is the phase-sensitive, laser interferometry, displacement-measuring system. A system with 0.1-nm resolution and a measurement time constant of > 1 ms has been built and bench tested. The optical imaging system for indent
placement is being installed on the instrument. Work is also proceeding on the indentation head. Several of the critical components have been fabricated, with only the indenter column still to be designed. Motorization of the sample manipulator is being implemented. Preliminary high-temperature indentation experiments should be performed during the 1991-92 calendar year.

3.6 DEFECT MECHANISMS — L. K. Mansur

Research in the group centers on the science underlying the effects of displacement- and ionization/excitation-producing particle fluxes on materials. The motivation for the work originates in both the need to understand the behavior of structural materials subjected to neutron irradiation in fission and fusion reactors and the opportunity to create new materials with improved properties by a variety of energetic particle irradiations. In the former area, principles are developed to underpin the design of radiation-resistant materials. In the latter, new materials knowledge and techniques are developed that are relevant to a variety of technologies. Microscopy studies and ion beam characterization techniques are used, together with a variety of more standard tools of materials science, such as miniaturized and conventional mechanical properties measurements. Both the radiation effects and materials modification work benefit from the same fundamental base of functional expertise.

3.6.1 Radiation Effects

Our radiation effects experiments are now concentrated on neutron irradiations aimed at elucidating certain aspects of embrittlement of RPV steels and verifying reports of radiation-induced softening in aluminum alloys. Low-temperature embrittlement of ferritic steels is a major focus of this work. We have collaborated with researchers from the University of California at Santa Barbara in their design of an extensive irradiation experiment to investigate the physical mechanisms responsible for this phenomenon. Two series of simple model alloys and a series of model commercial alloys have been fabricated. These materials will be irradiated at 50°C in the form of miniature tensile specimens, microhardness disks, and coupons for use in small-angle, neutron-scattering experiments. The microhardness disks are also suitable for TEM examination. The irradiation matrix encompasses a range of fluences from $2 \times 10^{21}$ to $2 \times 10^{23}$ n/m$^2$ with fluxes from $2 \times 10^{14}$ to $2 \times 10^{16}$ n/m$^2$.s. The post-irradiation examination (PIE) will permit us to determine the flux and fluence dependence of low-temperature embrittlement and to investigate the compositional sensitivity of the various defects, e.g., precipitates and point-defect clusters, that are believed to be responsible for the embrittlement. Several commercial alloys will be irradiated in this experiment along with the model alloys. The commercial alloys include the A212 and A350 alloys used in the HFIR pressure vessel and the A212 alloy used in the Shippingport Reactor-Neutron Shield Tank (SR-NST). The HFIR and SR-NST specimens include those fabricated from unirradiated archive material and specimens cut from Charpy bars used in the reactor surveillance programs. These commercial alloys will be used to investigate post-irradiation annealing and re-irradiation embrittlement and to determine the relative irradiation sensitivity of the HFIR and SR-NST materials in a single radiation environment.

In previous reports in this series, we have described the early embrittlement of the HFIR vessel and our research to fully describe the mechanistic origins of the effect. Our work
centers on the highly thermalized neutron spectrum at the vessel, and a special spectral effect experiment is now under way to explore this explanation in detail. We have continued to develop our ability to model the embrittlement of ferritic steels. The use of the fully time-dependent rate theory has enabled us to investigate the low temperatures and low doses where this embrittlement occurs. Two types of defects are believed to contribute to the hardening that leads to embrittlement. One type is a small, copper-rich precipitate, and the other is likely to be some type of point-defect cluster. Our modeling work has focused on determining the characteristics of this latter defect. A model has been developed that examines the formation and evolution of both interstitial and vacancy clusters. These clusters can be formed either by essentially classical nucleation via reactions between individual point defects or directly as the displacement cascades collapse. Initial calculations with the clustering model have shown that interstitial clusters may play a dominant role at low temperatures and high damage rates and that vacancy clusters are more important at higher temperatures or low damage rates. The level of hardening induced by the predicted point-defect cluster concentrations is comparable to that observed experimentally. It is also comparable to the level of precipitation-induced hardening that has been calculated by others. These results imply that more than one type of defect may contribute to ferritic steel embrittlement and that hardening may be dominated by different defect types in different flux, fluence, and temperature regimes. In such a case, the potential for crossing regime boundaries requires that caution be used when the prediction of property changes involves any significant data extrapolation.

Usually, metals undergo hardening under neutron irradiation. Aluminum is no exception. But there are isolated reports that cold-worked or precipitation-hardened aluminum alloys display radiation-induced softening. The possibility of such softening is important for the HFIR, whose core contains many components built from 6061 aluminum alloy in the precipitation-hardened condition. Therefore, we have searched for signs of softening in 6061-T6 tensile specimens irradiated in the HFIR hydraulic tube to a range of fluences encompassing those for which softening was claimed. We have not confirmed the softening. However, the displacement rate in the HFIR hydraulic tube is very much higher than those used in the softening claims. It is possible that because of enhanced bulk recombination at high displacement rates, a smaller fraction of freely migrating point defects is available, and, hence, softening is pushed to higher fluences or is avoided. We are now seeking softening at lower displacement rates.

3.6.2 Materials Modification

In the last report in this series, we summarized our initial, highly encouraging results on the ion beam modification of polymer surfaces. We have now expanded this work to examine in more detail the physical bases of the observed large improvements in hardness and wear resistance. Additional materials and conditions have also been examined.

Polyimide Kapton and spin-cast polyamic acid (PAA) on sapphire have been implanted with 1 MeV Ar-ions to a dose of $4.7 \times 10^{18} \text{ m}^{-2}$ at ambient temperature. The hardness of both pristine and implanted surfaces was characterized by a depth-sensing, low-load nanoindentation technique. The hardness of Kapton was found to be increased by over 30 times after Ar implantation, from 0.43 to 13 GPa at 100-nm indentation depth. The ion-beam-treated material is thus about 5 to 8 times harder than 316 type stainless steel. Similar
increase of hardness was also observed for PAA. This result suggests that ion-beam-modified, spin-cast PAA film may have potential technological applications for protective coatings where hardness and wear resistance are required. In order to put this hardness measuring technique on an established basis for polymeric materials, experiments were carried out to investigate the effects of substrate, indentation rate, relaxation, and indentation mode. The results showed that (1) hardness was depth dependent and decreased with increasing indentation depth; (2) measurements of the ion-beam-hardened surface on soft substrate underestimated the hardness at greater depths, whereas measurements of the treated material on the sapphire substrate overestimated it; (3) although strain-hardening became noticeable with increasing indentation rate, the hardness values measured in the range of indentation rates from 2 to 50 nm/s were almost invariant; and (4) hardness values measured using an AC-modulated load showed very little depth dependence. The hardness value at 100-nm depth is recommended to be used as a standard reference value, since the hardness value at this depth was found to be almost independent of substrate, indentation rate, indentation method, and artifacts at or near the surface.

In related work, polycarbonate (Lexan) was implanted with 100- and 200-keV B ions to doses of 0.26, 0.78, and 2.6 x 10¹⁹ ions/m² at room temperature (< 100°C). Mechanical characterization of implanted materials was carried out by nanoindentation and sliding wear tests. The results showed that the hardness of implanted polycarbonate increased with increasing ion energy and dose, attaining hardness up to 3.2 GPa at a dose of 2.6 x 10¹⁹ ion/m² for 200-keV ions, which is more than 10 times that of the unimplanted polymer. Wear properties were characterized using a reciprocating tribometer with nylon, brass, and Society of Automotive Engineers (SAE) 52100 Cr-steel balls with 0.5 and 1 N normal forces for 10,000 cycles. The wear mode varied widely as a function of ion energy, dose, wear ball type, and normal load. For given ion energy, load, and ball-type conditions, there was an optimum dose that produced the greatest wear resistance and lowest friction coefficient. For polycarbonate implanted with 0.78 x 10¹⁵ ions/m², the nylon ball produced no wear after 10,000 cycles. Moreover, the overall friction coefficient was reduced by over 40% by implantation. The results suggested that the potential of ion beam technology for improving polycarbonate is significant and that surface-sensitive mechanical properties can be tailored to meet the requirements for applications demanding hardness, wear, and abrasion resistance.

In order to understand the ion-polymer interactions that lead to improved surface properties of ion-irradiated polymers, we have made quantitative measurements of the gases evolved from the polymers polyethylene and Kapton during irradiation. The singly-charged ions used for irradiation in this work were 200-keV H₂⁺, 200-keV He⁺, 200-keV B⁺, and 1000-keV Ar⁺. The penetration depth of each of these ions is approximately 1 μm. Chemical G-values (number of molecules released per 100 eV of absorbed energy) for the gases H₂, CH₂, C₂H₂, CO, and CO₂ were measured. It was found that the G-values were strongly dependent upon the ion atomic number. It was shown that this atomic number dependency could be related to the probability of atomic recoil or displacement in the bulk of the polymer. In addition, the G-values appear to be correlated with the large increases in surface hardness after ion irradiation was measured for these polymers.
3.7 STRUCTURAL MATERIALS — A. F. Rowcliffe

The primary focus of the group is the development of structural materials for fusion and fission reactor applications; funding is mainly from the DOE, Office of Fusion Energy, and from the NPR Program.

The multi-national program to design and build the International Thermonuclear Experimental Reactor (ITER) is moving into the engineering design phase. Correspondingly, the Fusion Materials Program at ORNL is being refocused to address the following major tasks in the ITER R&D program in structural materials: (a) austenitic stainless steels for the FWB structure; (b) structural materials for the divertor and blanket stabilizer; (c) structural alloys for an advanced blanket module; and (d) ceramic materials for diagnostic systems, insulators, feedthroughs, and windows.

An assessment was completed of the properties of a range of U.S. and Japanese steels irradiated to 7 dpa in an Oak Ridge Research Reactor (ORR) experiment spectrally tailored to reproduce the damage and helium generation rates characteristic of ITER. Further irradiation of this experiment to 20 dpa is continuing satisfactorily in the HFIR at temperatures of 60 and 330°C. Capsules for operation at 200 and 400°C will be inserted in the HFIR in mid-1992. The results of the first phase of the spectrally tailored experiment raised several issues requiring further investigation. Irradiation at 60 to 400°C produced large increases in yield stress and severe reductions in work-hardening capacity, which will have some impact on fracture toughness. Accordingly, three HFIR capsules were designed, constructed, and irradiated in HFIR target positions to a dose of 3.5 dpa. The capsules operate at 100 and 250°C and contain miniature compact tension specimens, sheet tensiles, and TEM discs fabricated from U.S., Japanese, and European Community alloys. A second important issue is iASC in water. Preliminary electrochemical measurements on materials irradiated in the spectrally-tailored experiments indicate that the ITER radiation environment will not induce susceptibility to cracking at temperatures below 330°C. Confirmation through in-cell SSRT in an autoclave is planned. A third ITER FWB-related issue is that of repair welding once the machine has been activated. In a study conducted jointly with Auburn University, autogenous, single-pass, full-penetration welds were produced in type 316 stainless steel using a technique to introduce compressive stresses during welding. Helium was uniformly introduced into steels prior to welding using tritium doping and decay. Experimental results showed that the grain-boundary helium bubble growth was effectively suppressed by the stress technique. Additionally, the bubble growth was altered such that the bubbles occurred predominantly on grain boundaries perpendicular to the weld, whereas previously they occurred principally on grain boundaries parallel to the weld direction. These results suggest that the proposed stress GTA welding technique may be used to eliminate the catastrophic cracking encountered during welding of helium-containing materials.

For advanced blanket modules, several ferritic-martensitic steels are being evaluated, and reduced activation versions are being developed. Neutron irradiation experiments in HFIR, which simulated the simultaneous generation of helium and displacement damage characteristic of a fusion reactor first-wall structure, showed that ferritic-martensitic steel structural alloys such as HT-9 and 9Cr-1MoVNb are highly resistant to both void swelling and grain-boundary embrittlement. Irradiations were carried out at 400 to 600°C to displacement doses (~80 dpa), which approach expected lifetime doses in fusion power reactors.
Neutron irradiation of HT-9 and 9Cr-1MoVNb at temperatures ≤ 400°C typically induces an increase in the DBTT of at least 100°C. These materials may then be susceptible to rapid-cleavage crack propagation during certain low-temperature operating conditions. Alternative ferritic-martensitic steels have been developed with compositions tailored to reduce long-term radioactivity by three to four orders of magnitude. Initial results indicate that one of these new alloys based upon 9Cr-2W,V,Ta has excellent resistance to radiation-induced loss of toughness. Neutron irradiation to ~7 dpa at 365°C resulted in an increase in DBTT of only 4°C. This is an encouraging indication that it should be possible to overcome the low-temperature radiation embrittlement problem in this class of alloys.

Experiments have been conducted to measure the dielectric properties of candidate ceramic materials for Ion Cyclotron Resonant Heating (ICRH) and Electron Cyclotron Resonant Heating (ECRH) systems. Measurements were made in situ in both an ionizing irradiation field (HFIR spent fuel) and in a combined ionizing and displacive irradiation field [Training, Research, Isotope Production-General Atomic Reactor (TRIGA)]. The irradiation fields are comparable to those in ITER. The purely ionizing radiation did not increase the loss tangent in any of the materials tested. However, in the pulsed TRIGA reactor experiment, loss tangents increased significantly. For alumina, the loss tangent increase was sufficient to raise concerns about the cooling requirements and design for RF windows and insulator supports for ITER.

Significant progress was made in three other areas of materials development. FeAl alloys were produced with a combination of major and minor element additions that significantly improve the creep-rupture resistance of these alloys at 600°C and 207 MPa. The alloys were designed on the basis of controlling microstructural phenomena, including recrystallization/recovery/grain-growth processes and the formation of fine-precipitate dispersions that pin dislocations. A set of 3Cr bainitic steels is being developed as radiation-resistant structural steels for fusion reactor applications with lower radioactivity after service and faster decay of that radioactivity after disposal. These new steels also show high strength and toughness properties that make them candidates to replace commercial modified 9Cr-1Mo and 2%Cr-1Mo steels in many non-nuclear, high-temperature applications. These new steels have high strength and good impact toughness even without tempering, a unique metallurgical condition for service not available with martensitic and most bainitic steels, which require tempering. The third area involves the possible application of SiC/SiC composite materials for fusion reactor systems. Microindentation techniques are being used to measure the elastic modulus of CVD silicon carbide/Nicalon composites irradiated to 30 dpa with carbon ions at room temperature. Samples were prepared in cross section, and the microstructure was analyzed and modulus was measured along the damage path of the carbon ions. Both CVD silicon carbide and Nicalon silicon carbide were seen to amorphize at room temperature with the threshold for the CVD being approximately 15 dpa. Amorphization of Nicalon silicon carbide fiber was seen to occur over the entire range of the carbon path, implying a much lower threshold for amorphization. Elastic moduli were seen to decrease significantly (from 440 to 280 GPa) for the CVD material with the minimum modulus corresponding to the maximum damage region of the carbon beam. The modulus of Nicalon showed the opposite behavior. The modulus for the fiber was increased over most of the damaged region from an undamaged value of 170 GPa to a maximum of 210 GPa.

Incoloy 800H has been selected for several in-vessel components for the MHTGR. The temperatures and fluences required are outside the scope of the existing data base for
irradiated 800H. Accordingly, an evaluation was made of available irradiation facilities to achieve the proper neutron spectrum needed to obtain the correct concentrations of transmutation-induced helium to properly simulate the MHTGR irradiation conditions. In addition, an HFR rabbit irradiation experiment has been designed in order to do scoping experiments in a thermal reactor with the correct spectral conditions expected for the upper-core internal components. Such experiments will evaluate the effects of helium on the high-temperature tensile properties of Incoloy 800H. It will be the first such experiment to evaluate the effect of radiation on welds in Incoloy 800H.

3.8 SUPERCONDUCTING MATERIALS — D. M. Kroeger

The goal of the research conducted in the Superconducting Materials Group is to develop high-temperature oxide superconductor materials, which can support large-current densities in high magnetic fields. Materials based on two different systems have shown some promise, and problems associated with each type are being investigated. Results on thin films show that the intrinsic properties of YBa$_2$Cu$_3$O$_x$ are not severely degraded at the boiling point of nitrogen, but so-called weak links associated with grain boundaries provide a high resistance to current flow in polycrystalline samples. This resistance may be caused by structural disorder, nonstoichiometry, secondary phases, or microcracks, and studies are in progress concerning the microstructural origin of the resistance. This research also includes work on the synthesis in properties of two additional superconducting YBaCuO compounds.

Weak links do not appear to offer as serious a limitation to the application of materials containing one of the BiSrCaCuO compounds, but the properties fall off rapidly at temperatures above ~ 30 K. This limitation is also being investigated along with methods for production of phase-pure, Pb-doped superconductor powder. For both classes of materials, approaches to fabrication of continuous lengths of conductor that are compatible with development of favorable texture and reduction of grain-boundary resistance are studied.

Phase stability is being studied because the information is needed for developing processing strategies, and much of the published information is unreliable. Recently, our studies of the YBaCuO system have been concentrated on Y124, YBa$_2$Cu$_4$O$_{8}$. This compound has several advantages over YBa$_2$Cu$_3$O$_{7-x}$, including its enhanced stability, lower thermal expansion, freedom from the tetragonal-orthorhombic phase transformation, and relatively temperature-independent oxygen content. Methods for synthesizing dense, phase-pure samples have been developed, and stability studies have shown that Y124 does not decompose at oxygen pressures up to 150 atm. These studies also show that at 950°C and 150 O$_2$, atm Y123 - CuO mixtures decompose into Y$_2$BaCuO$_5$, Ba$_2$Cu$_3$O$_6$, and Y124.

A disadvantage of Y124 is that its critical temperature is 80 K, as opposed to 91 to 93 K for Y123. Partial substitution of Ca for Y has been reported to increase the T$_c$ of Y124 to 90 K, but efforts to synthesize this material have been only partially successful. An extensive set of experiments has shown that (Y$_{0.9}$Ca$_{0.1}$)Ba$_2$Cu$_3$O$_x$ is not a single-phase material and contains Y123, Y124, and insulating compounds. Both microprobe and analytical TEM data showed that the Ca content of the Y123 phase is about 2.5 times higher than that of the Y124. Synthesis of the Ca-substituted Y123 and Y124 showed that Ca additions depress the T$_c$ of Y123 and increase T$_c$ of Y124.
Melt processing involves slowly cooling material from the liquid-plus-solid region and has been shown to produce high-current density samples. This improvement involves texture produced by the process and the special nature of the boundaries, which are present in the samples. Melt-textured Y123 samples contain grains which are ~ 4 to 5 mm long and 3 to 4 mm wide, and TEM has been used to show that the physical properties are related to a unique microstructure. The domains consist of parallel platelets approximately 5 to 10 μm thick, the c-axes of which are aligned. The plates are separated by planar boundaries, which are parallel to the basal plane. Similarly prepared samples have been shown to carry high currents in high magnetic fields. Weak link behavior (i.e., rapid decrease of J_c with field) is not seen for conduction either parallel or perpendicular to the common c-axis of the platelets.

TEM confirmed results from Auger analyses of fracture surfaces, which showed that the boundaries are highly non-stoichiometric. Of more interest was the finding that the boundaries tend to be more than 100 Å thick, ranging up to 1000 Å, and contain largely amorphous material. The boundaries examined are clearly not conducting. However, convergent-beam electron diffraction (CBED) indicated that not only the c-axes, but also the a- and b-axes of adjacent platelets are parallel (to within less than 1°). Also, SEM examination of fracture surfaces, TEM, and light microscopy of polished cross sections suggest that the boundaries between plates, although long, are not always continuous. There are occasional terminations of boundaries which, probably, provide a continuous path for current transmission in the c-direction. These results suggest that the domains in melt-processed material are, in fact, large single crystals with planar defects (the boundaries). Metallographic examination of the growth-front region between domains suggests that the boundaries result from rapid growth in the a-b plane of parallel plates, separated by liquid, which subsequently grow together, trapping the liquid between them. In areas where 211 is available, the plates may grow together forming bridges with, at most, a very small-angle grain boundary.

A suitable substrate material is required for melt processing superconductors. The substrate physically supports the superconducting oxide and should be both electrically conducting and chemically compatible with the liquid. Silver is compatible with Y123, but its melting point is too low for melt processing of Y123. Research has shown that Pd additions, which significantly increase the solidus temperature, also do not promote chemical interactions between the metallic solid solution and liquid oxide solution. Current research involves an Ag-12% Pd substrate.

Although they are even more complex than the YBaCuO superconductors, the Bi-Sr-Ca-Cu-O compounds have several potential advantages. These include lower processing temperatures and current density results which suggest weak link behavior is not too important. Very encouraging preliminary results have been obtained on powder-in-tube conductor containing Bi(Pb)-2223 powder prepared by aerosol pyrolysis.

The Bi_{1.94}Pb_{0.34}Sr_{1.91}Ca_{2.03}Cu_{3.06}O_x powder was prepared by pyrolysis of an ultrasonically atomized nitrate solution. Process parameters that result in no measurable loss of lead have been devised. The powder particles are approximately spherical, have a narrow size distribution, and an average diameter of about 1 μm. The phase content depends on such aerosol pyrolysis parameters as hot-zone temperature and residence time. Powder-in-tube conductor was fabricated with this powder by drawing and rolling to a final
thickness of 250 \mu m. The core diameter was \sim 40 \mu m. Two heat treatments at a temperature near the point of partial melting were performed, the first for 3 h at a thickness of 500 \mu m and the second at the final thickness, for 100 h. The results suggest that use of highly homogeneous aerosol powders may permit greatly shortened heat-treatment times. Metallography indicates that the cores are nearly 100\% dense and contain a low-volume fraction of secondary-phase particles, which are no larger than a few microns. In conductor made from conventionally prepared powders, secondary phases tend to be much larger and interfere with the fabrication process.

A critical current density of $1 \times 10^4$ A/cm$^2$ at 77 K and $H = 0$ was obtained in conductor fabricated by drawing and rolling. This $J_\sigma$ value is among the highest reported for powder-in-tube material prepared by rolling. The highest $J_\sigma$ have been obtained by others in material that was deformed by pressing in final fabrications steps, a process not amenable to fabrication of continuous lengths.

3.9 IRRADIATED MATERIALS EXAMINATION AND TESTING — L. J. Turner

The primary mission of the group is to provide support for the PIE effort for structural materials research conducted by several groups within the M&C Division. As such, the group is responsible for operation and maintenance of the Irradiated Materials Examination and Testing (IMET) Facility in Building 3025E. Funding is provided from several sources, among these: the MFE Program, the Space Nuclear Power (SP-100) Project, the HSSI Program of the NRC, the NPR-MHTGR Program, the BES Program, and several smaller WFO Programs. During this reporting period, most of our experimental work involved the support of the Structural Materials Group of the Materials Science Section and the Fracture Mechanics Group of the Engineering Materials Section.

Work with the Structural Materials Group was primarily in support of the MFE Program. During this period, TEM disk density measurements were performed on ferritic and austenitic developmental alloys from the HFIR-CTR-50 experiment. Tensile testing of FWB structural developmental alloy candidates from the U.S./Japan collaborative testing program continued on specimens from ORR MFE 6J and 7J, two spectrally tailored experiments. Photography and magnetometer measurements were performed on 24 of these specimens. SEM of the fracture surfaces of broken, 1/3-size CVN specimens from the Materials Open Test Assembly (MOTA) 1E low-activation experiment and tensile specimens from ORR MFE 6J and 7J were conducted in cell 1. Leak detection tests were completed on pressurized tubes from ORR MFE 6J and 7J on the new equipment in cell 4. Disk fracture and SEM fractography were accomplished on TEM disks from experiments HFIR-CTR-50 and MOTA 1C, tube KA02, and MOTA 1E, tube KA01.

Tensile tests were conducted in cell 2 in support of radiation softening experiments conducted for the BES Program on precipitation-hardened 6061-T6 aluminum alloys used in the HFIR core and other structural components.

Tensile testing was begun on high-temperature Rhenium specimens for the DOE Space Nuclear Power Program, SP-100 Project. This work will continue into FY 1992.
Our work with the Fracture Mechanics Group dealt predominantly with support for the NPR-MHTGR Structural Materials Program. During this reporting period, tensile testing was started on specimens from experiment FNR-3, units A and B, capsules 5 and 7. This work will continue into FY 1992. The first of 20 crack-arrest duplex specimens from the HSSI Sixth Series Phase II experiments were sent to the Building 3525 hot cells to enlarge the crack starter holes. Crack-arrest testing will be completed on these initial specimens, and if the results are as expected, the remaining specimens will be prepared accordingly. Crack-arrest testing for the remaining specimens will be completed in FY 1992.

3.10 RESEARCH SUPPORT GROUP — A. T. Fisher

The work of the Research Support Group centers around specimen preparation for TEM and AEM studies and related services. Structural characterization of ceramic materials by TEM has increased considerably in the last year. Ceramics cannot be thinned to electron transparency by electrolytic thinning as can metal samples. New techniques for sample preparation by use of mechanical erosion for prethinning, followed by ion milling to electron transparency, are constantly being developed. We continued to produce all needle specimens for the field-ion microscopy (FIM).

Support for the Atom Probe Facility (APF) continued to increase. Two Fischione tip polishers were purchased. One polisher will be used for regular work, and the other will be used to prepare radioactive samples. We also upgraded our Mac plus computer to a Mac Ilsi and acquired a LaserWriter, which increases our capability to produce quality graphs, diagrams, schematics, etc., for publication. In specimen preparation of FIM specimens, a substitute for the carcinogen carbon tetrachloride was found. A procedure was developed using Galden Electronic Fluid, and at least three other probe laboratories around the world have adopted this procedure. Assistance was provided for the continuing assembly of two atom probe instruments, and 174 auto-probe samples were produced.

A new ceramic specimen preparation area was developed to handle the increased workload of preparing specimens for irradiation in various reactors. New specimen preparation techniques were also developed that ensured high-quality specimens in minimum time. This past year, the group prepared 204 work orders, resulting in 1150 specimens prepared for TEM/AEM analysis. In addition, 213 ceramic specimens were ion milled, 70 ceramic interfaces were prepared for the Nanoindenter, and 475 photographic work orders were completed, resulting in over 13,000 pictures printed.
4. CERAMIC SCIENCE AND TECHNOLOGY

R. L. Beatty

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


The CMT Group was formed during this reporting period to enable the Ceramic Science and Technology Section to better respond to the growing interest in carbon-based materials. The majority of the group's activities were associated with the NPR-MHTGR Program. Other research and development tasks active in this reporting period included the Fusion Energy Carbon-Carbon Composites Program, the Graphite Impact Shell Materials Improvement Program, the Continuous Fiber Ceramic Composites (CFCC) Program, the commercial MHTGR Program, and coal characterization studies. These activities were supported by the ONPR; the Office of Fusion Energy; the RTG Program, Office of Special Application; the Office of Industrial Technologies; the Office of Advanced Reactors; the Pittsburgh Energy Technology Center, and the U.S. DOE. Summaries of the CMT Group's activities in this reporting period are given below.

4.1.1 Fusion Energy Carbon Materials

Research activities for fusion energy application have been in support of plasma interactive or high-heat-flux materials needs. Graphite and carbon-carbon composite materials are selected for these applications because their low atomic number minimizes radiative heat losses from the plasma. Plasma-facing materials requirements include extremely good resistance to thermal shock, erosion, and neutron damage.

Fusion energy carbon studies are focused in two major areas: experimental determination of the effects of neutron damage on candidate plasma-facing materials and the development of theoretical models for neutron-induced, crystal-structure damage in carbons. PIE of specimens from two HFIR irradiation experiments, HTFC I and II, was undertaken. These two capsules were irradiated at 600°C to peak damage levels of 1.6 and 4.7 dpa, respectively. The experiments contained a variety of carbon materials including: nuclear-grade graphite (H-451); one-, two-, and three-directional carbon-carbon composites; and a random fiber carbon-carbon composite. Specimen PIE included dimensional, thermal conductivity, electrical resistivity, and strength (brittle-ring) measurements. These data were analyzed in
terms of composite fiber type (precursor), architecture, and final heat-treatment temperature. Analysis of HTFC I data indicated that the most irradiation-stable carbon-carbon composite materials were three-directional materials manufactured from pitch precursor fibers and had been heat treated (graphitized) above 3000°C. These data were presented at the 20th Biennial Conference on Carbon, University of California at Santa Barbara, and have been written up and submitted to the Journal of Nuclear Materials.

A computer model has been developed to simulate the behavior of self-interstitials with particular attention to clustering. Owing to the layer structure of graphite, atomistic simulations were performed using a large, parallelepiped supercell containing two layers. Frenkel pairs were randomly produced. Vacancies were assumed immobile, whereas interstitials were given a certain mobility. Two point-defect sinks were considered, direct recombination of Frenkel pairs and interstitial clusters. The conditions under which interstitial clustering takes place were studied. It was found that when clustering occurs, the cluster size population gradually shifts toward the largest size clusters. The implications of the present results for irradiation growth and irradiation-induced amorphization were discussed.

4.1.2 Commercial MHTGR Program

Activities during this reporting period have been in support of the U.S. DOE's international agreements on gas-cooled reactor development with the JAERI and with Germany. Two coordinating committee meetings were held under Annex 3, Graphite, with JAERI personnel. Annex 3 activities have centered on three areas: (1) HFIR irradiation capsule HTK-7, containing specimens of U.S., Japanese, and German nuclear graphites, has been in-core throughout this reporting period; (2) specimen volume effects on the tensile strength and fracture toughness of U.S. and Japanese nuclear-grade graphites; and (3) a theoretical model of fracture in graphites has been improved and applied to the tensile fracture statistics of U.S. nuclear graphite, grade H-451.

The specimen volume effects studies were in two parts. The effect on tensile strength was examined using two grades of graphite, Great Lakes H-451 and Toyo Tanso IG-110. Further details of this study are reported under the NPR-MHTGR section of the CMT group's activities. The fracture toughness study utilized three nuclear-grade graphites (Great Lakes H-451, Stackpole 2020, and Toyo Tanso IG-110) and is reported in the NPR-MHTGR section of the CMT group's activities. Data obtained from these two studies were reported at an International Atomic Energy Agency (IAEA) Specialists Meeting on the "Status of Graphite Development for Gas-Cooled Reactors" held at JAERI, Tokai-mura, Japan, September 9-12, 1991. Two other topics were reported at the IAEA Specialists Meeting. The first reported spatial variations of tensile strength within a single billet of grade H-451 graphite. The second reported the improved graphite fracture model and its applicability to H-451 tensile fracture statistics. The fracture model combines a microstructural approach to fracture with a fracture mechanics-based failure criteria. Model inputs are filler particle size, particle Kf, density, pore-size distribution, number of pores, and stressed volume. The model has been shown to predict the tensile failure statistics of grade H-451 graphite. Moreover, the model qualitatively predicts the effects of texture-related parameters on the tensile strength of H-451 graphites. One subprogram management meeting was held under the U.S. DOE-Germany umbrella agreement. Activities under this agreement are related to the irradiation of German graphites in HFIR capsule HTK-7.
4.1.3 New Production Reactor MHTGR

A wide range of technology development activities were ongoing through this reporting period in support of the NPR-MHTGR Program. These included: thermal, mechanical, fracture, and fatigue data base development activities for grade H-451 graphite; air oxidation studies of grade H-451 graphite; examination of candidate cokes for the production of H-451, including irradiation of pilot-scale developmental H-451 graphites; an alternate graphite vendor program; and a carbon-carbon composite control rod materials development program.

4.1.3.1 Thermal properties

Thermal properties work in this reporting period was largely associated with the preparation of: (1) an experimental plan, (2) acquisition and commissioning of new test equipment, and (3) preparation of grade H-451 graphite thermophysical property specimens. A comprehensive test plan has been written describing the approach, methods, techniques, and apparatus to be used in generating the NPR-MHTGR graphite thermophysical properties data base. The plan gives details of the test matrix, materials, cutting diagrams, and specimen configurations to be used. Several new pieces of equipment have been brought into service. A Xenon flash thermal diffusivity system has been developed to measure room-temperature thermal diffusivity and conductivity. Moreover, a laser thermal-pulse system has been installed to allow elevated-temperature (up to 2000°C) measurement of thermal diffusivity, conductivity, and specific heat. A high-temperature dilatometer has been acquired to allow determination of coefficient of thermal expansion (CTE) at temperatures up to 1600°C. In accordance with our approved test plans, a graphite billet was cut up and 768 specimens machined from a 2-in.-thick center slab for evaluation of the spatial variation of thermal properties.

4.1.3.2 Mechanical properties

Employing a recently obtained state-of-the-art test system, room-temperature tensile tests were conducted on 368 specimens taken throughout an entire billet of class 11, grade H-451 graphite. Concurrent studies were made on spatial variations in strength within the billet and on the effects of stress volume (specimen size) on the tensile strength. The tensile strength of H-451 exhibited a dependence on location within the billet. Generally, the strength near the edge of the billet was greater than at the central area of the billet. Further, the strength was found to progressively increase from one end of the billet to the other. It appeared, however, that the profile (magnitude and shape) of spatial variations within the billet was affected by specimen size. Generally, the strength increased with increasing specimen size. However, the popular Weibull statistical theory predicted behavior to the contrary. A new graphite fracture model incorporating microstructural elements and the fracture toughness of the material, as well as specimen size parameters, showed good agreement with the experimental data from the billet of H-451 and shows promise for characterizing the interaction of volume effects and microstructural variations through the bulk of the material.

4.1.3.3 Fracture mechanics

The plane-strain, chevron-notched fracture toughness test method (ASTM E1304-89) has been employed to measure fracture toughness of several grades of nuclear graphites including:
Great Lakes Carbon grade H-451, Stackpole grade 2020, and Toyo Tanso grade IG110. Specimen size effects were investigated using the chevron-notched, short-rod (CNSR) specimen geometry. Fracture toughness was found to increase with increasing specimen size. This behavior was attributed to a rising "R-curve" behavior for these graphites. The small volume requirements of the CNSR specimen allowed for a localized measure of fracture toughness, permitting an evaluation of the effects of spatial location and orientation within a fuel element graphite billet. The CNSR specimen is compatible with the specimen size limitations of HFIR irradiation capsules. Fracture mechanics data are needed for the design of the MHTGR.

4.1.3.4 Fatigue behavior

A specimen geometry and test procedure was developed for fully reversed, load-controlled fatigue testing of nuclear graphites. The test method employed: a rigid load train, a means for achieving precise alignment, and a constant-pressure cylindrical collet gripping system. The optimum specimen geometry was determined to be a monolithic axial specimen with a smooth, cylindrical gage section and cylindrical grip ends. The cost and time associated with fabricating this test specimen was less than 10% of that required for a previously used composite configuration. The simple specimen geometry allows for efficient use of test facilities, permitting a large number of fatigue tests to be performed as required to establish a statistically sound data base for the design of the MHTGR.

4.1.3.5 Oxidation studies

The study of graphite air and water-vapor oxidation behavior has been ongoing throughout the year. The High-Pressure Test Loop (HPTL) has been in commission and will allow oxidation experiments at the high temperatures and pressures typical of reactor operation (1000°C and 6.9 MPa). Thermogravimetric analysis has yielded quantitative data on the kinetics of the graphite-air reaction. Variation of corrosion rate occurs as a result of continuous, morphological modification of porous structures with differing fractal dimensions. Diffusion limitations are noted to predominate in the higher temperature regimes. Reaction mechanisms are strongly dependent upon the grain structure, porosity, and chemical purity of the specific graphites. An air oxidation study was performed to provide H-451 kinetics data in support of the Los Alamos National Laboratory/Idaho National Engineering Laboratory (LANL/INEL) air oxidation integral experiment.

4.1.3.6 Coke source examination

An investigation of the influence of precursor isotropic petroleum coke on graphite properties and irradiation response continued in this reporting period. Working in collaboration with a manufacturer, experimental graphites were made, each with an isotropic coke from a different source. The cokes and graphites were subjected to a series of analytical measurements to assess the influence of coke structure on properties. Two HFIR irradiation capsules containing specimens of the experimental graphites were constructed, and irradiation commenced during this reporting period. The first of these experiments, capsule HTN1-900°C, 9.75 x 10^23 n/m^2 [E > 50 keV], completed irradiation, and the specimens were subjected to PIE. PIE data indicated that the precursor coke influenced the irradiation performance of the graphites. One of the experimental graphites manufactured from a candidate isotropic coke exhibited
irradiation-induced dimension changes, which closely match those of the production-grade H-451 graphite. These results suggest that future supplies of H-451 can be assured through appropriate selection of the precursor coke structure and properties.

4.1.3.7 Alternate vendors program

At the request of DOE ONPR, a program was initiated in collaboration with Combustion Engineering General Atomics (CEGA) to solicit alternate vendors of nuclear-grade graphite for the NPR-MHTGR. During this reporting period, four graphite vendors (other than Great Lakes) were visited and appraised of the NPR Program’s needs. The vendors visited were Stackpole Carbon Company; POCO Graphite, Inc.; The Carbon/Graphite Group, Inc.; and UCAR Carbon Company, Inc. These companies were invited to supply material to ORNL for inclusion in an irradiation program to determine the acceptability of their product for NPR-MHTGR needs. Three companies, UCAR, Carbon/Graphite Group, and POCO, supplied experimental materials for evaluation.

4.1.4 Improved Graphite Impact Shell

The general-purpose heat source provides power for space missions by transmitting the heat of 239Pu decay to thermoelectric elements. Because of the possibility of an aborted mission, the heat source must be designed and constructed to survive both reentry, heat, and earth impact. Cylindrical graphite impact shells (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 carbon-carbon composite and is susceptible to longitudinal fracture during impact. Cylindrical architecture carbon-carbon composites have been evaluated as a potential improvement to GIS impact performance. Characterization included measurement of physical, thermophysical, and mechanical properties. Mechanical properties tests were designed to simulate (as well as possible) the loading experienced during impact.

4.1.5 Chemistry and Structure of Coals

Work described last year on the chemistry and structure of coals was continued using diffuse reflectance infrared spectroscopy (DRIS) to evaluate the bonding and structural elements. Three samples of interest to the Clean Coal Program were used to show the merit of the DRIS technique for the evaluation of the rank and composition of coals. The band intensities were good measurements of the content of minerals, moisture, oxygen, hydrogen, and sulfur in coals ranging in rank from lignites to anthracite. DRIS also identifies relative amounts of organic species for hydrogen (aliphatic, aromatic, phenolic, alcoholic, etc.) and oxygen (aldehydic, ketonic, acidic, anhydrotic, carbonic, etc.). DRIS is equally well suited for evaluating the nature of the mineral components (kaolinic, illitic, carbonate, silica, etc.). Analyses of the physical absorption of nitrogen gas on coal surfaces were performed utilizing a computer-controlled volumetric adsorption apparatus. TEM has shown that much of the sulfur exists as very small, frambooidal inclusions in the organic matrices. Both well-crystallized and polycrystalline pyrite are noted to exist within the frambooids. Progress was made in the study of the oxidation of pyrite minerals (which are found in bituminous coals) using TEM and XPS.
4.1.6 Continuous Fiber Ceramic Composites

A new activity this reporting period was directed toward developing thermal models for CFCC materials. To date our activities have centered on planning an experimental program. Discussions have taken place with a supplier, FMI, regarding the manufacture of test materials. Additional materials have been acquired from sources within the M&C Division.


Group research and development activities include ceramic forming by gelcasting, microwave processing, composite development, high-temperature corrosion, materials characterization, and sensor development. These activities are supported by the CTAHE Project, Office of Transportation Systems; the AIC and Advanced Industrial Heat Exchanger Programs, Office of Industrial Technologies; the Fossil Energy Advanced Research and Technology Development (AR&TD) Materials Program, Office of Technical Coordination; the RTG Program, Office of Special Applications; the ONPR; and the Office of Energy Research, U.S. DOE.

Ceramic gelcasting is being developed as a net-shape-forming method that may confer both manufacturing advantages and improved reliability of products compared with forming by slip casting or injection molding. The gelcasting process disperses ceramic powder in an aqueous solution of monomer, and "gelling" is accomplished by thermal polymerization. As reported last year, initial studies were very successfully conducted using alumina powder and acrylamide monomer.

The gelcasting process was then extended to silicon nitride, and several powders and compositions were successfully processed. A Cooperative Research and Development Agreement (CRADA) was initiated with Garrett Ceramic Components (GCC) Division of Allied Signal Aerospace Company to evaluate gelcasting of T-25 turbine rotors using Garrett formulation GN-10. This was completed with gelcasting of nine rotors and companion flat plates to be used for property testing. Parts were presintered at ORNL and glass-encapsulation HIPed at GCC. Test specimens from the plates will be evaluated at ORNL.

Fabrication of numerous ceramic materials by gelcasting has shown the process to be very versatile and attractive for complex shapes and potentially for large parts. However, the acrylamide monomer used for most of the development work has a toxicity level that renders it undesirable or unacceptable for scaled-up manufacturing applications. Thus, an important part of the work has been a search for alternative monomers to replace the neurotoxic acrylamide. Two monomers produced good gels, but their high charge interference limits the dispersion of solids in their solutions. Therefore, the search continued, and two new systems were identified. These two systems need further work but show good promise for adequately replacing acrylamide in the aqueous gelcasting process.
The gelcasting process has also been a focus of technology transfer activities. Over 300 inquiries for information on the process have been received. Seventy-five companies have signed Proprietary Information Agreements to obtain additional information, and representatives from over 40 companies attended five gelcasting workshops held at ORNL. To date, 15 companies have expressed serious interest in a joint effort with ORNL to evaluate gelcasting for their specific applications and material systems. Gelcasting experiments with materials provided by six companies are under way at ORNL.

Microwave processing development was continued with several ceramic materials. In the AIC Materials Program, zirconia-toughened alumina (ZTA) with excellent mechanical properties was made by microwave sintering. Average strengths exceeding 700 MPa and fracture toughnesses, $K_{\text{ic}}$, in the range of 6 to 6.5 MPa-m$^{1/2}$ were obtained. It was also demonstrated that ZTA could be fired in a 2.45-GHz microwave furnace without the need for external heating by incorporating a lossy additive (third phase) in the starting raw materials. Using this method, samples weighing up to 200 g were successfully fired at 2.45 GHz without cracking; heating rates up to 10°C/min were demonstrated.

In research for the Fossil Energy Materials Program, we found that no "microwave effect" occurs in either the sintering or long-term annealing of LaCrO$_3$-based ceramics (i.e., there is no enhancement or acceleration of processing.). LaCrO$_3$ is an electronic conductor and, therefore, couples to the microwaves through its electrons rather than through its point defects and lattice vibrations. Systems that have shown a "microwave effect" in the past (alumina and zirconia) are either insulators or ionic conductors. Thus, it is the electronic conductivity that is thought to be responsible for the lack of a "microwave effect" in this system.

In the CTAHE Program, we studied microwave sintering of silicon nitride. Microwave heating of silicon nitride-based materials occurs predominantly via power absorption by the sintering additives and/or the intergranular phases and, therefore, enhances diffusional processes in those phases. At high levels of additives, densification occurs predominantly by particle rearrangement, and, hence, sintered densities are similar but not equal for both conventional and microwave heating since diffusion is not a factor. A modest contribution from enhanced diffusion during the α-to-β Si$_3$N$_4$ transformation in the microwave sintering improves their densities slightly. Thus, to enhance the microwave effect during sintering, one would choose to use lower additive contents where solution-diffusion-reprecipitation is a more dominant densification process. However, at low additive contents, problems arise because heating and densification inhomogeneities lead to specimen cracking. By increasing the microwave frequency from 2.45 to 28 GHz, in the current study, improved heating uniformity and sintering behavior is obtained.

Three CRADAs have been established to investigate the microwave processing of silicon nitride. Two are with Norton Company, and the third is with GCC. Norton Company will investigate the annealing of dense silicon nitride materials with low additive contents (< 5%) and also the reaction-bonding of silicon compacts to form silicon nitride. GCC will examine microwave annealing of dense silicon nitride materials with high additive levels (> 5%).
Two additional CRADAs on microwave work were initiated under Energy Research Programs. One is with AVX Tantalum Corporation to evaluate microwave sintering of tantalum capacitors, and the other is with Microwave Laboratories, Incorporated, to develop a variable frequency furnace.

A new project was started in May 1991 to examine intermetallic-bonded, non-oxide ceramic composites, with the initial effort on Ni$_3$Al alloys as the binder phase. The fracture toughness of WC-Ni$_3$Al and TiC-Ni$_3$Al composites agrees reasonably well with the relationships derived for the WC-Co- and TiC-Ni-bonded materials from the literature. If these relationships also describe the Ni$_3$Al-bonded systems, the grain size of the ceramic phase needs to be on the order of 5 μm with a binder phase content of 10% to achieve toughness of > 20 MPa√m.

Ceramic matrix composite development was redirected. Initially, this work involved development and characterization of SiC whisker-reinforced ceramic composites for improved mechanical performance. In addition, studies of whisker-growth processes were initiated to improve the mechanical properties of SiC whiskers by reducing their flaw sizes and, thereby, improving the mechanical properties of the composites. Currently, in situ acicular grain growth is being investigated to improve fracture toughness of silicon nitride materials.

Previous work identified optimized SiC whiskers that were calculated to give high toughness to silicon nitride matrix composites. Thus, studies were initiated to determine process parameters that would improve SiC whiskers. During the same time frame, however, concerns about the cost of the whiskers, the processing difficulties encountered during fabrication of whisker composites, and the health and safety issues associated with the acicular nature of the whiskers caused the emphasis of the work to shift from whisker-reinforced composites to in situ grain growth to achieve high-toughness silicon nitride materials. The work that was done to determine the process parameters affecting SiC whisker growth investigated the effects of raw materials, catalyst type, atmosphere, and heating rate.

In situ toughening of silicon nitride by microstructure development is now being investigated. Gas-pressure sintering (GPS) is one technique used to grow elongated grain structures and obtain high-toughness silicon nitrides with refractory grain-boundary phases. Initial samples sintered at 1900°C and 2 MPa nitrogen pressure showed toughness values comparable to those obtained from hot-pressed materials. However, the values are low in comparison to other published work on high-toughness silicon nitrides fabricated by GPS. Further examination of the available literature on high-toughness materials fabricated by GPS showed that all of those materials were processed at temperatures ≥ 1950°C to grow the large grains. Future work will explore the use of these higher fabrication temperatures.

Materials technology support was provided for an Industrial Technologies Program to assist contractors in developing high-temperature heat exchangers. Materials exposed to simulated steam-reformer environments were characterized to support selection of candidate materials for further testing at ORNL. This work was a cooperative effort with the Corrosion Science and Technology Group.

Characterization work was conducted on a ceramic material for the NPR-MHTGR. The commercially available material, Coors AD85 alumina, is a candidate for use in large blocks
as a core support insulator. Detailed mechanical and physical property measurements were made, which will be part of a design data base.

Fabrication work continued on carbon-bonded carbon-fiber (CBCF) thermal insulators for the RTG Program. Schedules were met for production and documentation of 130 flight-quantity sets of CBCF parts. These parts and 80 additional sets scheduled for production next year are for use in NASA’s planned Comet Rendezvous Asteroid Flyby (CRAF) and Cassini missions. Parts produced earlier are currently flying in the Galileo and Ulysses crafts.

Development work was completed on a "Rapid Fuel Analyzer," which can detect the presence of alcohol in motor fuels and classify gasoline by octane rating. Fuels have distinctive signatures that are learned by a neural network evaluating signals from 10 metal oxide sensors. The resistance of the metal oxide decreases in the presence of organic vapors. This work, which received an R&D-100 Award, was done in cooperation with the Instrumentation and Controls Division.

4.3 CERAMIC SURFACE SYSTEMS — T. M. Besmann, P. J. Blau, R. A. Lowden, J. C. McLaughlin, D. P. Stinton, and C. S. Yust

In a program supported by the USAF, Office of Scientific Research, basic studies of nucleation and growth in CVD were conducted. The first in situ observation of nucleation and growth using angular-resolved laser light scattering was accomplished using SiC growing on SiC. Electron microscopy using lattice imaging has revealed specific independent nucleation sites where nano-scale features have grown epitaxially. This observation is the first in which polycrystalline growth was observed to be controlled by the crystallographic orientation of the substrate.

Development of coatings to protect SiC or Si₃N₄ turbine engine components from sodium and steam corrosion continued this period. After an extensive search, a limited number of stable materials (Al₂TiO₅, ZrTiO₄, HfTiO₄, Ta₂O₅, and 3Al₂O₃·2SiO₂) with very low CTEs were identified. Corrosion testing of these materials in combustion atmospheres revealed considerable improvement over SiC or Si₃N₄. Further testing is being performed to determine the most corrosion-resistant materials for this application. After the best materials have been identified, CVD processes will be developed for application of coatings.

Ceramic coatings were examined as the optical baffle surfaces for infrared sighting systems in advanced missile applications. Specifically, boron carbide coatings with excellent non-reflective properties were deposited on graphite substrates utilizing CVD techniques. Coating morphology and composition have been tailored to perform in wavelength regions from 0.6 to 10.6 µm. Deposition parameters such as temperature, pressure, gas composition, and reactant flux were examined to produce a variety of coating surface structures and compositions. The deposition conditions were optimized to produce coatings features that interact with radiation of a specific wavelength. It has been demonstrated that the shape, size, and composition of the surface features can be readily modified by altering deposition conditions. Coatings with bidirectional reflectance distribution functions (BRDF) approaching 10⁴ with a uniform response over a range of angles and specular reflectances of < 0.5% have been produced, rivaling the performance of the best optical coatings (Martin Black).
The coatings have been subjected to 200-G shake tests and exposed to high-energy bursts with little or no change in properties and no evolution of particles or other debris.

The USAF Wright Laboratories has sponsored an effort to model chemical vapor infiltration (CVI) for the fabrication of continuous fiber-reinforced ceramic composites. A 3-D process model using a steady-state finite volume technique has been developed together with Georgia Tech Research Institute. The model includes mass and heat transport effects and chemical kinetic relationships. The model has been verified using partial infiltration experiments in which SiC was infiltrated into Nicalon fiber preforms. The model shows good agreement with the observed density gradients.

As part of the modeling effort, it was necessary to develop a kinetic relationship for the deposition of SiC from methyltrichlorosilane in hydrogen. This was a complex problem due to the poisoning effect of reaction product HCl. Deposition experiments over a range of temperature-flow-concentration values allowed derivation of a useful relationship based on competitive absorption.

CVI of tubular preforms of different fiber architectures continued this period. Tubular preforms containing ~15 vol % Nicalon fibers prepared by 3-D braiding failed to infiltrate properly because of large gaps between fiber bundles that extended nearly to the inner diameter of the preform. Filling these large gaps with molded, chopped fibers produced a hybrid preform that could be effectively infiltrated. Tubular preforms prepared by filament winding (47 vol % fiber) were infiltrated to a density of ~85% of theoretical. Composites of both types are being characterized at Argonne National Laboratory by non-destructive evaluation and at Virginia Polytechnic Institute by mechanical property testing. Braided preforms containing higher fiber contents (33 to 39 vol %) have been received and are currently being infiltrated.

Fabrication of full-scale, fiber-reinforced candle filters (60 mm diam and 1.5 m long) continues in a collaborative effort with the 3M Company. Candle filters have been fabricated from Nextel (alumina-boria-silica) and Nicalon (Si-C-O) fiber preforms and infiltrated with an SiC matrix. Chopped or discontinuous fiber preforms were found to have an appropriate permeability but were quite brittle. Woven filters exhibited appropriate mechanical properties; however, the permeability was too high. Therefore, current filters are fabricated from a combination of continuous fibers for strength and chopped fibers for permeability. Several full-scale filters have been fabricated and supplied to Westinghouse Science and Technology Center for testing and evaluation.

Thin coatings deposited on ceramic fibers prior to densification have been used to control fiber-matrix interfacial stresses in Nicalon/SiC composites fabricated employing the forced-flow, thermal gradient CVI technique. Improvements in strength and toughness at room and elevated temperatures have been observed in composites fabricated from coated fibers. A thin, graphitic carbon layer has been shown to increase product reproducibility and improve mechanical properties. The low-modulus interlayer appears to accommodate a portion of the residual clamping stresses caused by the thermal expansion mismatch of the constituents. This is reflected in that the thickness of the carbon interlayer strongly influences interfacial stresses and the mechanical behavior of composite materials. The effects of the coating thickness on matrix fracture stress and ultimate strength of Nicalon fiber-SiC matrix composites continued to be of interest as well as the influence of the fiber-matrix interface on
interlaminar and impact properties of composites. Interfacial shear stresses were measured using both a standard hardness indenter and the Nanoindenter, and good agreement between the techniques was shown. Correlations between interfacial forces and composite behavior continue to be examined.

Although carbon has proven to be an effective interlayer in Nicalon/SiC and other composite systems, carbon lacks resistance to oxidation at elevated temperatures. Thus, the elevated-temperature stability of Nicalon/SiC composites with a graphitic carbon interface layer at elevated temperatures in oxidizing and simulated fossil fuel environments was investigated. Composite specimens with and without an external SiC surface coating were oxidized in air and exposed to a variety of combustion environments at a temperature of 1273 K. A burner rig furnace was constructed for simulating corrosive fossil fuel environments containing water vapor, sulfur, and sodium. Post-exposure, room-temperature flexure tests were performed to measure the strengths of the samples, and optical and electron microscopy were employed to analyze corrosion, glass layer formation, and compositional changes at the surface of the specimens and at the fiber-matrix interface. The mechanical properties of uncoated composite samples were degraded after short periods of oxidation in air.

ORNL is responsible for the "Tribology by Design" task area of the Advanced Transportation Materials Tribology Program. Emphasis at ORNL is further divided into two subtasks: tribosimulation and tribomaterials evaluation. The former subtask addresses the development of effective test methods by which advanced, emerging materials can be effectively screened for engine-related applications. To establish the basis for simulations, FY 1991 studies have involved determining the effects of contact size (scale effects) and geometry on kinetic friction coefficients. The latter subtask has involved the development of graphical methodology to depict time-dependent wear behavior for SiC whisker-reinforced silicon nitride composites. A subcontract to Georgia Institute of Technology led to the development of thermal-mechanical surface damage prediction maps for unlubricated ceramics, and an ongoing follow-on effort is extending the work to lubricated ceramics. This subcontract will be completed in FY 1992 when a series of wear maps will be published for ceramics of current technological interest for engine applications.

ORNL has a task to develop a multi-year project plan for a new thrust in Cost-Effective Ceramic Machining. This project is part of the larger Ceramic Technology Project, sponsored by DOE's Office of Transportation Materials. The new effort will involve identifying major cost drivers in machining ceramic parts and developing R&D subcontracts and in-house efforts to attack those issues. Issues such as economic modeling of machining, inspection of surface damage, standard machinability tests, and cooperative programs with other agencies will be addressed.


Advanced ceramic materials are important to the energy and industrial sectors due to their inherent chemical stability, high hardness, wear resistance, electrical properties, stiffness, and elevated-temperature capability. The useful lifetime of these ceramic components in service, however, depends on the ability of the ceramic to resist impact, thermal shock, and creep
damage. Efforts to increase the fracture toughness and damage resistance of ceramics are, therefore, necessary to enhance the reliability of ceramic components.

Our research concentrates on the design of toughened ceramics that are intended for use in a variety of environments. Our efforts are interdisciplinary in nature and combine both theoretical and experimental approaches. By identifying the mechanisms that control the strength, toughness, and elevated-temperature mechanical behavior of these materials, a basis for the microstructural, interfacial, and compositional design of these advanced ceramic materials can be established. These activities are supported by the Division of Materials Sciences, the CTAHE Project, and the Office of Industrial Technologies at the U.S. DOE.

4.4.1 Toughening Processes in Ceramics

The fracture toughness of ceramics and ceramic composites can be improved by the incorporation of reinforcing phases that generate a bridging zone when cracking occurs. Our studies involve theoretical modelling of crack-bridging processes and experimental verification of these processes via a combination of microstructural observations and mechanical property evaluation. Based on micro-/macro-mechanical modelling, an analytical description has been generated, which describes the criteria necessary to obtain optimum toughening in fiber-reinforced ceramic composites. Two results emerge from these studies. First, to achieve interfacial debonding, the ratio of the fiber strength to the interfacial shear strength must exceed a critical value. Second, to achieve a frictional interface after interfacial debonding, the ratio of the interfacial residual clamping stress to the interfacial shear strength must also exceed a critical value. The above findings can thus serve as a basis for material design. Microstructural observations of crack interactions with ceramic composite microstructures using carefully controlled low-voltage imaging have provided concrete evidence of various crack-bridging mechanisms. High-resolution electron microscopy (HREM) of SiC whisker-reinforced alumina composites has allowed us to examine the physical nature of the whisker/matrix interface in detail. Combined, these studies have provided us with a more thorough understanding of the role of interfacial properties on the toughness of reinforced ceramic composites. The studies of crack-bridging phenomena in toughened ceramics resulted in a 1991 MMES Technical Achievement Award to the group members involved in this effort.

An alternative method to reinforcing ceramic matrices with elastic crack-bridging reinforcements is to use ductile-phase reinforcement to increase the fracture toughness of ceramic materials. We are currently examining the role of microstructural parameters and interfacial characteristics on the toughening mechanisms and the resultant mechanical properties of ductile-phase-reinforced, oxide-based ceramics.

Studies of transformation toughening in alumina composites containing ceria-stabilized zirconia have shown that the martensitic transformation in zirconia is a function not only of the zirconia grain size, but also of the zirconia content and spatial distribution. This has been shown to result from the role of local environment on the internal residual tensile stresses in the untransformed zirconia phase.

All of the above research areas show that improved fracture toughness can be obtained by incorporating a variety of reinforcing or transforming phases into inherently brittle ceramic
matrices. A thorough understanding of the operative toughening mechanisms allows for a prescriptive approach to microstructural design in order to obtain optimum toughening performance.

4.4.2 Creep Resistance of Ceramic Composites

Creep studies of in situ reinforced silicon nitride (Si₃N₄) ceramics, which contain highly elongated grains, provide insight into the role of elongated grains during high-temperature creep deformation. Our creep studies indicate that in situ reinforced Si₃N₄ ceramics exhibit high-temperature creep resistance comparable to SiC whisker-reinforced alumina and silicon nitride-based composites. The excellent creep resistance arises from the interlocking elongated grain structure, which inhibits grain-boundary sliding and grain rotation during high-temperature deformation. The creep resistance of in situ reinforced Si₃N₄ ceramics is substantially diminished by a decrease in grain size. This is due to the fact that the finer elongated grains are less effective at bridging the cracks developed and at inhibiting crack propagation during creep deformation. The addition of rare-earth oxides and/or alumina promotes densification as well as growth of elongated grains through the formation of a glassy liquid phase. It is expected that the presence of intergranular amorphous phases plays a key role in determining the creep properties at elevated temperatures. At high temperatures, the intergranular amorphous phases become viscous and thus promote grain-boundary sliding processes, which then result in higher creep rates. In addition, the glassy liquid phase provides a fast diffusion path for oxidation reactions, thus leading to further degradation of creep resistance. To ensure long-term stability for high-temperature applications, sintering aids that result in more refractory and stable intergranular phases must be employed.

4.4.3 Ceramic Processing

Theoretical and experimental studies on the processing of ceramics, including the colloidal and surface chemical aspects of powder processing, allow us to control the compositional uniformity and the microstructural evolution of powder compacts. The relationship between presintering processing variables, such as slurry pH, powder particle sizes, and volume fraction of components, and the resultant microstructure must be fully understood. By identifying processing conditions in Al₂O₃/ZrO₂ (12 mol % CeO₂) slurries that avoid agglomeration and differential settling of oxide particles, powder compacts with uniform spatial distribution of both phases and high green density can be achieved. By tailoring the initial powder particle size distributions and then selecting appropriate sintering conditions, the grain sizes of both the alumina and zirconia can be systematically varied. These studies emphasize the need to regulate the starting powder, the powder processing, and the sintering conditions to optimize the resultant zirconia grain size and, thus, the fracture toughness in zirconia-based, transformation-toughened ceramics.

Similar considerations are being used (1) to optimize the slurry characteristics of gelcast ceramics and (2) to control the green state microstructure of ceramic pieces prior to microwave sintering. Gelcasting of ceramics requires a highly concentrated, pourable slurry, which allows the near-net-shape casting of components with high green density and strength. Our studies have identified the optimum processing conditions for various ceramics (SiC, ZrO₂, Al₂O₃/ZrO₂ composites) as well as contributed to a basic understanding of the gelcasting process. Microwave sintering shows promise as a means to densify green pieces
at much lower temperatures than those used during conventional sintering. Our studies have shown that by controlling the processing conditions, we can tailor the microstructure such that microwave sintering is most effective.

4.5 BUILDING MATERIALS — T. G. Kollie, R. S. Graves, G. M. Luttka, F. J. Weaver, and D. W. Yarbrough

The Building Materials Group conducts research and monitors subcontracts to other installations primarily for the DOE's Assistant Secretary for Conservation 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. Other research is performed for the Environmental Protection Agency (EPA), the Department of Defense (DoD), and other DOE programs and facilities in areas related to thermal insulation and properties of materials.

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. The materials program objectives are to: (1) demonstrate the validity of alternative materials for chlorofluorocarbon (CFC)-foamed insulations (both hydrochlorofluorocarbon (HCFC)-foamed insulations and high-resistance powder-filled evacuated panels (PEPs)); (2) improve test procedures to determine properties of existing and advanced building materials; (3) conduct technology transfer to the U.S. building industry, and (4) recommend measures that conserve energy.

A CRADA (ORNL 90-0028) has been established with the Polysiocyanurate Industry Manufacturers Association (PIMA), the Society of the Plastics Industry (SPI), EPA, and associated trade organizations and industrial members to evaluate the thermal performance of rigid foam blown with CFC-11 (control), HCFC-123, HCFC-141b, and two blends of HCFC-123/HCFC-141b. Industry-produced boards are being evaluated by field tests in the ORNL Roof Thermal Research Apparatus (RTRA) and by laboratory thermal resistance (R-value) tests of thin-board specimens. After 450-d exposure in the RTRA, the R-values of the foams decreased by about 20%. The R-value change is accelerated in the thin boards, and this change in R produced two linear regions when the conductivity (1/R) is plotted versus time/thickness, with the slopes of these two regions proportional to the effective diffusion coefficients for air components and the blowing agents. The R-values after 10- and 20-year aging were predicted for the unblended and blended blowing agents, respectively. Models for the aging phenomena are being derived in conjunction with the National Research Council of Canada and the Massachusetts Institute of Technology (MIT); a homogenous model yielded reasonable agreement with the experimental results, based on experimental measurements of diffusion parameters determined at MIT. Plans for the possible extension and broadening of this CRADA were presented to the industrial partners, who are currently evaluating them.

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 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. A CRADA (ORNL 91-0042) with the Appliance Research Consortium (ARC) was signed to develop a test procedure to determine the lifetime of PEPs in dry air. Four new powders have been identified with promising thermal performance; patent disclosures have been filed with the DOE on these powders. We are developing a new gage to measure the internal pressure of PEPs nondestructively; early tests yielded results in reasonable agreement with the hand-held gage developed last year. Construction on a facility to fabricate PEPs was begun. (The work on powders and barriers has been funded by the EPA and DOE.)

We are developing a procedure to accurately measure the resistivity of high-R insulation such as PEPs. We are employing our Heat Flow Meter Apparatus (HFMA), which is a commercially available device. 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. To do this, we have modified the HEATING-7 heat transfer code to run on an IBM-compatible 386 computer. A user-friendly interface for this code has been written to facilitate use by the insulation industry. In addition, we have modified the HFMA by inserting a 5 by 6 array of heat flux meters to measure the 3-D heat flow in the HFMA; these data are required by the HEATING-7 code. This work will be complete in FY 1992.

We studied the effect of varying the solar reflectances of roofs from 0.1 to 0.85 to demonstrate the changes on heating and cooling loads of applying Radiation Control Coatings (RCCs) to roofs. The computer simulation code "BLAST" was used to show that increasing the solar reflectance of a roof in Minneapolis, Minnesota, decreased the cooling loads about the same as for Las Vegas, Nevada; however, the sum of the annual loads was insensitive to the range of solar reflectances, indicating that in cold climates, the savings in cooling loads with high solar reflectances would not necessarily result in increased annual heating plus cooling loads. The maximum cooling demands were decreased, but the maximum heating demands were not affected since they occur during the night. Accelerated solar exposures of five RCCs showed only about a 3% decrease in reflectance when the coatings were protected from dust/dirt accumulation.

Besides the above mentioned CRADAs, we performed technology transfer in several ways. For example, we measured the thermal conductivity of gas-filled panels for Lawrence Berkeley Laboratory. These measurements demonstrated that their panel design achieved the conductivity of the gas without conduction; in-leakage of air was a problem with their early design. We performed an interlaboratory comparison with Saint Gobain on a fiberglass blanket material they provided; the agreement of the results was within the imprecision of the equipment employed. An interlaboratory comparison of four HFMA's on planed polyisocyanurate boards foamed with CFC-11 showed a two-standard deviation imprecision of 2.2%. 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. For example, we recently helped write a test procedure for thin slicing for accelerated aging of cellular foam insulation for ASTM Subcommittee C 16.30; we participated in an interlaboratory comparison on procedure ASTM C 167, "Standard Test Methods for Thickness and Density of Blanket or Batt Thermal Insulation." One member of our group, R. S. Graves, served as co-chairman of the "Second Symposium on Insulation Materials: Testing and Applications" sponsored by the ASTM.
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 for two ORNL reactor programs and three international collaborative programs and through the coordinated activities of five technical groups within the M&C Division with shared expertise and resources in materials characterization, modeling, irradiation testing, unique remote equipment development, and facility operation.

Program guidance and coordination are provided for two separate DOE-sponsored reactor programs by the section. For the Nuclear Energy (NE) MHTGR Program, this includes the multidivision activities of the Fuel Materials Development and Fission Product Behavior Subtasks and the management of three international programs: the Fuels, Fission Products, and Graphite Subprogram within the U.S./Germany Umbrella Agreement on High-Temperature Reactor (HTR) Development; the U.S. DOE/ JAERI Collaborative Program for Coated Particle Fuel Performance Testing; and the U.S. DOE/Commissariat A L'Énergie Atomique (CEA) Collaborative Program on the Corrosion, Migration et Distribution Irradiation Experiment (COMEDIE) Loop Fission Product Behavior Tests. In the ANS Program, guidance is provided for the Fuel Materials Development Subtask.

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); 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 fuels materials (Fuel Materials Testing Group); characterization/evaluation of fuel and fission product behavior under off-normal conditions (High-Temperature Fuel Behavior 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 NE-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.

5.1 IRRADIATED FUELS EXAMINATION LABORATORY — C. E. DeVore

The Irradiated Fuels Examination Laboratory (IFEL) is a major hot cell facility located in Building 3525. Operation of the IFEL was assumed by the M&C Division from the Chemical
Technology Division on October 1, 1990. The historical name of the facility is the High Radiation Level Examination Laboratory (HRLEL), which will eventually be phased out. The purpose of the facility is to handle irradiation experiments or irradiated materials for examination, testing, evaluation, or processing. Operation of the IFEL must be in a safe and efficient manner through compliance with all safety standards, orders, and regulations. The facility must be maintained and updated with both documentation and equipment. Much of the in-cell equipment used for handling experimental work is nearly 30 years old and has reached its expected life. Equipment failures requiring maintenance have become more difficult to perform because of the highly contaminated condition from years of service. Maintenance can be too costly in terms of personnel exposure. New decontamination techniques are being sought that will allow equipment maintenance with lower exposure until modern replacements can be installed. Facility safety documentation is being updated to reflect current operations and hazards. Old procedures are being reviewed for revision to meet today's rigor of documentation. An alternative method of disposing of low-level liquid waste (LL LW) through a tanker truck is being developed to keep the facility operational when a federal facilities agreement (FFA) is implemented between the State of Tennessee and DOE. The FFA will require all leaking tanks of LLLW to be removed from service.

During the past year, immediately after assuming facility operational responsibility, operational safety requirements (OSR) compliance became a Laboratory-wide issue with DOE. The OSR for the IFEL was outdated and did not reflect the current status of operations. Variances were requested and approved until a revised OSR was written and approved. Tiger Team inspections began in October 1990 with three facility tours. Numerous audits and tours have been provided including the DOE Office of Nuclear Safety, Satellite Accumulation Inspections, Radioactive Operations Committee Review, Transportation Safety Committee Review, DOE Nuclear Materials Audit, and an OSR compliance audit.

In addition to the oversight activities, three experimental capsules were disassembled and their components sent to other ORNL facilities, TEM specimens were prepared, disassembly equipment was installed in the cells, and areas were decontaminated. Shipping services were coordinated for off-site facilities to other ORNL facilities.

5.2 FUEL MATERIALS EVALUATION — N. H. Packan

Beginning in FY 1992, the Fuel Materials Evaluation (FME) Group will be conducting PIE on three major irradiation experiments presently in HFIR: HRB-21, NPR-1, and NPR-2 capsules, as well as capsule 1 of the HFR-B1 experiment to be shipped from Jülich, Germany. Capability improvements are under way in preparation for these and future tasks. The improvements include: providing improved particle inspection and a second Irradiated Microsphere Gamma Analyzer (IMGA) system for the IMGA hot cell; renovation of the SEM hot cell with a new evaporator coater, shielded specimen transfer device to the JEOL 840A SEM/microprobe, and refurbishment of the in-cell X-ray radiography equipment; adding metallographic polishing and specimen inspection equipment to the main specimen examination hot cells; installation of precision dimensional measurement and weighing instruments; constructing a glove box facility to house the post-irradiation gas analyzer (PGA) and a new laser drill that is being acquired to perforate individual particles and metal tubes containing piggyback specimens; acquiring a new liquid scintillation analyzer for beta-emitter
counting as well as additional gamma analysis apparatus; and designing a computer-controlled gamma-ray scanning system that will allow precise remote positioning and scanning of large objects such as entire irradiation capsules. Neutron fluence dosimetry is also being designed and fabricated to support irradiations for the HSSI Program.

During the past year, successful PIE efforts were carried out on four HFIR target rod capsules. Three of these contained graphite specimens for the Fusion and NP-MHTGR programs, while the fourth had experimental fuel for the ANS project. Group members also supplied and encapsulated the piggyback specimens for the NPR-1 and -2 irradiation experiments. Irradiated, unbound particles with high-enriched uranium (HEU) kernels from prior HRB-17 and -18 experiments were acquired from General Atomics and characterized by IMGA and metallography before and after accident-simulation heating tests in the Core ConductionCooldown Test Facility (CCCTF). The PGA facility was reactivated with an improved particle-breaking mechanism, and the first phase of samples from a WFO program was received and characterized with the IMGA system. Dosimetry was prepared for capsule 10-0D of the HSSI program, and a new HFIR dosimetry capsule featuring on-line retrievable samples was designed and received concept approval by the reactor experiment coordinator. Metallography was undertaken on specified particles that exhibited good and poor fission product retention from the HFR-K3 spheres that had been subjected to post-irradiation heating tests in Germany. Efforts were also applied to the characterization of the properties of SiC coatings on HEU fuel particles, which affect the transport of fission products. To this end, equipment was procured to measure (using a mercury porosimeter) the pore size distribution in the coatings and the strength of SiC ring specimens prepared from fuel particles.

5.3 HOT CELLS REVITALIZATION PROGRAM — P. E. Arakawa

The Hot Cells Revitalization Program (HCRP) was initiated in FY 1987 in response to Laboratory management’s request to consolidate metallurgical examination hot-cell work into two facilities: Buildings 3025 and 3525. This work is scheduled to be completed in two phases: (1) Phase I will bring both Buildings 3025 and 3525 to fully operational status and (2) Phase II will include correcting remaining design deficiencies and will modify or add systems that will promote safe and efficient operations in the hot cells of both buildings. Phase II is planned to be completed under a Multiprogram, General-Purpose Facility (MGPF), FY 1995 line item.

During FY 1991, approximately $970,000 was spent in support of the revitalization program. The major emphasis of this work in Building 3025 included the procurement and installation of two new hot-cell windows, the preparation of technical specifications to procure two additional hot-cell windows, and preparation to procure an in-cell bridge crane.

The major emphasis of work in Building 3525 included basement decontamination activities; procurement of five Central Research Laboratory, Model E manipulators to replace existing decontamination cell manipulators; refurbishment of the hot off-gas system; preparation to procure new in-cell bridge cranes; and repair of existing manipulators and cranes to bring them to operational condition.
Also accomplished under the revitalization program was the prototype testing and design of a trucking station that will sample and transfer LLLW generated in Building 3525 to a waste operations tanker truck. This work is in response to the FFA, which will no longer allow LLLW to be transferred to existing underground storage tanks. Work was also performed to characterize the liquid wastes generated in Building 3525 in order to provide in-cell filtration and recycle systems, which will reduce the amount of LLLW generated at this facility.

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

The CCCTF under development in the High-Temperature Fuel Behavior Group is designed to assess the performance of MHTGR fuel under simulated core conduction cooldown events. Upon completion, the facility will be capable of simulating both depressurized and pressurized core conduction events. The CCCTF is a remote facility located in a hot cell for the testing of irradiated MHTGR fuel compacts. Key components are: (1) high-temperature furnaces with operating temperatures up to 2300°C, (2) on-line fission product collection systems for both gaseous and condensable species, (3) automated control and data collection systems, and (4) a gas purge system with capability for injection of coolant impurities. The system concept is similar to that in operation at the Forschungszentrum (KFA) in Jülich, Germany. However, actual components are significantly different (furnace, automated control, and gas purge systems), and additional considerations for remote fuel handling, furnace component replacement, and capability for performing a fission product mass balance were included. Under normal operation, the CCCTF will be expected to monitor and collect fission products released from irradiated fuel for times as long as 1000 h at fuel specimen operating temperatures up to 2000°C in a controlled atmosphere.

The initial procurement of CCCTF components began at the end of FY 1987. Design modifications to the high-temperature furnace for fission product collection and fuel specimen handling occurred during FY 1988 and 1991. Extensive operational testing and development of the total CCCTF began in FY 1990 and continues. At the end of March 1991, the first high-temperature furnace and its attendant control, monitoring, and utility systems was installed in the remote hot cell. Full-scale, in-cell operational testing and fission product detection calibrations began in April 1991 with rare gas radionuclide and radioactive tracer tests. The results of these tests were encouraging from the point of being able to track and collect the released radioactive material, but also that a realistic mass balance was achieved among the initial source, the collection system, and the internal furnace components.

The first CCCTF test with irradiated fuel from capsule HRB-18 was initiated in August 1991 and was conducted for a period of nearly 260 h at a temperature of 1400°C. This test successfully demonstrated all phases of operation including automated control of the planned heat-up program, detection of fission product release (metallic cesium with no gaseous release), use of approved operational procedures, and fail-safe furnace shutdown. The fuel from this first test is currently undergoing detailed post-test characterization of performance and fission product retention.

A second high-temperature furnace and its attendant control, monitoring, and utility systems was completed in May 1991 to meet an NP-MHTGR program milestone. Installation of this second furnace into the CCCTF was delayed until the first part of FY 1992.
The successful development and operational testing of the CCCTF represents a significant accomplishment by the High-Temperature Fuel Materials Group. This facility will play a major role in demonstrating and qualifying coated-particle fuel performance under design basis accident events for the MHTGR. For both the commercial MHTGR and NP-MHTGR programs, this remains a key technology need.

5.5 FUEL MATERIALS TESTING — G. L. Copeland

Fuel materials testing resumed in the HFIR near the end of last fiscal year with the irradiation of a target capsule for the ANS candidate fuel and continued this fiscal year with the insertion of three gas-cooled reactor fuel capsules. Evaluation of the ANS target capsule has been delayed in the hot cells due to equipment problems and schedule conflicts with required ES&H actions but is now progressing (see Sect. 5.2).

The resolution of all open quality assurance (QA) audit items for the HRB-21 capsule for the NE-MHTGR Program required a partial rebuilding of the experiment, which was completed in March 1991. Calibration and operational procedures for the instrumentation and control facility (MIF-1) were brought up to current standards. Following installation and calibration, irradiation in the HFIR RB-3B facility began in cycle 298 in June 1991. Three successful test cycles were completed in this FY, and irradiation is continuing.

Two fuel capsules for the NP-MHTGR Program were built, installed, and began irradiation in compliance with the NP Program QA requirements. The NPR-1 capsule began irradiation in the HFIR VXF-5 position in cycle 299 in July 1991 and completed two successful test cycles by the end of the FY. The NPR-2 capsule began irradiation in the HFIR VXF-18 position in cycle 300 in August 1991 and completed one successful test cycle by the end of the FY. Irradiation of both capsules is continuing.

Operational surveillance of all three capsules includes continual monitoring of the effluent cover gas stream for fission products and calculating R/B ratios. Thermal analysis of the experiments on a daily basis, based on the thermocouple temperatures, provides guidance for control of the average and peak fuel temperatures through cover gas composition adjustments.

Work was initiated during the year to enhance the irradiation testing capability in three areas: two additional instrumentation and control facilities are being installed to support test needs, an improved flux mapping for the experimental facilities is being done to reduce uncertainties in the calculated nuclear reaction rates, and preliminary assembly and checkout was completed for an on-line fission gas sampling station to simplify the fission product analysis and to reduce the radiation exposure associated with the current sampling station.

Additional development work for the ANS fuel this year has been concentrated on fabrication of the fuel plates and modeling of the irradiation behavior. This work is centered at Argonne National Laboratory (ANL) and B&W. A mechanistic model has been developed that correctly describes the current irradiation data for $U_3S_i$. Fabrication efforts have been directed toward obtaining and verifying the required fuel gradients for the ANS core.
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 U.S. DOE: BES-Materials Sciences, Electric Energy Systems, Reactor Materials, Conservation Materials, Space and Defense, Fossil Energy, Fusion Energy Materials, and High-Temperature Superconductivity.

6.1 BASIC ENERGY SCIENCES-MATERIALS SCIENCES PROGRAM — L. L. Horton

The BES-Materials Sciences 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 of the BES Program is to develop an understanding of structural materials and materials processes at all levels, from the atomic structure to the macroscopic properties.

The program reflects the materials emphases within the division: structural ceramics/composites, high-temperature ordered intermetallic alloys, and radiation-resistant materials. It includes the research components required to better understand and utilize materials: synthesis, processing, fabrication, characterization, and development of models/mechanisms. Research in synthesis and processing science includes the development of fabrication and joining techniques for advanced intermetallic alloys, ceramics, and composite structures. The Welding Task serves as the coordinator for the national BES Welding Program. A strong first-principles theory effort is integrated with the materials development programs. Important to all of the tasks is the development and application of advanced characterization techniques, including AEM, X-ray techniques, APFIM, MPM, and ion beam techniques. Ion implantation is used to study defect interactions, radiation effects, and to modify surface-related properties of ceramics.

Several significant developments have impacted the program during the past year. The most important of these is the increasing national focus on synthesis and processing of materials. The division's BES programs played an important role in the BES Synthesis and Processing Center established through a congressional initiative in FY 1991. This center represents a collaborative effort among the BES-funded materials laboratories. Central management of the center is through Sandia National Laboratories (Albuquerque, New Mexico) with technical management distributed to five of the National Laboratories; ORNL is responsible for the Ceramics component of the center activities. As part of the center R&D activities, the division received support for postdoctoral fellow appointments in our ceramics, intermetallics, welding, and nanoindentation research. The future of the center beyond FY 1991 is not clear; Congress did not include funding for it in FY 1992. However, it is likely that there will be a presidential initiative in materials science in the near future. The probable focus of this initiative will again be synthesis and processing with an emphasis on industrial collaborations.

Another significant development during the past year is an increased emphasis, in the national BES program, on radiation effects, especially with regard to the effects of neutron
environments. As a result, the division's BES Radiation Effects Program had an infusion of funding to support our neutron irradiation efforts. While the growth in the funding of this program was significant compared to the relatively flat budgets for the balance of the R&D program, the costs of performing research with neutron environments have escalated dramatically over the past 5 years—and are still rising. ES&H issues have also increased the costs of accelerator operation, another major facility used in radiation effects investigations.

As a result of continuing flat-dollar R&D budgets and escalating Laboratory overhead rates, an assessment of the division's BES programs has led to the dissolution of the Structure and Properties of Surfaces and Interfaces Task. The Nanoindenter research component of this task has been merged with the Microscopy and Microanalysis Task. Investigations of the effects of ion implantation on ceramics will be continued, at a reduced level, in the Radiation Effects Task. These changes will consolidate related research tasks to strengthen the remaining program.

The national program continues its strong support of national user facilities and programs. The BES Program supports the SHeARE Cooperative Research Program and the Oak Ridge Synchrotron Organization for Advanced Research (ORSOAR). The SHeARE Program allows scientists from universities, industries, and other national facilities to have access to facilities in the M&C Division, especially the AEMs, the APFIMs, and the MPMs. The ORSOAR Program contributes to the support of an X-ray station at the National Synchrotron Light Source at Brookhaven National Laboratory (BNL). This station is used for cooperative research by scientists from more than 20 universities and industrial institutions that are part of a user group organized by ORNL and ORAU.

6.2 REACTOR MATERIALS — P. L. Rittenhouse

6.2.1 Liquid-Metal Reactor Materials Technology

Studies involving long-term creep, thermal aging behavior, fatigue and creep fatigue, and fracture mechanics of Modified 9Cr-1Mo steel were completed during 1991. The work was conducted under an agreement between DOE and the JAPC and emphasized confirmation of the suitability of this material for liquid-metal reactor (LMR) steam generator applications. Extension and continuation of several aspects of this work have been proposed, but no agreements are yet in place.

6.2.2 Civilian Gas-Cooled Reactor Materials Programs

The DOE's gas-cooled reactor efforts are directed to the MHTGR. Development of the fuels and materials technology necessary for the design and licensing of the MHTGR has been assigned to ORNL. Areas of particular interest are the performance of low-enriched uranium (LEU)-coated fuel particles; behavior and transport of fission products from the fuel; properties and behavior of the graphites used for fuel blocks and core internals; and properties of the metals and ceramics used in-reactor, for the vessel system, and the steam generator. Those efforts, which are generic to the civilian MHTGR and the MHTGR version
of the NPR, are being funded by DOE's ONPR as discussed in the next section. Those efforts specific to the LEU fuel and to international cooperation are funded by the civilian program.

The major fuel technology accomplishment during the year was the construction and irradiation of a fuel capsule (HRB-21) in the HFIR. The irradiation is continuing into FY 1992. Major effort was also devoted to preparations for the fission product transport experiments, which are planned in an in-reactor high-pressure loop (COMEDIE) at a government research facility in France. The experiments will involve plateout of fission products from intentionally defected fuel and subsequent measurements of liftoff during simulated MHTGR depressurization events. A preliminary test (BD-0) of the system without fission products was successfully completed.

Cooperation with Germany and Japan on fuels, graphites, and metals continued under formal agreements. The most significant areas of activity were PIE of LEU fuels and the continued irradiation in HFIR of a capsule containing U.S., German, and Japanese nuclear graphites.

6.2.3 Materials Technology for the MHTGR NPR

A four-module MHTGR for weapons-materials production is under study and development for the ONPR. All technology confirmatory development efforts on structural materials are the responsibility of ORNL. Confirmatory development effort on the HEU fuel required for the MHTGR NPR is a charge of EG&G Idaho, but almost all of the experimental work is performed by ORNL. All of the work is carefully interfaced and controlled through formal test specification, test plans, and test procedures.

The design and construction of two fuels irradiation capsules (NPR-1 and -2) was completed late in the year, and six-cycle irradiations in HFIR were initiated and continue. Performance of both capsules has been excellent with no indications of fuel failure. Construction and commissioning of a high-temperature facility for heating of irradiated fuel particles under simulated off-normal conditions was completed, and the first in a series of planned experiments was run. This test involved 80 fuel particles heated to 1400°C over a period of 60 h and held at that temperature for 260 h. No fuel particle failure was observed.

Two irradiation capsules containing H-451 production graphite and a series of H-451 experimental graphites made with isotropic cokes from various sources were fabricated and inserted in the HFIR. The first capsule (HTN-1 at 900°C) was removed from irradiation after four cycles in HFIR to provide early information on the performance of the experimental graphite grades relative to that of production H-451. Dimensional stability equivalent to that of production H-451 was exhibited by a number of the experimental graphites. Irradiation of the second capsule (HTN-2 at 600°C) is continuing. Construction of the HPTL for oxidation studies of graphites was completed, and commissioning of the system is in progress. Scoping tests for graphite mechanical and physical properties were also conducted.

Preparations were made for irradiation testing of Alloy 800H material used in the MHTGR internals. This included calibration of temperatures and flows in the HFIR rabbit tube in which the first irradiations will be performed. Weldments of Alloy 800H were manufactured and
specimens of base metal and the weldments machined in preparation for aging, irradiation, and mechanical testing. Considerable effort was also devoted to the acquisition and setup of test chambers and test equipment.

Full-sized blocks of the Coors AD-85 alumina ceramic to be used as insulation in the lower core support structure were obtained and characterized. This characterization included measurements for flaw size and distribution, physical properties, thermal properties, and strength.

Irradiation of four capsules containing tensile and impact specimens of RPV steels was conducted in the Ford Nuclear Reactor at the University of Michigan. The specimens were returned to the hot cells at ORNL, and their testing is in progress. Design of additional irradiation capsules with neutron energy spectrum tailored to match that expected in the MHTGR is in progress.

Evaluation of several austenitic and ferritic alloys as candidates for MHTGR steam generator evaporator-economizer-superheater (EES) and finishing superheater (FS) tubing was a major effort throughout the year. Included in the evaluation was the collection and analysis of tensile, creep, and fatigue data for a number of Alloy 800 variants with different chemistry and heat-treatment specifications. An agreement was reached among all organizations involved with the MHTGR to use an Alloy 800 with chemistry and heat treatment similar to that used in the Fort St. Vrain (FSV) reactor as FS tubing. The EES tubing will be the 2¼ Cr-1Mo steel also used in FSV.

6.2.4 Materials Technology for the Heavy-Water Reactor NPR

ORNL also has a major role in the technology development of structural materials for the HWR NPR. The Savannah River Laboratory (SRL) and ANL are also involved in these efforts. Ongoing efforts at ORNL include materials mechanical properties characterization, irradiation effects, and aluminum corrosion behavior.

The mechanical properties task is aimed primarily at pre-irradiation characterization of vessel materials. Design of the instrumented HFIR irradiation capsules for the vessel materials was completed. The irradiations test program will be used to evaluate irradiation-induced degradation such as loss of toughness and IASCC. A corrosion loop and fatigue test system were designed and procured for the hot cells to permit IASCC investigations on the irradiated specimens.

The aluminum corrosion task is evaluating the corrosion performance of the 8001 aluminum alloy HWR fuel cladding under bounding conditions of temperature, water chemistry, and thermal hydraulics. The corrosion loop being used is an ANS Program facility. The first 7-week test in the ANS loop was completed with a coolant inlet temperature of 49°C and a heat flux of 4.5 MW/m².

Our materials R&D programs for energy conservation have grown significantly with DOE emphasis on increased energy efficiency and with the realization that materials are a key technology need for advanced energy conversion and utilization systems. We have established lead laboratory roles and/or major materials support tasks in the following conservation projects: (1) Ceramic Technology, (2) AIC Materials, (3) Tribology, (4) Materials for Industrial Technologies, and (5) Building Materials. In the Ceramic Technology and the AIC Materials projects, 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.

The Ceramic Technology Project was initiated in FY 1983 to meet the ceramic materials technology needs of the companion DOE engine programs. The goal of the program is to establish the technology base that will allow private industry to supply reliable and cost-effective ceramics for use in advanced engines and other energy conversion applications. The program is being 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 program includes 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) data base 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.

It is expected that the program goal of reliable ceramics will largely be met by the 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 is clouded by the relatively high cost of the ceramic components. Based again on extensive input from industry, the direction of the Ceramic Technology Project 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 Gas Turbine (AGT) 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 data base development.

ORNL is responsible for the "Tribology by Design" task area of the Transportation Materials Tribology Program. The ORNL Tribology task is divided into two subtasks: triboscience and tribomaterials evaluation. The former addresses the fundamental aspects of friction and wear of materials, and the latter involves friction and wear testing of advanced ceramics, composites, and intermetallic compounds, which show potential for wear-critical applications.
Research in triboscience has resulted in the development of a unique "friction microprobe," a device that uses a computer-controlled stylus to measure friction forces on micrometer-sized contacts. It is being used to study point-to-point variations in the friction of composite materials and the effects of powder (debris) layers on friction. A subcontract to the Georgia Institute of Technology has led to the development of thermal-mechanical surface damage prediction maps for unlubricated ceramics, and an ongoing follow-on effort is extending the work to lubricated ceramics.

Research in tribomaterials evaluation has involved studies of the conditions that lead to transitions between acceptable and catastrophic wear rates in ceramic-based composite materials. Silicon nitride reinforced with silicon carbide whiskers has been the subject of this work. A wear map has been developed for silicon nitride sliding on the composite under lubricated conditions. This map helps identify pressure-velocity-cycles combinations that cause undesirable wear and friction. Next year, new projects will study in situ reinforced silicon nitride and carbon-based materials like carbon-graphite composites.

The Advanced Industrial Concepts Division (AICD) is a new unit in Conservation and Renewable Energy in DOE. AICD includes many of the programs in the previous Energy Conversion and Utilization Technologies (ECUT) Division. The mission of AICD is to develop and maintain a balanced program of R&D focused on high-risk, long-term, directed interdisciplinary research efforts for the industrial sector in its efforts to improve energy efficiency and enhance fuel flexibility. The AIC Materials Program supports this mission, giving attention to materials engineering in the context of goals, needs, and opportunities for advanced industrial systems. The program initiates and conducts applied research and exploratory development in technical areas encompassing structural engineering materials, materials with unique (nonstructural) properties, materials processing for manufacturing, and environment-compatible materials. Projects under the AIC Materials Program at ORNL, along with subcontracts funded through ORNL, focus on the development of high-temperature materials such as intermetallic alloys (e.g., NiAl, NiAl, and FeAl); continuous fiber-reinforced TiB2 for improved Hall-Cell electrodes in the alumina smelting industry; ultratough metal-bonded ceramics; high-temperature superconductors for magnets and power electronics for high-efficiency electric motors; and materials processing technology, including microwave processing and surface-modified polymers with improved properties.

Two industrial projects are being conducted for the Industrial Energy Efficiency Division of the Office of Industrial Technologies (OIT). The objective of the first project is to provide materials technology support to develop advanced ceramic heat exchanger systems for industrial applications. ORNL is determining the corrosion limits of the ceramic materials and developing cost-effective methods for fabricating heat exchanger tubes cooperatively with several industrial suppliers and users. The second project for this office was to assist them in implementing a new program on 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 increased productivity. ORNL assisted DOE-OIT in developing the program plan and is now providing technical support to implement the program.

The objective of the Building Materials Project is to establish the technical data base for building materials that is needed to reduce the energy used for buildings. The research is
developing testing techniques, new standard reference materials, analytical models for heat transfer, and alternatives for insulations containing chlorinated fluorocarbons, including new foam-blowing agents and PEPs of high thermal resistivity.

The interaction of these projects in the Conservation Materials Program with related research on the Materials Sciences and on the Fossil Energy Program at ORNL, and with research at other federal agencies and in industry, is synergistic and very productive. We anticipate continued growth in the Conservation Materials Program.

6.4 SPACE AND DEFENSE — R. H. Cooper

During the past year, ORNL's DOE-sponsored Space Nuclear Power Programs combined with activities from the NASA, DoD, and industrial organizations have proven to be an exciting opportunity for materials technology development. The status of the DOE Space Nuclear Power Programs and selected NASA and DoD programs are discussed below.

6.4.1 DOE Space Nuclear Power Programs

The M&C Division continues to provide a significant contribution to DOE Space Nuclear Power Programs. ORNL plays a major role in the effective application of advanced materials technology to the achievement of the goals of the RTG Materials Production and Technology Tasks and the SP-100 Project. Further, ORNL is identifying the material technologies that must be successfully developed to enable space nuclear propulsion concepts required for the nation's Space Exploration Initiative.

For the SP-100 Project, we perform a significant role in the development of the engineering data base for the refractory metals required for successful operation of a multi-100-kW(e) space nuclear power system. The principal focus of this work is the determination of mechanical properties, optimization of fabrication processes for refractory metal product forms used for fuel system and structural applications, characterization of irradiation effects, and evaluation of the compatibility of the refractory alloys under anticipated operating conditions. Further, the M&C staff provides the managerial oversight for the characterization of materials and their subsequent fabrication into large radiation-shield components, which is performed at the Y-12 Plant, and for the development of temperature sensors carried out in the Instrumentation and Controls Division.

In the area of RTG systems for space and terrestrial applications, ORNL plays a major role in materials development and production. Production includes fabrication of iridium alloy blanks and foil for subsequent manufacture into clad vent sets, which contain the heat-generating radioisotopes, and the manufacture of CBCF thermal insulators (sleeve and disc), which minimize temperature changes at the surface of the fuel cladding during off-normal RTG operations. To assure ourselves, DOE-OR, and DOE-HQ that all special actions necessary to ensure uninterrupted manufacture of quality hardware have been implemented, an operational readiness review of all manufacturing processes was performed. This review assessed and documented equipment and material adequacy; manpower training and availability; compliance with ES&H requirements; and QA planning and implementation. The
successful completion of the operational readiness review for these manufacturing activities has allowed production of this flight-quality hardware for potential use in NASA's CRAF and Cassini missions.

Important materials-development activities continue in parallel with RTG flight-hardware production. These activities include characterization of the thermal conductivity of the CBCF material for temperatures to 2300 K using thermal diffusivity methods, optimization of state-of-the-art NDE methods to support the fabrication of advanced thermoelectric elements, and design of a high-temperature carbon composite material with enhanced kinetic energy absorption capability.

During this year, the M&C staff provided managerial coordination of MMES contributions in planning the implementation of a new Space Nuclear Propulsion (SNP) Program in support of the final Exploration Initiative. MMES staff were appointed to a number of technology panels chartered to inventory existing technical capabilities and facilities and to plan for the development and optimization of those technologies to meet the performance goals of the SNP Program. In this capacity, the M&C staff members prepared a structural materials technology plan for the program. Implementation of technical activities described in this materials plan is expected to begin in FY 1992.

6.4.2 NASA Programs

An important task being performed in the division in support of the NASA Marshall Space Flight Center is the determination of the mechanical properties of superalloys at room temperature 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 test methods include tensile, fatigue, and fatigue-crack growth and will be followed by all NASA contractors. During the upcoming year, an ORNL facility should be qualified to perform mechanical properties tests in a high-pressure hydrogen environment at elevated temperatures.

6.4.3 DoD Programs

Significant materials development activities are being performed in support of the U.S. Army and the USAF. Highly successful work continues for the Army's Strategic Defense Command in the development of optical materials. Work is being performed for Wright Laboratory in characterizing the CVI process that is being considered for the fabrication of ceramic composites for advanced man-rated turbine engine components. This work complements efforts supported by the Air Force Office of Scientific Research to evaluate the nucleation kinetics of surface films deposited by chemical vapor methods. In work sponsored by Wright Laboratory, exciting progress is being made in the area of MBE growth of refractory oxide coatings for waveguide applications.

6.5 FOSSIL ENERGY PROGRAM — R. R. Judkins

The ORNL Fossil Energy Program Office is a part of the M&C Division, and it is from this office that the activities of the Program, both in the M&C Division and in several other
Some to provide us with an understanding of the microwave-sintering phenomenon. The focus of ORNL's Fossil Energy Program is on materials R&D, ES&H activities, bioprocessing of coal to produce liquid or gaseous fuels, combustion research, and modeling activities on the operational requirements for the Strategic Petroleum Reserve. Materials R&D covers research on ceramics, new alloys, and the mechanisms of erosion and corrosion. Transfer to industry of the technology covering the knowledge, materials processes, and the procedures generated is an important activity of the program. Ceramic composites and new alloys are being examined for filtration and structural applications. Advanced ceramic membranes are being developed for hydrogen and other gas separation. Materials technical support and failure analyses are provided to projects on the Clean Coal Technology Program. Virtually all the materials research on the Program within ORNL is performed in the M&C Division.

Work is also being conducted on the chemistry and structure of coal surfaces and interfaces to understand the complex processes occurring in coal during combustion. In other ORNL divisions, the EH&S activities primarily address documentation required by the National Environmental Policy Act (NEPA) for projects on the Clean Coal Technology Program. In addition, technical support is being provided to DOE on issues surrounding global warming, air quality, and solid waste. In the combustion area, characterization and combustion of products from mild gasification processes are being examined.

The major portion of the Fossil Energy Program is the materials research activities, including management (with DOE-OR) of the Fossil Energy AR&TD Materials Program. Fiber-reinforced ceramic composites with improved strength and toughness are being produced by a special chemical vapor infiltration and deposition (CVID) process developed at ORNL. With the forced flow, temperature gradient of the ORNL process, thicker material can be produced, whether it be plate or tubing. Thicker SiC composites have potential application as tubing and headers in high-temperature heat exchangers. Control of the interface between the fiber and matrix in composites allows greater toughness through fiber pullout during fracture. The ability to control the porosity of these ceramic composites through the CVID process means that both highly dense (for structural purposes) as well as porous composites (for hot-gas cleanup filters) can be fabricated.

Ceramic membranes for the separation of gases in high-temperature and hostile environments are being developed and tested. Investigators at the Oak Ridge K-25 Site have produced ceramic (alumina) membranes with pore diameters approaching 10 Å, a size close to the pore size necessary for hydrogen separation. Membranes have already been fabricated and successfully tested for their permeability to nitrogen, helium, and carbon dioxide at room temperature in the pressure range of 15 to 150 psi. The fabrication of these membranes is based on classified barrier technology, which has been downgraded from SECRET to CONFIDENTIAL. Declassification has been delayed, but industries with clearances are expected to participate in the transfer of this technology.

Other work is devoted to the microwave sintering of ceramics. ORNL has developed the ability to sinter certain ceramics to high densities at temperatures several hundreds of degrees below those required using conventional radiant heating. Basic studies have begun to provide us with an understanding of the microwave-sintering phenomenon. The mechanisms by which microwaves interact with materials depend on their electronic structure. Some interact with the microwave electric field with electronic charge carriers, and others
interact with ionic charge carriers. This technology could be important in the fabrication of electrode and electrolyte materials with improved electrical properties for solid oxide fuel cells.

ORNL is developing advanced austenitic alloys for use in fluidized-bed and advanced steam cycle coal combustion power plants. The objective of this work is to modify existing alloys and to develop new alloys that will satisfy the strength and corrosion resistance requirements of high-temperature and high-pressure, second-generation power plants. The modified 800 alloys and the lean austenitic stainless steels, termed "lean" because of their lower-than-usual content of the strategic metal chromium, are high-strength steels developed for high-temperature applications typical of those in coal combustion environments. The high-temperature creep life of these alloys is several orders of magnitude greater than that of conventional alloys. It has been demonstrated that the ORNL-developed modified 800 and 316 advanced austenitics can be fabricated with commercial equipment, and many compositions are weldable. Due to their exceptionally high creep strength at 500 to 750°C, higher strength welding filler metals are being explored. Current materials and designs for tubesheet and manifolds for hot-gas filter systems have been examined to recommend a tubesheet material suitable for long-term operation of these systems.

Intermetallic alloys based on Fe₃Al are being developed for applications in which superior oxidation and sulfidation resistance and strength are required. The iron aluminides are intermetallic compounds that, for many years, have been known for many outstanding properties, but their brittleness at ambient temperatures precluded many applications. Alloy modifications and special heat treatments developed at ORNL have produced tensile ductilities of over 15% at room temperature. Several compositions are weldable with the use of pre-heat and post-weld heat treatments, as is often needed with other high-strength materials.

Basic studies of erosion and corrosion have been conducted to develop a fundamental understanding of these processes and their relationship to materials properties. Corrosion research in the M&C Division centers on studies of the formation and breakdown of protective oxide scales, particularly in sulfur-containing atmospheres, and on the effect of environment on corrosion and mechanical properties of iron aluminides. A technique for measuring the hardness and, thus, the mechanical properties of the scales is being developed.

Assessments of materials problems and of the needed research to solve those problems for a variety of fossil energy technologies is an important part of ORNL's materials effort. Materials failure analyses, a significant factor in the success of advanced clean coal technologies, continue to be conducted for the Pittsburgh Energy Technology Center. Similar technical support is provided to operators of coal conversion and utilization plants in the identification of and solutions for materials problems.

In addition, ORNL has a commitment to transfer the technology developed on the Fossil Energy Materials Program to industry and to others in the fossil energy community. Licensing agreements have been signed with three industrial firms for the ORNL-developed iron aluminides technology. One has been negotiated with Ametek Specialty Metal Products Division for the purpose of producing Fe₂Al powders. Licenses for other product forms have been awarded to Harrison Alloys and Hoskins Manufacturing Company. A CRADA has been signed between 3M and ORNL for work on an SiC filter process.
Research is also being conducted on the structure and chemistry of coal surfaces and interfaces to understand the complex processes and reactions occurring during the combustion of coal. Recent results have revealed that the hydrogen-bonding structures of coal and the surface area of finely divided coal are expected to have a significant impact in the understanding of the properties of coal.

6.6 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 ITER and the development of low-activation ferritic steels, vanadium alloys, structural ceramics, and ceramic composites (e.g., SiC/SiC).

Austenitic steels are the leading candidate for structural applications in 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 displacements per atom (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 (EDA) [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, 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 $1 \times 10^7$ 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 carbon-carbon research activities are part of the plasma interactive and high-heat-flux materials programs. Graphite and carbon-carbon 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 carbon-carbon composites with extremely good thermal shock, erosion, and neutron damage resistance. Optimum thermal shock resistance is assumed to be offered by appropriately designed carbon-carbon composites (i.e., selected fibers, matrices, and architectures). Current work is directed toward the optimization of these materials for neutron-damage resistance.
7. COLLABORATIVE RESEARCH FACILITIES AND TECHNOLOGY TRANSFER

7.1 ORNL/ORAU SHARED RESEARCH EQUIPMENT PROGRAM (SHaRE) — 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 is aimed at collaborative research in materials science in areas pertinent to the U.S. DOE and the ORNL mission 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, Auger electron spectroscopy (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, the Division of Materials Sciences, Office of BES, provided funds through ORAU to support the SHaRE activity. Program funds are used for travel and living expenses for university SHaRE participants while at ORNL and for the support of Mr. Neal Evans, a liaison between M&C Division research staff and the SHaRE participants. His principal responsibilities included participation in SHaRE research when appropriate and familiarizing SHaRE participants with the electron microscope and computer facilities. His presence has 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 1991 were: E. A. Kenik and K. B. Alexander, ORNL; M. G. Burke, Westinghouse Science and Technology Center; G. M. Pharr, Rice University; and N. D. Evans and R. Wiesehuegel, ORAU.

During this reporting period, 17 of 18 approved SHaRE projects were active; five of the active projects did not require travel support. The active projects involved 23 outside participants (users). At least 22 papers based on SHaRE research were published in the past 12 months, and approximately 14 presentations have been made at technical meetings. Currently, another six 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. One Master of Science dissertation and four Doctor of Philosophy degrees were completed. Additionally, ten more graduate students are progressing in their dissertation studies by participating in the SHaRE Program.

7.2 ORNL/ORAU SYNCHROTRON ORGANIZATION FOR ADVANCED RESEARCH — C. J. Sparks

The M&C Division X-Ray Group, in collaboration with the Materials Research Laboratory at the University of Illinois, operates an intense X-ray beamline on the National Synchrotron Light
Source to study the crystallographic structure of weakly scattering materials. This beamline is open to outside users for qualified experiments. During this past year, 21 different experiments were performed by 56 scientists from 4 different universities, 3 industries, and 6 government laboratories with 16 published papers, including 3 Ph.D. theses and 4 Physical Review Letters. Our use of the beamline took advantage of the ability to select an X-ray energy to interact with an absorption edge of a specific element. This highlighted the atom and revealed its crystallographic symmetry site so that we were able to determine site substitution parameters. In this work, we studied the phase stability of Al_{5}Ti alloyed with iron and determined how much iron substituted on each site before the tetragonal-to-cubic phase transformation occurred. Similar studies were made on nickel atoms substituted in a CuAu intermetallic compound. Structures of icosahedral phase alloys, monolayers on liquid surfaces, interfaces created by overlayers on single-crystal substrates, amorphous oxides, and solid neon are examples of the variety of research problems tackled with this X-ray probe. Spectroscopy measurements of the band states and 1-s core state of lithium in LiH have revealed new features that are providing a better understanding of the chemical bonding as related by the electron energy levels.

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

Substantive activities this year involved providing technical assistance to current and potential licensees, conducting one technology transfer meeting, successfully negotiating six new licenses, negotiating collaborations to further develop ORNL technologies, and providing information on technologies through oral presentations and written communication.

To date, the 23 licensees to the M&C Division technologies are:

1. American Matrix; Advanced Composite Materials; Cercom, Inc.; Dow Chemical; GTE; Hertel; High Velocity; 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_{5}Al alloys);
4. 3M (novel ternary ceramic alloy);
5. Coors Ceramics (gelcasting method of making complex ceramic shapes);
6. Ametek*; Harrison Alloys*; and Hoskins Manufacturing Company* (iron aluminides); and
7. Microscience* (atom probe software/field ion microscope).

A technology transfer meeting on nickel and iron aluminides was held on August 27-28, 1991. The attendance at this meeting was restricted to the licensees and those organizations that had shown substantial interest in the technologies. Approximately 27 representatives attended.

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 CRADAs in the M&C Division:

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<th>Client</th>
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<td>Eaton &amp; Johnson Controls</td>
<td>Shape Memory Alloy</td>
<td>06/13/91</td>
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<tr>
<td>Garrett (Allied Signal)</td>
<td>Microwave Annealing of SiN with High Additive Content</td>
<td>08/22/91</td>
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<tr>
<td>Norton Company</td>
<td>Microwave-Sintered Reaction-Bonded SiN</td>
<td>09/05/91</td>
</tr>
<tr>
<td>Norton Company</td>
<td>Microwave Annealing of SiN with Low Additive Content</td>
<td>09/05/91</td>
</tr>
<tr>
<td>Appliance Research Consortium</td>
<td>Powder-Evacuated Panel</td>
<td>07/22/91</td>
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<tr>
<td>Microwave Labs</td>
<td>Wideband Microwave Processing Equipment</td>
<td>In progress</td>
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<tr>
<td>AVX Tantalum Corporation</td>
<td>Microwave-Sintering Tantalum Capacitors</td>
<td>In progress</td>
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<tr>
<td>United Technologies Research Center</td>
<td>Structural Ceramics for an Advanced Coal Combustor</td>
<td>In progress</td>
</tr>
<tr>
<td>3M Company</td>
<td>Chemical Vapor Infiltration of Ceramic Composites</td>
<td>In progress</td>
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</tbody>
</table>

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

Through actions of the M&C Division Technology Transfer person, the twin-cities region of Scottsbluff-Gering, Nebraska, has decided to form a technology transfer "hub" in Scottsbluff. The function of this hub will be to reach the small and medium-sized industries in Nebraska and regions in Colorado, South Dakota, and Wyoming through their community college network with technologies emanating from the federal laboratories and improving the college curricula in technological areas. This technology transfer concept arose from a joint ORNL, Michigan Technological University, and DOE-funded project called the University-Laboratory-Industry Technology Brokerage System.
M&C Division's involvement in educational activities has dramatically increased over the past few years. Our traditional college-age cooperative intern, graduate student, and postdoctoral programs have expanded, and we have become involved with pre-collegiate education. In FY 1991, there were more than 100 "paid" guests (~15 professors, 2 high school teachers, over 70 graduate/undergraduate students, and 12 postdoctoral fellows) in the division. These personnel are brought into the division by a host of programs coordinated by ORAU and the Southeastern University Research Association, by the ORNL co-op program, and under university and personal services subcontracts. In addition, ~2,000 pre-collegiate students and over 300 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 350 pre-college students and over 100 teachers actually toured division facilities. These tours included presentations about materials science and hands-on demonstrations of electron microscopy (most participants operate an SEM and take a micrograph), ceramic processing, and superconductivity. The students were given something to remember their visit: a couple of SEM micrographs, a Materials Science brochure, the National Geographic issue on materials science, and/or a one-page handout that outlines the evolution of materials usage.

Two years ago, our pre-college education programs began with the establishment of a "Fun with Materials" presentation. Mike O'Hern, our main presenter, is currently on leave from ORNL but continues to give these presentations at local schools under subcontract. M&C Division provides all of the necessary supplies and equipment, including the aluminum disks presented to each student. A slide show, "The Microscopic World," has also been developed for in-school and auditorium presentations. School outreach programs involved over 1,200 students and more than 200 teachers.

M&C Division was involved with several other major educational outreach programs including "Science in Action," the ORNL National Science Foundation (NSF) National Teacher Enhancement Workshop on Materials, the Saturday Academy for Computing and Mathematics, and a new initiative, the Appalachian Regional Honors Workshop. The Science in Action Program is a one-week, multidisciplinary program held during Engineer's Week in February. It is affiliated with the WATTeC conference and involves local technical professional societies. M&C Division provides one of the co-chairmen and several of the speakers and exhibits. In 1991, Science in Action expanded to include elementary and high school students in addition to middle school students. Over 600 students and more than 50 teachers participated. M&C-sponsored presentations included "Fun with Materials" and "Ceramic Processing," as well as exhibits on microscopy and high-temperature materials. In addition, M&C Division staff were involved with the Saturday Academy for Computing and Mathematics, a program for ~25 upper-level high school students; one of eight sessions was given in the division by the Theory Group.
The 1991 ORNL-NSF Teacher Enhancement Workshop focused on materials science. This is the second year of M&C Division involvement with this program. We presented over two days of presentations, interactive demonstrations, and tours to 45 elementary and middle school teachers. We also gave a half-day presentation/tour program for 75 high school teachers as part of the University of Tennessee Academy for Teachers of Science and Mathematics.

Our largest, single effort for this year was a new initiative, the Appalachian Regional Honors Workshop. This was a 2-week research experience for 31 high school students and 11 teachers from disadvantaged counties in the Appalachian region. The student activities included research projects, a recycling-oriented theme session (including a tour of Alcoa Aluminum Company and interactive, role-playing discussions), lunch seminars, a blacksmithing experience, and diverse social activities. The group was divided into 11 research teams, with a teacher assigned to each team. The research topics were:

- Processing of Zirconia-Strengthened Alumina Ceramics (Alan Bieier)
- Reproducibility of Thermal Diffusivity Measurements of Carbon-Bonded Carbon-Fiber (CBCF) Insulation (Ralph Dinwiddie)
- Laboratory Growth of the Hardest Material in the World: Diamond (Lee Heatherly)
- Where do All Those Alloys Come From? (Dick Heestand)
- ZAP! Microwave Processing of Ceramics (Mark Janney)
- High Tc Superconductors: Synthesis, Processing, and Characterization (Fred List)
- Investigation of Creep and Tensile Strength of Various Metals and Alloys (Claudette McKamey)
- The Microscopic World: Characterization of Ceramics Using Electron Microscopy (Karren More)
- Putting Things Together: Evaluation of Welding and Brazing Techniques (Mike Santella and Jim King)
- Damage-Tolerant Ceramics (Dave Stinton and Millicent Clark)
- Selection of Corrosion-Resistant Materials for Vinaigrette Dressing Production (Dane Wilson)

During the second week of the program, the students wrote a short report of their research and gave an oral presentation of their results. This program was a "trial" for next year when M&C Division will also be involved with the ORNL-DOE High School Honors Workshop. The DOE Honors Workshop is a 2-week research experience for 60 high school students, one from each state and several foreign countries.
Appendix B

PERSONNEL SUMMARY

October 1, 1990, to September 30, 1991

Compiled by Barbara Lovelace

New Staff Members

A. Scientific Staff

P. A. Carpenter  Space and Defense Programs
N. C. Cole  Fossil Energy Materials Program
J. T. Parks  Fuel Materials Testing Group
J. E. Pawel  Structural Materials Group

B. Administrative and Technical Support Staff

D. A. Ellis  Conservation Materials Programs
J. L. Fair  Fossil Energy Materials Program
V. M. Gibson  Space and Defense Programs
M. L. Hodges  Communications and Records Support Services Group
J. H. Miller  Ceramic Surface Systems Group
C. J. Overton  Communications and Records Support Services Group

Staff Transfers and Terminations

A. Scientific Staff

P. E. Arakawa  Transferred from Engineering Division to Hot Cells Revitalization Program
E. Bolling  Retirement
R. A. Cunningham  Retirement
C. K. H. DuBoise  Retirement
G. Farquharson  Transferred to K-25 AVLIS Division from Enrichment Materials Group
J. I. Federer  Retirement
L. A. Harris  Retirement
H. W. Hayden, Jr.  
Transferred to K-25 AVLIS Division from Enrichment Materials Group

C. L. Jackson  
Retirement

C. R. Kennedy  
Retirement

H. E. Kim  
Voluntary resignation

E. J. Lawrence  
Retirement

D. L. McElroy  
Retirement

C. J. McHargue  
Retirement

H. O. McNabb  
Transferred from Chemical Technology Division to Irradiated Fuels Examination Laboratory

H. O. McNabb  
Retirement

J. M. Miller  
Transferred from Chemical Technology Division to Irradiated Fuels Examination Laboratory

J. M. Miller  
Retirement

R. N. Morris  
Transferred from Computing and Telecommunications Division to High Temperature Fuel Behavior Group

D. L. Moses  
Transferred from Engineering Technology Division to NPR Project Office

R. A. Padgett  
Retirement

M. Payne  
Transferred from Chemical Technology Division to Irradiated Fuels Examination Laboratory

H. E. Resor  
Disability

A. C. Schaffhauser  
Transferred to Central Management Organization from High-Temperature Superconductivity Pilot Center

M. A. Schmidt  
Transferred to K-25 Decommissioning and Decontamination Division from Materials Analysis Group

B. W. Sheldon  
Voluntary resignation

R. D. Taylor  
Transferred from Chemical Technology Division to Fuel Materials Evaluation Group

N. F. Wright  
Transferred to Central Management Organization from Theory Group

B. Administrative and Technical Support Staff

S. C. Barnwell  
Voluntary resignation

A. B. Boatwright  
Transferred to K-25 AVLIS Division from Enrichment Materials Group

D. L. Byrum  
Transferred from Chemical Technology Division to Irradiated Fuels Examination Laboratory

S. D. Childs  
Transferred from Chemical Technology Division to Irradiated Fuels Examination Laboratory

T. L. Collins  
Transferred from Solid State Division to Fuel Materials Evaluation Group

K. M. Cooley  
Transferred from K-25 Analytical Support Services Division to Ceramic Surface Systems Group

M. J. Gardner  
Transferred from Y-12 Product Certification Division to Metallography Group
J. R. Gilley
Transferred from Chemical Technology Division to
Irradiated Materials Examination and Testing Group

A. R. Hawkins
Transferred to Central Management Organization from
Microscopy and Microanalytical Sciences Group

P. J. Hughes
Transferred from High-Temperature Superconductivity Pilot
Center to Central Management Organization

P. M. Humphreys
Transferred from Human Resources Division to High
Temperature Materials Section

S. S. Knee
Transferred from Human Resources Division to Administrative
Services Assignment Office

K. E. Long
Transferred from Chemical Technology Division to Irradiated
Fuels Examination Laboratory

G. C. Marsh
Transferred to K-25 Technical Services Division from
Metallography Group

J. Z. Palmer
Transferred from Engineering Technology Division to
NPR Project Office

P. S. Rice
Transferred to Office of Environmental and Health Protection
Division from High Temperature Materials Section

M. R. Rogers
Transferred from instrumentation and Controls Division
to Alloing Behavior and Design Group

C. L. Rose
Transferred from Energy Division to Administrative and
Engineering Services Group

K. Spence
Transferred from Y-12 Engineering Division to
Communications and Records Support Services Group

B. F. Thomas
Retirement

M. B. White
Retirement

P. A. White
Transferred to K-25 AVLIS Division from Enrichment
Materials Group

G. N. Worley
Transferred from Chemical Technology Division to
Irradiated Fuels Examination Laboratory

L. M. Wright
Transferred from Engineering Technology Division to
Administrative Services Assignment Office

C. B. Yount
Transferred to K-25 Policy and Management Systems
Division from High Temperature Materials Section

Co-Op Assignments

S. F. Alexander
University of Florida

N. H. Asuncion
Georgia Institute of Technology

N. S. Bell
Georgia Institute of Technology

G. L. Edgemon
Georgia Institute of Technology

P. A. Ferguson
North Carolina Sate University

D. Guerguerian
Georgia Institute of Technology

S. E. Hawkins
North Carolina State University

S. D. Knowles
Clemson University

D. M. Melton
Clemson University

J. H. Miller
University of Tennessee
L. M. Pike          University of Tennessee
C. R. Pritchard     University of Tennessee
O. J. Schwarz       Tennessee Technological University
M. D. Teske         Georgia Institute of Technology
J. S. White         Tennessee Technological University

Summer Assignments (1991)

A. Summer Research Interns

K. S. Ailey-Trent   Tennessee Technological University
T. M. Beavers       Cleveland High School
D. L. Elder         University of North Carolina
J. T. Hart          Ohio State University
P. A. Reichle       University of Tennessee
L. C. Russell       North Carolina A&T State University
D. V. Squire        North Carolina A&T State University
M. J. Swindeman     University of Tennessee

B. Administrative Support Staff

C. B. Erickson      Brigham Young University
W. L. Fair          Carson-Newman College
A. R. Kaufmann      University of Tennessee
L. M. Kendrick      Roane State Community College
S. R. Odom          Roane State Community College
H. L. Pigman        David Lipscomb University
A. I. Price         East Tennessee State University
J. A. Russell       Middle Tennessee State University
L. Woods            Paducah Community College

Guest Assignments

A. Scientific Staff

G. M. Adamson       Consultant
L. Adler            Ohio State University
I. Alexeff          University of Tennessee
W. R. Allen         University of Tennessee
F. W. Averill       Judson College
B. P. Bandypadhyay  University of North Dakota
G. T. Barlow        Consultant
E. S. Bomar, Jr.    Consultant
J. A. M. Boulet     University of Tennessee
R. H. Brown         Luther College
M. Brun             General Electric Corporate Research & Development
<table>
<thead>
<tr>
<th>Name</th>
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<tr>
<td>J. V. Cathcart</td>
<td>Consultant</td>
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<tr>
<td>K. K. Chawla</td>
<td>New Mexico Institute of Mining &amp; Technology</td>
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<td>C. Nishimura</td>
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<td>L. J. Schioler</td>
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<td>P. A. Thevenard</td>
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<td>K. G. Tschersich</td>
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<td>P. Zschack</td>
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**B. Post-Doctoral Program**

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**C. Graduate Students**

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<tr>
<td>D. Behboudi</td>
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<td>Virginia Polytechnic Institute &amp; State University</td>
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<td>A. N. Gubbi</td>
<td>Auburn University</td>
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<td>D. L. Joslin</td>
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<td>T. H. Krukemyer</td>
<td>University of Toledo</td>
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<td>K. S. Leshkivich</td>
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<td>J. J. Liao</td>
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<td>Y. Lin</td>
<td>Auburn University</td>
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<td>B. N. Lucas</td>
<td>University of Tennessee</td>
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<td>K. P. Monar</td>
<td>University of Tennessee</td>
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<td>D. D. Paul</td>
<td>Tennessee Technological University</td>
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<td>J. E. Pawel</td>
<td>Vanderbilt University</td>
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<td>G. R. Rao</td>
<td>Auburn University</td>
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<tr>
<td>P. G. Sanders</td>
<td>Michigan Technological University</td>
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</table>
J. M. Schmitz  University of Tennessee
L. Sneed  Rensselaer Polytechnic Institute
R. A. Vesey  Rensselaer Polytechnic Institute
D. M. Walukas  University of Tennessee
C. A. Wang  Auburn University

D. Undergraduate Students

V. R. Albee  North Carolina A&T State University
N. Chawla  New Mexico Technological University
C. M. Cowan  Stanford University
B. M. Evans  University of Tennessee
T. E. Farrow  Lincoln University
J. A. Francis  Emory and Henry College
J. C. Hooker  Albion College
T. S. Moss  Georgia Institute of Technology
P. C. Sundby  University of Wisconsin
M. B. Tanner  U.S. Naval Academy

E. Science and Engineering Research Semester Program (SERS)

U. K. Abdali  Cornell University
M. B. Chermside  Earlham College
T. A. Dolney  Sam Houston State University
A. J. Gifford  California Polytechnic Institute
C. E. Haberlin  Cornell University
K. M. Keys  Kalamazoo College
M. B. Manie  Arkansas State University
S. C. Martin  Oklahoma Panhandle State University
O. L. G. Medley  Illinois Technical College
E. E. Meyer  Mount Holyoke College
S. J. Miller  Florida Atlantic University
B. J. Reardon  Alfred University
B. R. Shelton  East Tennessee State University
S. M. Vyas  Rice University
D. A. Walko  Cornell University

F. Science Teachers Research Involvement for Vital Education Program (STRIVE)

L. T. Hixson  Cleveland High School
L. E. Long  Lookout Valley School

G. Southeastern University Research Association (SURA)

W. B. Alexander  University of Florida
A. J. Duncan  University of Florida
V. Iyer  Virginia Polytechnic Institute & State University
User Facilities

A. High Temperature Materials Laboratory (HTML)

W. K. Baxter  Coors Electronic Package Company
R. Bhattacharya  Universal Energy Systems
J. Birkbeck  EG&G Mound Applied Technology
D. A. Bowers  McDonnell Douglas Corporation
C. R. Brooks  University of Tennessee
W. D. Cao  Teledyne Alvac
K. Chawla  New Mexico Technological University
J. A. Connally  Massachusetts Institute of Technology
N. B. Dahnre  University of Tennessee Space Institute
B. Davis  University of Kentucky
S. D. Davis  Vanderbilt University
J. E. Denton  Cummins Engine Company
R. J. Dill  Corning Incorporated
L. J. Farthing  Stanford University
M. Foley  Norton Company
G. R. Fox  Penn State University
J. Ghinazzi  Coors Technical Ceramics
D. C. Giles  Detroit Diesel Corporation
P. Gillespie  Rice University
M. Godbole  University of Tennessee
R. Gonzalez  University of Kentucky
S. C. Gopinathan  University of Tennessee Space Institute
L. Groseclose  Allison Gas Turbine
M. Gruijicia  Clemson University
D. S. Harding  Rice University
M. Harris  ReMaxCo
J. R. Hartenstein  Thermacore, Inc.
J. T. Hartman  Allied Signal
A. Haque  Tuskegee University
J. Hefter  GTE Laboratories Incorporated
B. W. Holman  Allison Gas Turbine
H. Hsu  American Superconductor
S. Jeelani  Tuskegee University
A. K. Knudson  Dow Chemical Company
W. R. Lacefield  University of Alabama
S. Lacki  The Carborundum Company
W. J. Lakso  Detroit Diesel Corporation
E. Lara-Curzio  Rensselaer Polytechnic Institute
H. R. Last  Georgia Institute of Technology
M. Loboda  Dow Corning Corporation
L. C. Lucas  University of Alabama
G. H. Ma  North Carolina State University
M. Martin  Cornell University
M. N. Menon  Allied Signal
W. E. Moddeaman  EG&G Mound Applied Technology
D. L. Mohr  Georgia Institute of Technology
C. P. Narayan  Clemson University
R. A. Newman  Dow Chemical Company
J. J. Nick  Allied Signal
J. L. Ong  University of Alabama
D. Ouellette  Ceramics Process Systems
A. E. Pasto  GTE Laboratories Incorporated
T. M. Pattison  Sundstrand Power Systems
G. M. Pharr  Rice University
R. A. Pogge  Vanderbilt University
E. R. Puknys  Vanderbilt University
T. Rachel  Michigan State University
C. A. Randall  Penn State University
J. W. Sapp  McDonnell Douglas Corporation
R. Schartman  University of Wisconsin
A. R. Sethuraman  University of Kentucky
R. Srinivasan  University of Kentucky
G. V. Srinivasen  The Carborundum Company
J. St. Amand  Engelhard Corporation
J. Stencel  University of Kentucky
C. M. Sung  GTE Laboratories Incorporated
S. Tangrila  Clemson University
S. R. Taylor  Teledyne Allvac
H. Wada  University of Michigan
L. Wang  University of Michigan
K. E. Weber  Detroit Diesel Corporation
A. Wereczczak  University of Delaware
P. J. Whalen  Allied Signal
E. D. Winters  Coors Electronic Package Company
J. S. Wolf  Clemson University
H. F. Wu  ALCOA
R. L. Yeckley  Norton Company
D. P. Zadoo  Tuskegee University
8 users  (Proprietary)

B. Shared Research Equipment Program (SHaRE)

I. M. Anderson  University of Minnesota
I. Baker  Dartmouth College
R. Beanland  Ohio State University
M. G. Burke  Westinghouse Science & Technology Center
R. D. Carter, Jr.  University of Michigan
F. Chen  University of California at Los Angeles
B. A. Chin  Auburn University
A. B. Doucet  Louisiana State University
B. D. Fabes  University of Arizona
A. G. Fox  Naval Post Graduate School
H. L. Fraser  Ohio State University  
B. K. Kad  Ohio State University  
G. H. Ma  North Carolina State University  
D. M. Maher  North Carolina State University  
P. K. Nagpal  Dartmouth College  
D. C. Paine  Brown University  
N. L. Petouhoff  University of California at Los Angeles  
J. A. Todd  Illinois Institute of Technology  
T. Y. Tsui  Rice University  
I. Vazquez  University of Arizona  
E. Y. Yankov  Illinois Institute of Technology  
J. Zhang  Lehigh University  
Y. Zhu  Brookhaven National Laboratory  

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

R. Aburano  University of Illinois  
B. Aepli  AT&T Bell Laboratories  
J. Anderson  University of Illinois  
R. Beech  University of Illinois  
M. Blander  Argonne National Laboratory  
T. Bohanon  Northwestern University  
J. Chang  University of Houston  
H. Chen  University of Illinois  
T. C. Chiang  University of Illinois  
P. Dutta  Northwestern University  
J. Eastman  Argonne National Laboratory  
T. Egami  University of Pennsylvania  
J. Enderby  Argonne National Laboratory  
E. Epperson  Argonne National Laboratory  
B. Everitt  University of Illinois  
E. Feng  University of Illinois  
M. Fitzsimmons  Los Alamos National Laboratory  
M. Fradkin  University of Illinois  
I. Fugita  University of Illinois  
H. Hong  University of Illinois  
R. Hu  University of Pennsylvania  
E. D. Isaacs  AT&T Bell Laboratories  
P. Jamian  Argonne National Laboratory  
X. Jiang  University of Houston  
X. Kan  University of Houston  
B. Lin  Northwestern University  
D. S. Lin  University of Illinois  
J. Mikrut  Northwestern University  
S. Moss  University of Houston  
M. Nelson  University of Illinois  
K. Pettit  University of Illinois  
P. Platzman  AT&T Bell Laboratories
<table>
<thead>
<tr>
<th>Name</th>
<th>Affiliation</th>
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<tr>
<td>D. L. Price</td>
<td>Argonne National Laboratory</td>
</tr>
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<td>B. Rabin</td>
<td>Idaho National Engineering Laboratory</td>
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<td>J. L. Robertson</td>
<td>National Institute of Standards &amp; Technology</td>
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<td>D. Rosenfield</td>
<td>University of Pennsylvania</td>
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<td>M. L. Saboungi</td>
<td>Argonne National Laboratory</td>
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<td>M. Salamon</td>
<td>University of Illinois</td>
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<td>T. Sendyka</td>
<td>University of Pennsylvania</td>
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<td>M. C. Shih</td>
<td>Northwestern University</td>
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<td>R. Simmons</td>
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<td>Argonne National Laboratory</td>
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<td>C. Venkataraman</td>
<td>University of Illinois</td>
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<td>Q. Wang</td>
<td>University of Houston</td>
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<td>J. Widom</td>
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<td>X. Yan</td>
<td>University of Pennsylvania</td>
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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 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 in degree 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 received the 1991 ASM International Oak Ridge Chapter Young Member Award.


R. L. BEATTY, B. R. CHILCOAT, C. HAMBY, AND J M ROBBINS received the Outstanding Achievement Award by the Ulysses RTG Team from the United States Department of Energy Office, October 6, 1990.

P. F. BECHER received the Alexander von Humboldt Research Award from the Alexander von Humboldt Research Foundation to conduct research at the Max-Planck-Institute, Stuttgart, for six months.

K. S. BLAKELY received a Martin Marietta Energy Systems Award for the processing of iridium sheets, iron aluminides, and superconducting ribbons and for training staff in the use of KCN and acid-room procedures, May 24, 1991.

S. W. COOK received a Martin Marietta Energy Systems Outstanding Performance Award for operating, upgrading, and maintaining the ORNL Multiple Ion Facility, May 24, 1991.

B. E. FOSTER was awarded a certificate by the Board of Governors of the American Society of Mechanical Engineers for the high regards of his co-workers and appreciation of the society for valued services in advancing the engineering profession.

G. A. GEIST received the 1990 Gordon Bell Award from the Institute of Electrical & Electronic Engineers, February 28, 1991.

G. M. GOODWIN received the William Irrgang Award from the American Welding Society, Detroit, Mich., April 15, 1991.

A. R. HAWKINS received a Martin Marietta Energy Systems Award for providing exceptional secretarial services in the ORNL M&C Division and while on assignment to the ORNL Tiger Team assessment, May 24, 1991.

M. A. JANNEY received a Martin Marietta Energy Systems citation as Inventor of the Year for his development of a "gelcasting" process for near-net-shape fabrication of ceramic components that should lower fabrication costs, May 24, 1991.


P. J. MAZIASZ was awarded the 1989 Significant Contribution Award for the paper entitled, "Microstructural Evolution of Martensitic Steels During Fast Neutron Irradiation," by the Materials Science and Technology Division (MSTD), November 13, 1991.

R. W. MCCLUNG was awarded the C. W. Briggs Award in recognition of continuous and outstanding contributions of an individual for the work of the ASTM Committee sponsored by the American Society for Testing and Materials, January 1991.

P. A. MENCHHOFER received a Martin Marietta Energy Systems Award for the conversion of the patented gelcasting process into a complex extrusion apparatus, May 24, 1991.


B. F. MYERS was awarded the 1990 Best Paper Award for a paper entitled, "Effect of Water Vapor on the Release of Fission Gases from UCO in HTGR Coated Fuel Particles," by the Nuclear Division of the American Ceramic Society.


O. O. OMATETE received a Martin Marietta Energy Systems Award for the development of a novel gelcasting process, which should lower the cost of advanced ceramics fabrication, May 24, 1991.

V. K. SIKKA received the Distinguished Alumnus Award from the faculty of the College of Engineering at the University of Cincinnati, October 23, 1991.

J. O. STIEGLER was awarded the 1991 FLC Award for Excellence in Technology Transfer by the Federal Laboratory Consortium, San Diego, Calif., April 24, 1991.


R. A. STREHLOW was named a member of the International Advisory Group to Infoterm, a UNESCO terminology organization, Corbec, Canada, October 1991.


APPOINTMENTS

J. BENTLEY has been appointed a member of the Department of Energy Review Panel D2 for the University Research Instrumentation Program.

J. BENTLEY has been appointed to the Editorial Board of the Journal of Microscopy Research and Technique.

E. E. BLOOM has been appointed a Fellow by the American Nuclear Society, November 12, 1991.

S. A. DAVID has been appointed Chairman of the Joining Division Council of ASM International to serve from November 1, 1990, to October 31, 1992.


M. L. GROSSBECK has been appointed a Fellow by the American Nuclear Society Board of Directors, June 4, 1991.

R. L. HEESTAND has been appointed a Fellow of the ASM International, October 22, 1991.

G. E. ICE has been appointed to serve on the Advanced Photon Source Users Executive Committee, May 8, 1991.

R. R. JUDKINS has been appointed Chairman of the Coal Utilization Committee, International Gas Turbine Institute of the American Society of Mechanical Engineers, July 1, 1991.

C. T. LIU has been appointed Chairman of the Committee on Alloy Phases, The Metallurgical Society (TMS), 1991-93.

C. T. LIU has been appointed as a Council Member of the Structural Materials Division and Electronic, Magnetic, and Photonic Materials Division, TMS, 1991.


L. K. MANSUR has been appointed vice-chairman of the TMS/ASM Nuclear Materials Committee.

L. K. MANSUR has been appointed guest editor for a special issue of JOM on the effects of radiation in non-metallic materials that was published in July 1991.

M. K. MILLER has been appointed chairman of the editorial board of the International Emission Society.

G. M. SLAUGHTER has been appointed a Fellow of the American Welding Society, April 15, 1991.

R. W. SWINDEMAN has been appointed a Member at Large of the Materials and Structures Technical Group from SAM 1991 to SAM 1992 by the American Society of Mechanical Engineers, July 3, 1991.

P. F. TORTORELLI has been appointed Chairman of the Proposal Review Committee of the ORNL Seed Money Fund, April 1, 1991.

CONFERENCES

Second AIST-NEDO/DOE-HQ Joint Technical Meeting on Materials for Coal Liquefaction, New Orleans, La., October 8-11, 1990

R. R. Judkins, Co-chairman


J. O. Stiegler, Co-chairman

ASU Workshop on Structure and Properties of Interfaces, Wickenburg, Ariz., January 2-6, 1991

L. F. Allard, Session Chair

Applications of Multiple Scattering Theory to Materials Science, Fall 1991, Materials Research Society

W. H. Butler, Symposium Organizer


L. L. Horton, Program Co-chairman


P. F. Tortorelli, Organizer

ASTM Symposium on Standardizing Terminology for Better Communication: Practice, Applied Theory, and Results, Cleveland, Ohio, June 12-14, 1991

R. A. Strehlow, Chairman
Oak Ridge Chapter of ASM International Symposium on image-Based Analysis Techniques, Oak Ridge, Tenn., April 18, 1991

K. B. Alexander, Chairman

Damage Exposure Units for Ferritic Steel Embrittlement Correlation, Workshop held in conjunction with Annual Meeting of International Group on Radiation Damage Mechanisms, Raleigh, N.C., April 21-26, 1991

R. E. Stoller, Organizing Chairman


N. C. Cole, Cochairman
R. R. Judkins, Cochairman


A. Choudhury, Chairman

NATO Advanced Research Workshop on Ordered Intermetallics - Physical Metallurgy and Metallurgy Behavior, Irsee, Germany, June 23-28, 1991

C. T. Liu, Director

49th Annual Meeting of the Electron Microscopy Society of America, San Jose, Calif., August 4-9, 1991

J. Bentley, Program Vice-chairman

Symposium on "Structural Ceramics" at the 1991 Electron Microscopy Society of America Meeting, San Jose, Calif., August 4-9, 1991

K. B. Alexander, Co-chairman
P. S. Sklad, Co-chairman


C. T. Liu, Organizing Co-chairman


A. F. Rowcliffe, Organizing Chairman

MRS 1991 Fall Meeting, Boston, Mass., December 2-6, 1991

M. H. Yoo, Co-chairman
PATENTS ISSUED


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:


A. WOLFENDEN, Mechanical Engineering Department, Texas A&M University, College Station, "Structure-Property Relations in Meltspun Ribbons of FeAl-X Alloys," April 5, 1991.

K. G. TSCHERSICH, KFA, West Germany, "In Situ Investigation of a-C:H Film Growth," April 12, 1991.


A. GOKHALE, Georgia Institute of Technology, Atlanta, "Microstructural Evolution During Thermal Cycling of Metal/Matrix Composites," April 19, 1991.


Appendix E

PUBLICATIONS

Compiled by Donna Walmsley

D. J. ALEXANDER


D. J. ALEXANDER, G. M. GOODWIN, AND E. E. BLOOM

Evaluation of Weldments in Type 21-6-9 Stainless Steel For Compact Ignition of Tokamak Structural Applications - Phase I, ORNL/TM-11739, June 1991.


D. J. ALEXANDER AND V. K. SIKKA


K. B. ALEXANDER, P. ANGELINI, AND P. F. BECHER

K. B. ALEXANDER AND P. F. BECHER


K. B. ALEXANDER, D. M. KROEGER, J. BENTLEY, AND J. BRYNESTAD


L. F. ALLARD AND T. A. NOLAN


W. R. ALLEN


J. D. ALLEN, C. V. DODD, J. R. PATE, AND F. M. SCHELL


W. R. ALLEN AND M. B. LEWIS


W. R. ALLEN AND D. F. PEDRAZA


B. K. ANNIA, E. D. SPECHT, N. THEOPHILOU, AND A. G. MACDIARMID

C. E. BAMBERGER, L. F. ALLARD, AND D. W. COFFEY

P. F. BECHER

P. F. BECHER, P. ANGELINI, C. H. HSUEH, AND T. N. TIEGS

P. F. BECHER, E. L. FULLER, JR., AND P. ANGELINI

P. F. BECHER AND H. T. LIN


J. BENTLEY, A. T. FISHER, E. A. KENIK, AND Z. L. WANG

J. BENTLEY, E. A. KENIK, K. B. ALEXANDER, Z. L. WANG, AND A. T. FISHER

J. BENTLEY AND P. S. SKLAD

T. M. BESMANN AND I. ABDEL-LATIF
T. M. BESMANN, B. W. SHELDON, AND M. D. KASTER

H. K. D. H. BHADESHIA, S. A. DAVID, AND J. M. VITEK


P. J. BLAU

A. BLEIER

A. BLEIER AND C. G. WESTMORELAND

E. E. BLOOM

C. R. BRINKMAN, D. J. ALEXANDER, AND P. J. MAZIASZ

C. R. BRINKMAN, J. L. DING, AND M. K. BOOKER

R. A. BUHL

T. D. BURCHELL AND W. P. EATHERLY

M. G. BURKE, S. P. GRANT, AND M. K. MILLER

M. G. BURKE AND M. K. MILLER

M. G. BURKE AND M. K. MILLER

W. H. BUTLER AND X. G. ZHANG

R. W. CAHN, M. TAKEYAMA, J. A. HORTON, AND C. T. LIU

P. T. CARLSON

J. B. O. CAUGHMAN, D. N. PUZIC, D. J. HOFFMAN, R. A. LANGLEY, AND M. B. LEWIS

A. CEREZO, M. G. HETHERINGTON, J. M. HYDE, AND M. K. MILLER
A. CEREZO, M. G. HETHERINGTON, J. M. HYDE, AND M. K. MILLER

A. CEREZO AND M. K. MILLER


A. CHAUDHURY, R. A. PADGETT, AND C. R. BROOKS

A. CHAUDHURY, C. L. WHITE, AND C. R. BROOKS


R. E. CLAUSING, L. HEATHERLY, E. D. SPECHT, AND K. L. MORE

W. R. CORWIN, R. K. NANSTAD, AND S. K. ISKANDER


S. A. DAVID


S. A. DAVID


S. A. DAVID AND M. L. SANTELLA


S. A. DAVID AND J. M. VITEK

*BES Welding Science Newsletter (No. 5)*, Materials Joining Group, Oak Ridge National Laboratory, Oak Ridge, Tennessee, November 1990.

S. A. DAVID, J. M. VITEK, AND T. ZACHARIA


N. D. EVANS, S. J. ZINKLE, J. BENTLEY, AND E. A. KENIK


B. D. FABES AND W. C. OLIVER


J. I. FEDERER


J. I. FEDERER, H. E. KIM, AND A. J. MOORHEAD

M. K. Ferber, M. G. Jenkins, T. A. Nolan, and R. Yeckley

A. J. Freeman, C. Li, and C. L. Fu

C. L. Fu

C. L. Fu and G. S. Painter

C. L. Fu and M. H. Yoo

C. L. Fu and M. H. Yoo

E. L. Fuller, Jr. and N. R. Smyrl

E. P. George and C. T. Liu

E. P. George, C. T. Liu, and J. J. Liao
E. P. GEORGE, D. P. POPE, C. L. FU, AND J. H. SCHNEIBEL

E. P. GEORGE, C. L. WHITE, AND J. A. HORTON

R. S. GRAVES, T. G. KOLLIE, D. L. MCELROY, AND K. E. GILCHRIST


M. L. GROSSBECK

M. L. GROSSBECK, K. EHRLICH, AND C. WASSILEW

M. L. GROSSBECK AND L. K. MANSUR

B. L. GYORFFY, A. BARBIERI, J. B. STAUNTON, W. A. SHELTON, AND G. M. STOCKS

B. L. GYORFFY, J. B. STAUNTON, AND G. M. STOCKS

F. M. HAGGAG, W. R. CORWIN, AND R. K. NANSTAD

S. HAMADA, M. SUZUKI, P. J. MAZIASZ, AND A. HISHINUMA

J. A. HANIGOFSKY, K. L. MORE, W. J. LACKEY, W. Y. LEE, AND G. B. FREEMAN

M. G. HETHERINGTON AND M. K. MILLER

M. G. HETHERINGTON AND M. K. MILLER

M. G. HETHERINGTON, J. M. HYDE, AND M. K. MILLER

M. G. HETHERINGTON, J. M. HYDE, M. K. MILLER, AND G. D. W. SMITH

D. O. HOBSON

S. C. HONG, A. J. FREEMAN, AND C. L. FU

J. A. HORAK, M. M. PAXTON, AND L. K. EGNER

J. A. HORTON, I. BAKER, AND M. H. YOO

J. A. HORTON, C. T. LIU, AND S. J. PENNYCOOK
C. H. HSUEH

C. H. HSUEH

C. H. HSUEH

C. H. HSUEH

C. H. HSUEH

C. H. HSUEH AND P. F. BECHER

C. H. HSUEH AND P. F. BECHER

C. H. HSUEH, P. F. BECHER, AND W. J. LACKEY

C. H. HSUEH, J. D. BRIGHT, AND D. K. SHETTY


T. INAZUMI, G. E. C. BELL, AND K. KIUCHI
S. K. ISKANDER, W. R. CORWIN, AND R. K. NANSTAD
Results of Crack-Arrest Tests on Two Irradiated High-Copper Welds, ORNL/TM-11575,

S. K. ISKANDER, G. C. ROBINSON, AND C. B. OLAND

R. JAYARAM, J. J. HREN, AND M. K. MILLER
"APFIM/AEM Characterization of Solute Partitioning in a Model Ni-Al-Mo-Ta

D. D. JOHNSON, D. M. NICHOLSON, F. J. PINSKI, B. L. GYORFFY, AND G. M. STOCKS

R. R. JUDKINS
Fossil Energy Program Semiannual Progress Report for October 1989 Through

R. R. JUDKINS AND P. T. CARLSON, COMP.
Fossil Energy Advanced Research and Technology Development Materials Program
Semiannual Progress Report for the Period Ending September 30, 1990,
ORNL/FMP-90/2, December 1990.

R. R. JUDKINS AND N. C. COLE
Proceedings of the Fifth Annual Conference on Fossil Energy Materials,

E. A. KENIK
"Loss of Grain-Boundary Segregant During Ion Milling," J. Electron Microsc. Tech. 18,

E. A. KENIK
"X-ray Microanalysis of Phosphorus Segregation in Type 304L Stainless Steels,"

E. A. KENIK AND J. BENTLEY
"Influence of Tilt Angle on Hole Count and Secondary Fluorescence in X-ray

E. A. KENIK, J. BENTLEY, AND N. D. EVANS
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B. L. KEYES

H. E. KIM AND A. J. MOORHEAD

H. E. KIM AND A. J. MOORHEAD

H. E. KIM AND A. J. MOORHEAD

H. E. KIM, H. D. KIMREY, AND D. J. KIM

H. E. KIM, S. J. ZINKLE, AND W. R. ALLEN

R. L. KLUEH

R. L. KLUEH AND D. J. ALEXANDER
R. L. KLUEH, P. J. MAZIASZ, AND D. J. ALEXANDER

R. L. KLUEH AND J. M. VITEK

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F. W. KUTZLER AND G. S. PAINTER

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R. J. LAUF AND C. HAMBY

R. J. LAUF AND B. S. HOFFHEINS

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H. T. Lin AND B. A. Chin

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C. G. McKamey and C. A. Carmichael

C. G. McKamey, J. H. Devan, P. F. Tortorelli, and V. K. Sikka

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C. NISHIMURA AND C. T. LIU


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J. E. PAWEL
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"Electrical Resistance of Metallic Contacts on Silicon and Germanium During

G. M. PHARR, W. C. OLIVER, AND D. S. HARDING
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A. F. ROWCLIFFE, A. HISHINUMA, M. L. GROSSBECK, AND S. JITSUKAWA
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B. W. SHELDON AND T. M. BESMANN

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R. E. STOLLER

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R. W. SWINDEMAN
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R. W. SWINDEMAN AND P. J. MAZIASZ

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M. SUZUKI, T. SAWAI, P. J. MAZIASZ, AND A. HISHINUMA

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P. F. TORTORELLI

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Z. L. WANG AND J. BENTLEY

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A. C. YOUNG, O. O. OMATETE, M. A. JANNEY, AND P. A. MENCHHOFER

H. ZABEL, N. LUCAS, H. MORKOC, AND C. J. SPARKS

T. ZACHARIA, S. A. DAVID, AND J. M. VITEK

T. ZACHARIA, S. A. DAVID, J. M. VITEK, AND H. G. KRAUS
B. ZHOU, S. L. YU, Y. T. CHOU, AND C. T. LIU

S. J. ZINKLE, K. FARRELL, AND H. KANAZAWA
Appendix F

PRESENTATIONS AT TECHNICAL MEETINGS

Compiled by Donna Walmsley

Materials Sciences Division Program Review (DOE), Argonne National Laboratory, Argonne, Illinois, October 1-2, 1990:


U.S./Japan Workshop on Low-Temperature Structural Materials, Vail, Colorado, October 1-2, 1990:

D. J. ALEXANDER* AND G. M. GOODWIN, "Evaluation of Weldments in Type 21-6-9 Stainless Steel"

Polyurethanes '90 Conference, Orlando, Florida, October 1-4, 1990:


37th Sagamore Conference on Structural Ceramics, Plymouth, Massachusetts, October 1-4, 1990:

G. M. CROSBIE,* D. M. NICHOLSON, AND R. L. PREDMESKY, "Synthesis of Silicon Nitride for Structural Ceramics"

M. K. FERBER,* M. G. JENKINS, AND V. J. TENNERY, "Comparison of Tension, Compression, and Flexure Creep for Alumina and Silicon Nitride Ceramics"

*Speaker.
O. O. OMATETE* AND R. A. STREHLOW, "Gelcasting of Sub-Micron Alumina, Sialon, and Silicon Nitride Powders"

Stanford Synchrotron Radiation Lab Users' Meeting, Stanford, California, October 2, 1990:
G. E. ICE, "X-Ray Resonant Magnetic and Resonant Raman Studies of Heat Fermion Systems"

Seminar at the University of Rochester, Rochester, New York, October 3, 1990:
T. M. BESMANN, "Vapor Phase Fabrication of Ceramic Coatings and Composites"

Steering Committee Meeting for the Cooperative Industry/Government Project on Alternative CFC Blowing Agents, Orlando, Florida, October 4, 1990:

16th MPA-Seminar, Stuttgart, Federal Republic of Germany, October 4-5, 1990:
R. K. NANSTAD, S. K. ISKANDER, F. M. HAGGAG, AND T. L. DICKSON,* "Effects of Irradiation on Initiation and Crack-Arrest Toughness of Two High-Copper Welds and on Stainless Steel Cladding"

STLE Annual Tribology Conference, Toronto, Canada, October 7-10, 1990:
P. J. BLAU, "Scale Effects in Steady-State Friction"
C. S. YUST* AND C. E. DEVORE, "The Friction and Wear of Lubricated Si₃N₄/SiC(w) Composites"

Joint U.S./Japan Meeting on Coal Liquefaction, New Orleans, Louisiana, October 7-11, 1990:
B. Z. EGAN, D. E. FAIN, AND R. R. JUDKINS,* "Development of Inorganic Membranes for Gas Separation"

1990 Fall TMS/ASM Symposium, Detroit, Michigan, October 7-11, 1990:
D. J. ALEXANDER, "High-Temperature Fracture Toughness of a Nickel Aluminide Alloy"
P. F. BECHER, "Toughening Brittle Matrices by Discontinuous Brittle Reinforcements"
S. A. DAVID, "Weldability of Intermetallic Alloys"
C. L. FU,* G. S. PAINTER, AND M. H. YOO, "The Role of Electronic Structure in the Mechanical Behavior of Aluminides"
E. P. GEORGE* AND C. T. LIU, "Critical Evaluation of the Ductilizing Effect of Boron in Ni$_3$Al"

G. M. GOODWIN, "Hot Cracking: Measurement, Mechanisms, and Modeling"

E. H. LEE* and L. K. MANSUR, "Relationship Between Phase Stability and Void Swelling in Fe-Ni-Cr Alloys During Irradiation"

J. K. LEE AND M. H. YOO,* "Bifurcated Nucleation Barrier Due to Coherency Strain"

C. T. LIU* AND E. P. GEORGE, "Environmental Embrittlement in Iron Aluminides (Fe$_3$Al and FeAl)"

D. F. PEDRAZA* AND J. A. CARO, "Irradiation-Induced Point Defects in NiAl"

V. K. SIKKA, "Near-Net Shaping of Nickel and Iron Aluminides"

C. J. SPARKS* AND G. E. ICE, "Local Atomic Arrangements and Their Affect on Materials Properties"

S. SPOONER, S. A. DAVID,* T. M. HOLDEN, AND J. H. ROOT, "Investigation of Strain Distributions in a One-Inch-Thick Stainless Steel Plate Containing a Multiple-Pass Weld"

P. F. TORTORELLI, "Corrosion of Aluminides by Molten Nitrate Salt"

M. H. YOO, "Internal Sources and Multiplication Mechanisms of Superdislocations in Ordered Intermetallic Alloys"

9th Topical Meeting on the Technology of Fusion Energy, Oak Brook, Illinois, October 7-11, 1990:


Technology Transfer Opportunities Materials Week 1990, ASM International, Detroit, Michigan, October 8-11, 1990:

K. C. LIU* AND H. PIH, "Static and Dynamic Strain Measurements in Hostile Environments Using Laser Diffraction Techniques"

37th Annual Meeting of the American Vacuum Society Mechanical Behavior of Thin Films, Toronto, Canada, October 8-12, 1990:

W. C. OLIVER, "The Development and Use of the Mechanical Properties Microprobe"
The AIST-NEDO/DOE-PETC Joint Technical Meeting, New Orleans, Louisiana, October 11, 1990:

R. R. JUDKINS,* B. Z. EGAN, AND D. E. FAIN, "Development of Inorganic Membranes for Gas Separation"

Eleventh International Conference on Chemical Vapor Deposition (CVD-XI), Seattle, Washington, October 14-19, 1990:

T. M. BESMANN, "Chemical Vapor Infiltration"


B. W. SHELTON AND T. M. BESMANN,* "The Nucleation and Growth of Polycrystalline Silicon Carbide During Chemical Vapor Deposition"

C. VAHLAS AND T. M. BESMANN,* "Thermodynamic Approach to the CVD of the YBa2Cu3O7-x Phase Deposited from Organometallics"

Appalachian Regional Electron Microscopy Society, Kingsport, Tennessee, October 19, 1990:

M. A. SCHMIDT, "Recent Experience with a Confocal Tandem-Scanning Microscope"

ASME Steam Power Conference, Boston, Massachusetts, October 21-25, 1990:

C. R. BRINKMAN,* D. J. ALEXANDER, AND P. J. MAZIASZ, "Modified 9Cr-1Mo Steel (T-91) for Advanced Steam Generator Applications"

Werkstoffwissenschaftliches Kolloquium, Max-Planck-Institut, Stuttgart, October 22, 1990:

J. H. SCHNEIBEL, "Mechanisms of Deformation of the Intermetallic Phases Ni3Al and Al3Sc"

18th Water Reactor Safety Information Meeting, Rockville, Maryland, October 22-24, 1990:

W. R. CORWIN,* R. K. NANSTAD, S. K. ISKANDER, F. M. HAGGAG, AND D. E. MCCABE, "Heavy-Section Steel Irradiation Program Overview"

C. V. DODD* AND J. R. PATE, "Improved Eddy-Current Inspection for Steam Generator Tubing"
1990 Annual Automotive Technology Development Contractors Coordination Meeting, Dearborn, Michigan, October 22-25, 1990:


O. O. OMATETE,* R. A. STREHLOW, AND B. L. ARMSTRONG, "Forming of Silicon Nitride by Gelcasting"

V. J. TENNERY* AND M. K. FERBER, "Cooperative International Program on Mechanical Strength Measurements of Ceramics"

ORAU Traveling Lecture, University of New Mexico, Albuquerque, New Mexico, October 23, 1990:

L. F. ALLARD, "High-Resolution Electron Microscopy of BN on MgO"

43rd Pacific Coast Meeting of the American Ceramic Society Meeting, Seattle, Washington, October 25-27, 1990:

M. G. JENKINS,* M. K. FERBER, J. A. SALEM, AND A. GHOSH, "Increased Fracture Resistance Due to Residual Stresses in a Particulate Ceramic Composite"

T. N. TIEGS,* J. O. KIGGANS, H. D. KIMREY, AND C. E. HOLCOMBE, "Microwave Processing of Reaction-Bonded Silicon Nitride"

International Forum on Tribology of Advanced Ceramics, Tokyo, Japan, October 26, 1990:

C. S. YUST, "Evaluation of Test Duration Effects on the Mild-to-Severe Wear Transition in Ceramics"

Japan International Tribology Conference, Nagoya, Japan, October 29-November 1, 1990:

C. S. YUST, "Wear of Advanced Ceramics: An Overview"

35th Annual Conference on Magnetism and Magnetic Materials, San Diego, California, October 29-November 1, 1990:

R. KUMAR,* C. J. SPARKS, AND W. B. YELON, "New Results on Site Occupations — Implications on Phase Stability"

Seminar at the Department des Materiaux, Ecole Polytechnique Federale de Lausanne, Switzerland, October 30, 1990:

J. H. SCHNEIBEL, "Deformation Mechanisms of the Intermetallic L₁₂-Phases Ni₃Al and Al₅Sc"
11th International Conference on the Application of Accelerators in Research and Industry, Denton, Texas, November 4-8, 1990:

W. R. ALLEN,* M. B. LEWIS, AND L. K. MANSUR, "The Lattice Site of Helium in Ceramic Oxides"

9th International Congress on Applications of Lasers and Electro-Optics, Boston, Massachusetts, November 4-9, 1990:

N. B. DAHOTRE, M. H. MCCAY, T. D. MCCAY, C. M. SHARP, S. GOPINATHAN, AND L. F. ALLARD,* "Laser Welding of a SiC/Al-Alloy Metal Matrix Composite"

Conference on Fire, Safety, and Thermal Insulation, St. Petersburg, Florida, November 5-6, 1990:

R. S. GRAVES* AND D. W. YARBROUGH, "The Effect of Compression on the Material R-Value of Fiberglass Batt Insulation"

American Physical Society Meeting, 11th Conference on the Application of Accelerations in Research & Industry, Denton, Texas, November 5-8, 1990:

R. A. BUHL, "Data Acquisition and Control for the Triple Ion-Irradiation Facility"

Werkstoffwissenschaftliches Kolloquium, Universitat Erlangen-Nurnberg, FRG, November 6, 1990:

J. H. SCHNEIBEL, "Verformungsmechanismen Intermetallischer L12-Phasen am Beispiel von Ni3Al und Al3Sc"

The International Congress on Technology and Technology Exchange, Champion, Pennsylvania, November 6-8, 1990:


Building Thermal Envelope Coordinating Council Symposium, Washington, D.C., November 7, 1990:

R. S. GRAVES, "CFC Alternatives for Closed-Cell Foam Insulation"

Industry/Government Briefing, Oak Ridge National Laboratory, Oak Ridge, Tennessee, November 9, 1990:

T. M. BESMANN,* B. W. SHELDON, AND T. STARR, "Processing Science for Chemical Vapor Infiltration"
Symposium on Ceramic, Polymer, and Metal Matrix Composites, Orlando, Florida, November 12, 1990:

L. L. SNEAD,* S. J. ZINKLE, AND D. STEINER, "Interfacial Push-Out Measurements of Fully Bonded SiC/SiC Composites"

Second International Ceramic Science and Technology Congress, Orlando, Florida, November 12-15, 1990:

K. B. ALEXANDER,* P. ANGELINI, AND P. F. BECHER, "Effect of Whisker Surface Treatments and Processing Conditions on the SiC/Al₂O₃ Interface"


P. F. BECHER, "Reinforced Ceramics Employing Discontinuous Phases"

T. M. BESMANN,* R. A. LOWDEN, B. W. SHELDON, AND D. P. STINTON, "Chemical Vapor Infiltration"

N. D. CORBIN,* K. N. SIEBEIN, AND R. A. PADGETT, "Interfaces in SiC Whisker-Reinforced Si₃N₄ Composites"


C. H. HSUEH, "Stress-Displacement Relation During Fiber Pullout"


D. J. KIM* AND D. M. KROEGER, "Optimization of Critical Current Density of Bulk YBCO Superconductor Prepared by Coprecipitation in Oxalic Acid"


Damage Detection and Quality Assurance in Composites, San Antonio, Texas, November 13-14, 1990:

W. A. SIMPSON, JR., AND R. W. MCCLUNG,* "Ultrasonic Detection of Fatigue Damage in Glass-Epoxy Composites"
STLE Symposium on Wear Testing of Advanced Materials, San Antonio, Texas, November 14, 1990:

P. J. BLAU* and C. S. YUST, "Sliding Wear-Testing and Data Analysis Strategies for Advanced Engineering Ceramics"

ASTM Committee C-28, Advanced Ceramics for the Probabilistic Procedures Task Group, San Antonio, Texas, November 14-16, 1990:


V. J. TENNERY, "Recent Observations of Unexplained Variations in Measured $\alpha$ and $\beta$ Parameters of 30 Specimen Data Sets of GN-10 Silicon Nitride - IEA Annex II, Subtask 5 - Room Temperature"

Department of Materials Engineering, Virginia Polytechnic Institute and State University, Blacksburg, Virginia, November 16, 1990:


ORAU Traveling Lecture, University of Michigan, Ann Arbor, Michigan, November 16, 1990:

L. F. ALLARD, "High-Resolution Electron Microscopy of BN on MnO"

Seminar at the University of Houston, Houston, Texas, November 16, 1990:


Graduate Seminar, Department of Mechanical and Aerospace Engineering, University of Tennessee, Knoxville, Tennessee, November 20, 1990:

J. E. PAWEL, "Cladding Corrosion Studies for the Advanced Neutron Source"

Modelling of Sintering Processes, Bad Honnef, FRG, November 26-28, 1990:

J. H. SCHNEIBEL, "Modelling of Grain-Boundary Mass Transport"

Materials Research Society Fall Meeting, Boston, Massachusetts, November 26-December 1, 1990:

A. CEREZO,* M. G. HETHERINGTON, J. M. HYDE, AND M. K. MILLER, "Spinodal Decomposition in the Fe-Cr System"

F. CHEN, A. J. ARDELL,* AND D. F. PEDRAZA, "Miniaturized Disk-Bend Testing of 3.8 MeV Zr$^{2+}$ Irradiated and Unirradiated Zr$_3$Al"

C. L. FU* AND M. H. YOO, "First-Principles Theory of Mechanical Behavior of B2-Type Aluminides"


E. P. GEORGE* AND C. T. LIU, "Environmental Embrittlement and Grain-Boundary Chemistry of MoSi₂-Based Materials"

J. A. HORTON* AND C. T. LIU, "Grain-Boundary Structure as a Function of Aluminum Level in Ni₃Al"

J. A. HORTON* AND Z. L. WANG, "On Charge Density Determination in Intermetallics by Convergent Beam Electron Diffraction"


C. T. LIU* AND E. P. GEORGE, "Effect of Aluminum Concentration and Boron Dopant on Environmental Embrittlement in FeAl Aluminides"

C. G. MCKAMEY* AND C. A. CARMICHAEL, "Microstructure and Mechanical Properties of Ni₃Al-Based Alloys Reinforced"

A. R. MIRSHAMS,* R. H. BALDWIN, AND V. K. SIKKA, "High-Temperature Ductility of Intermetallic Nickel Aluminide (IC-218LZr)"

D. C. PAINE,* D. J. HOWARD, N. D. EVANS, D. W. GREVE, M. RACANELLI, AND N. G. STOFFEL, "In Situ TEM Studies of the Growth of Strained Si₁ₓGeₓ by Solid Phase Epitaxy"

D. F. PEDRAZA,* A. J. CARO, AND D. FARKAS, "The Stability of Irradiation-Induced Defects in Ni₃Al"
J. H. SCHNEIBEL AND P. M. HAZZLEDINE,* "The Crystallography of Cleavage Fracture in Al₃Sc"

B. W. SHELDON* AND T. M. BESMANN, "The Nucleation and Growth of Polycrystalline Silicon Carbide"

V. K. SIKKA, "Production of Fe₃Al-Based Intermetallic Alloys"


P. S. SKLAD AND J. BENTLEY,* "Domain Boundaries Formed During Epitactical Growth of α-Al₂O₃"


F. J. WALKER,* E. D. SPECHT, AND R. A. MCKEE, "Anomalous X-ray Scattering from a Buried Copper-Silicon (111) Interface"

Z. L. WANG* AND J. BENTLEY, "In Situ Observations of High-Temperature Surface Processes on (α)-Alumina Bulk Crystals"

N. F. WRIGHT* AND G. S. PAINTER, "Interatomic Potentials for MgO Based Upon First-Principles Quantum Cluster Calculations"

M. H. YOO,* C. L. FU, AND J. K. LEE, "The Role of Twinning in Deformation and Fracture of Ti-Aluminides"

Department Seminar at Lehigh University, Bethlehem, Pennsylvania, November 27, 1990:

P. J. BLAU, "Friction and Wear Transitions in Coated Materials"

Neutron Source Workshop, Argonne National Laboratory, Argonne, Illinois, November 27-28, 1990:

F. W. WIFFEN, "Japan's ESNIT Program"

Advanced Heat Exchanger Conference, Dallas, Texas, November 29, 1990:

Materials Characterization Group, GTE Laboratories, Waltham, Massachusetts, November 30, 1990:

C. R. HUBBARD, "The High Temperature Materials Laboratory User Program and X-ray Diffraction and Thermophysical Properties of Selected Commercial and Experimental Silicon Nitrides"

Tennessee Technological University, Cookeville, Tennessee, November 30, 1990:

R. K. WILLIAMS, "Oxidation-Induced Decomposition of YBa$_2$Cu$_3$O$_{7-x}$"

Joint VTT-ORNL Seminar on the Structural Safety Assessment of Nuclear Power Plant Components, Oak Ridge National Laboratory, Oak Ridge, Tennessee, December 3-6, 1990:

W. R. CORWIN, "Heavy-Section Steel Irradiation Program Overview"


D. E. MCCABE,* R. K. NANSTAD, A. R. ROSENFIELD, AND G. R. IRWIN, "Investigation of the Bases for Use of the $K_c$ Curve to Regulate Pressure/Temperature Limits During Ltop Conditions"

R. K. NANSTAD,* D. E. MCCABE, F. M. HAGGAG, K. O. BOWMAN, AND D. J. DOWNING, "Statistical Analyses of Fracture Toughness Results for Two Irradiated High-Copper Welds"

Japan/U.S. Workshop P165 on Critical Topics of Plasma-Facing Materials/Plasma-Facing Components Data for the Next Step Fusion Devices, National Institute for Plasma Science, Nagoya, Japan, December 3-7, 1990:

T. D. BURCHELL, "Neutron Damage Effects on Carbon-Based Plasma-Facing Materials"

University of Saarbrucken, Materials Science Department, Stuttgart, Germany, December 11, 1990:

J. H. SCHNEIBEL, "Grain-Boundary Sliding and Cobel Creep"

ASU Workshop on Structure and Properties of Interfaces, Wickenburg, Arizona, January 2-6, 1991:


Oak Ridge Chapter Meeting of ASME, Knoxville, Tennessee, January 3, 1991:

V. K. SIKKA* AND C. G. MCKAMEY, "Production of Fe$_3$Al-Based Intermetallic Alloys"
15th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Florida, January 13-16, 1991:

B. BALLARD, P. PREDECKI, AND C. R. HUBBARD,* "Residual Strains in Al₂O₃/SiC Composite from 25-1000°C"

M. K. FERBER* AND T. N. TIEGS, "Effect of Post-Sintering Microwave Treatments Upon the Mechanical Performance of Silicon Nitride"

W. S. HONG,* J. M. SATER, M. A. RIGDON, AND M. G. JENKINS, "The Effect of Varying Test Parameters on the Measured High-Temperature Strength of SCS-6 Fiber"

R. A. LOWDEN,* R. H. KRABILL, AND M. K. FERBER, "The Use of Fiber Coatings to Control the Mechanical Properties of Fiber-Reinforced Ceramic Composites"

K. L. MORE, D. A. KOESTER, AND R. F. DAVIS,* "Creep of an SiC Whisker-Reinforced Si₃N₄ Composite in Compression and Bending"

T. A. NOLAN,* M. K. FERBER, AND D. W. COFFEY, "Microstructural Characterization of Creep Deformation and Fatigue in an Si₃N₄ Ceramic"

O. O. OMATETE,* A. C. YOUNG, AND T. N. TIEGS, "Gelcast Reaction-Bonded Silicon Nitride Composites"

B. W. SHELDON* AND T. M. BESMANN, "Minimizing Densification Times During Composite Fabrication by Isothermal Chemical Vapor Infiltration"

D. P. STINTON,* B. W. SHELDON, AND J. I. FEDERER, "Corrosion of SiC-Matrix Composites"

T. N. TIEGS,* J. O. KIGGANS, AND H. D. KIMREY, "Microwave-Sintered Silicon Nitride"

A. C. YOUNG, O. O. OMATETE*, A. BLEIER, AND C. G. WESTMORELAND, "Gelcast Zirconia-Aluminia Composites"

International Panel on 14-MeV Intense Neutron Source Based on Accelerators of Fusion Materials Studies, University of Tokyo, Tokyo, Japan, January 14-16, 1991:

F. W. WIFFEN* AND A. F. ROWCLIFFE, "User-Developed Criteria for a Neutron Source"

Association for Excellence in Reactor Operations, Aiken, South Carolina, January 16, 1991:

K. FARRELL,* "New Findings in Neutron Radiation Effects in Aluminium Alloys"
Seminar at the Max-Planck-Institut fur Metallforschung, W-4000 Düsseldorf, Germany, January 16, 1991:

J. H. SCHNEIBEL, "Mechanical Properties of L1₂ Phases"

F Z Zulich, Institut fur Festkörperphysik, Stuttgart, Germany, January 17, 1991:

J. H. SCHNEIBEL, "Grain-Boundary Sliding and Coble Creep"

Seminar Presentation, Max-Planck-Institute, Stuttgart, Germany, January 23, 1991:

J. H. SCHNEIBEL, "Grain-Boundary Sliding and Diffusional Creep"

IEA Executive Committee Meeting, Cocoa Beach, Florida, January 17, 1991:


Seminar, Argonne National Laboratory, Argonne, Illinois, January 24, 1991:

G. M. STOCKS, "Parallelization of Electronic Structure Algorithms"

Seminar on DOE's Technology Transfer Program, Washington, D.C., January 24, 1991:

R. R. JUDKINS* AND P. T. CARLSON, "Thermomechanical Model Software Development Center (TMSDC), A Technology Transfer Initiative"

Defense Advanced Research Projects Agency (DARPA) Workshop in "HTS Bulk Technology Development" in Albuquerque, New Mexico, January 31-February 1, 1991:

D. M. KROEGER, "Microstructure and Jc in Textured Bi-2212 and Y123"

Steering Committee Meeting for the Cooperative Industry/Government Project on Alternative CFC Blowing Agents, Orlando, Florida, February 4, 1991:


Metallphysikalisches Seminar, WS 1990/91, Gottingen, Germany, February 5, 1991:

J. H. SCHNEIBEL, "Mechanical Properties of L1₂ Phases"

Golden Gate Materials Conference, San Mateo, California, February 5-7, 1991:

F. M. HAGGAG, "In Situ Material Mechanical Properties Measurement"

V. K. SIKKA, "Commercialization of Ductile Ni₃Al- and Fe₃Al-Based Intermetallics"
Seminar at North Carolina State University, Raleigh, North Carolina, February 7, 1991:

C. T. LIU, "Recent Advances in Ordered Intermetallics"

Bettis Atomic Power Laboratory, Pittsburgh, Pennsylvania, February 8, 1991:

E. A. KENIK, "Radiation-Induced Grain-Boundary Segregation and Sensitization of a Neutron-Irradiated Austenitic Stainless Steel"

ASM Chapter Meeting, Aiken, South Carolina, February 12, 1991:

J. R. WEIR, JR., "Technology Transfer: A New ASM Committee and the Technology Transfer Process at Oak Ridge"

1991 TMS/AIME Annual Meeting, New Orleans, Louisiana, February 17-21, 1991:

J. BENTLEY, "Application of Transmission EELS to Ceramics and Catalysts"

N. D. EVANS* AND S. ZINKLE, "The Identification of Aluminum in Al+ Implanted MgAl2O4 Spinel by Parallel Electron Energy Loss Spectroscopy"

P. M. HAZZLEDINE* AND J. H. SCHNEIBEL, "Computer Simulation of Coble Creep"

P. M. HAZZLEDINE* AND Y. Q. SUN, "Deformation Mechanisms in U2 Alloys"

E. A. KENIK AND J. BENTLEY* (SPEAKER ONLY), "Application of Electron Energy Loss Spectroscopy to Microanalysis of Irradiated Stainless Steel"

C. R. KENNEDY, "Neutron Irradiation Dimension Changes and Life Expectancy of Graphites"

D. M. KROEGER,* F. A. LIST, K. M. DOVERSPIKE, AND J. BRYNEWAD, "Processing of Thick Superconducting Deposits"

E. H. LEE, "Ion Implantation as a Tool for the Improvement of Surface Mechanical Properties of Polymers"

C. J. MCHARGE, "Ion-Implantation Damage in Ceramics"

W. C. OLIVER, "Mechanical Properties of Radiation-Damaged Ceramics"

D. F. PEDRAZA,* W. R. ALLEN, AND L. ROMAN, "High-Energy Zirconium Implantation in Sapphire"

S. J. ZINKLE, "Ion-Beam Irradiation of Ceramics at Fusion-Relevant Conditions"
Clinton Elementary School (4th Grade Class), Clinton, Tennessee, February 19, 1991:

K. F. RUSSELL, "Catch a Falling Star - A Study of Four Meteorites"

WATTec Conference, Knoxville, Tennessee, February 19-22, 1991:

L. F. ALLARD, "High-Resolution Electron Microscopy in Materials Science"

A. CHOU DHURY, "Secondary Ion Mass Spectrometry and Its Applications to Materials Science"

D. F. CRAIG, "Impact of Critical Trends in Materials Technologies on the Technical Professional Ceramics"

C. V. DODD, "Data Analysis for Eddy-Current Steam Generator Inspections"

R. W. MCCLUNG, "NDT Advances the Competitiveness of Ceramics"

J. R. WEIR, JR., "New Materials Technology"

Seminar at National Research Institute for Metals, Tokyo, Japan, February 27, 1991:

C. T. LIU, "Recent Advances in Ordered Intermetallic Alloys"

Materials Science & Engineering Colloquium, Cornell University, Ithaca, New York, February 28, 1991:

J. BENTLEY, "High Spatial Resolution Analytical Electron Microscopy of Materials"

Review of ATW Work Proposal, Los Alamos, New Mexico, February 28, 1991:

R. L. KLUEH, "Irradiation Damage Studies for Accelerator Transmutation of Waste"

Westinghouse Science and Technology Center Interdepartmental Colloquium, Pittsburgh, Pennsylvania, March 1, 1991:

J. BENTLEY, "Analytical Electron Microscopy of Materials"

Blow-in-Blanket Contractors Association (BIBCA) Seminar, Miami, Florida, March 3-10, 1991:

R. S. GRAVES* AND D. W. YARBROUGH, "The Effect of Compression on the Material R-Value of Fiberglass Batt Insulation"

Power Metallurgy in Aerospace and Defense Technologies Symposium, Tampa, Florida, March 4-6, 1991:

V. K. SIKKA, E. K. OHRINER,* AND L. F. ALLARD, "Processing and Mechanical Properties of Ni3Al-Based Intermetallic Composites"
V. K. SIKKA, E. K. OHRINER,* AND T. C. TSZENG, "Properties of Ni$_2$Al-Based Intermetallic Composites"

Seminar at Kyoto University, Kyoto, Japan, March 11, 1991:

C. T. LIU, "Recent Advances in Ordered Intermetallic Alloys"

Seminar at R&D Institute of Metals and Composites for Future Industries (RIMCOF), Tokyo, Japan, March 15, 1991:

C. T. LIU, "Environmental Embrittlement in Ordered Intermetallics"

C. T. LIU, "Intergranular Fracture and Alloy Design of Ordered Intermetallic Alloys"

American Physical Society Annual Meeting, Cincinnati, Ohio, March 18-22, 1991:

W. H. BUTLER,* A. GONIS, AND R. BROWN, "Basis Functions for Non-Muffin Tin Multiple Scattering Theory"

W. H. BUTLER* AND X. G. ZHANG, "Multiple Scattering Theory is NOT Wrong"

S. W. CHEONG, G. E. ICE, P. ZSCHACK, E. D. ISAACS,* AND M. NELSON, "Inelastic X-ray Scattering in LiH and La$_2$CuO$_4$"

R. E. CLAUSING, "Correlation of Structure-to-Growth Conditions for CVD Diamond"

A. GONIS AND W. H. BUTLER,* "Variational Derivation of Multiple Scattering Theory for Space-Filling Potentials"

G. E. ICE,* E. ISAACS, D. MCWHAN, AND P. ZSCHACK, "Splitting of Resonant Raman X-ray Peaks Near the M$_{iv}$ and M$_{v}$ Edges of Uranium"

X. JIANG, P. WOCNER, S. C. MOSS,* AND P. ZSCHACK, "X-Ray Diffuse Scattering Studies of a YBa$_2$ (Cu$_{1-x}$Al$_x$)$_3$O$_{7+z}$ Single Crystal (x = 0.045, 0.056) in the Tetragonal Phase"


D. M. NICHOLSON, G. M. STOCKS,* W. A. SHELTON, F. J. PINSKI, AND D. D. JOHNSON, "Multiple Scattering Theory and Harris Foulkes Energy Functionals"


P. E. A. TURCHI, M. SLUITER, AND G. M. STOCKS,* "Ni-Al: A First-Principles Study of the Interplay Between Chemical Order and Structural Effects"

X. G. ZHANG AND W. H. BUTLER,* "Calculation of Hellmann-Feynman Force Using Multiple Scattering Theory"


S. J. ZINKLE, "Radiation Effects in Ceramics for Fusion Diagnostic Systems"

American Society for Metals Meeting, San Diego, California, March 22, 1991:

V. K. SIKKA, "Processing and Joining of Nickel and Iron Aluminides"

Third International Conference on Improved Coal-Fired Power Plants, San Francisco, California, April 2-4, 1991:


Economic Development Executives, Lincoln, Nebraska, April 4, 1991:

J. R. WEIR, JR., "Technologies at Oak Ridge National Laboratory"

Seminar, Agency for Defense Development, Taejon, Korea, April 5, 1991:

L. F. ALLARD* AND T. A. NOLAN, "Microcharacterization of Advanced Ceramics at the High Temperature Materials Laboratory (HTML), Oak Ridge National Laboratory"

T. A. NOLAN* AND L. F. ALLARD, "The High Temperature Materials Laboratory at Oak Ridge National Laboratory (ORNL) - A National Resource"

8th International Conference on Wear of Materials, Orlando, Florida, April 7-11, 1991:

E. H. LEE,* G. RAO, AND L. K. MANSUR, "Improved Hardness and Wear Properties of B-Ion-Implanted Polycarbonate"

M. RAO* AND J. R. KEISER, "Erosion of an Iron Aluminide Alloy by Steel Shot"

Workshop on Low Activation Materials for Fusion Reactor Components, Abingdon, United Kingdom, April 8-11, 1991:

F. A. GARDNER* AND R. L. KLUEH, "U.S. Research Activities on Reduced Activation Austenitic Stainless Steels"

The Society of the Plastics Industry Annual Business Meeting, Washington, D.C., April 8-9, 1991:

R. S. GRAVES, "Joint Industry/Government Program: Update on Field and Laboratory Studies"

IEA Workshop on Low-Activation Materials, Culham Laboratory, United Kingdom, April 8-12, 1991:

E. E. BLOOM,* D. S. GELLES, AND R. L. KLUEH, "Development of Ferritic/Martensitic Steels as Low-Activation Fusion Reactor Structural Materials"

F. A. GARNER* AND R. L. KLUEH, "U.S. Research Activities on Reduced-Activation Austenitic Stainless Steels"

F. W. WIFFEN, "Ceramic Materials for First Wall/Blanket Structures in Fusion Reactors"

F. W. WIFFEN, "The United States Program on Low-Activation Materials for Fusion Energy Applications"

Seminar at Iowa State University, Ames, Iowa, April 9, 1991:

P. F. BECHER, "Advances in the Design of Toughened Ceramics"

Lawrence Berkeley Laboratory, Berkeley, California, April 9, 1991:

J. BENTLEY, "High Spatial Resolution Analytical Electron Microscopy of Materials"

Symposium on Nondestructive Testing Standards II: New Opportunities for Increased World Trade Through Accepted Standards for NDT Quality, Gaithersburg, Maryland, April 9-11, 1991:

R. W. MCCLUNG, "The Challenge of Standards for Emerging Technologies"

SCANNING '91, Atlantic City, New Jersey, April 9-12, 1991:

M. G. BURKE* AND E. A. KENIK, "Applications of STEM-EDS Microanalysis in Materials Science"
CAIMAF Conference—Application of Advanced Materials in the Resource Industries, Calgary, Canada, April 11-12, 1991:

J. R. WEIR, JR., "The Development and Commercialization of New Materials"

Women in Science, Knoxville, Tennessee, April 12-13, 1991:

T. DOLNEY, "Data Processing of 3-D SIMS Ion Images on a Macintosh Ilcx"

E. MEYER, "Microstructural Characterization of the Oxidation Processes of Pyrite Single Crystals"

American Chemical Society, Atlanta, Georgia, April 14-19, 1991:

R. R. JUDKINS,* W. FULKERSON, AND M. K. SANGHUI, "The Dilemma of Fossil Fuel Use and Global Climate Change"

72nd Annual AWS Convention, Detroit, Michigan, April 14-19, 1991:

D. J. ALEXANDER AND G. M. GOODWIN,* "An Assessment of High-Strength Stainless Steel Weldments for Fusion Energy Applications"

T. ZACHARIA,* S. A. DAVID, J. M. VITEK, AND H. G. KRAUS, "Computational Modelling of Linear GTA Welds and Comparison of Experimental Results"

Computational Research on Materials Conference, West Virginia University, Morgantown, West Virginia, April 15-17, 1991:

C. L. FU* AND M. H. YOO, "Synthesis of Atomistic and Continuum Modelling of Intermetallic Alloys"

Materials Science and Engineering Seminar, University of Tennessee, Knoxville, Tennessee, April 16, 1991:

R. B. DINWIDDIE, "An Introduction to Thermal Conductivity for Material Scientists"

Martin Marietta Advanced Quality Technology Meeting, Milan, Tennessee, April 16, 1991:

D. J. MCGUIRE, "Materials Testing at Oak Ridge National Laboratory"

22nd AWS International Brazing and Soldering Conference, Detroit, Michigan, April 16-18, 1991:

M. L. SANTELLA, "High-Strength Silicon Nitride Braze Joints"
K. B. ALEXANDER,* A. BLEIER, AND P. F. BECHER, "Grain Size Analyses in Alumina-Zirconia Composites"

L. F. ALLARD, "Image Processing in High-Resolution Electron Microscopy"

M. K. MILLER, "Visualization of Complex Microstructures from Atom Probe Field-Ion Microscope Data"

M. A. SCHMIDT, "Recent Confocal Microscopy Work at ORNL"

Workshop on Applications of Synchrotron Radiation to Chemical Engineering Science, Argonne National Laboratory, Argonne, Illinois, April 22, 1991:

G. E. ICE* AND C. J. SPARKS, "X-Ray Microimaging of Elemental Composition and Microstructure for Materials Science"

North Carolina State University, Raleigh, North Carolina, April 22, 1991:

M. K. MILLER, "Imaging and Analysis at the Atomic Level: The Atom Probe Field-Ion Microscope"

International Conference of Metallurgical Coatings and Thin Films, San Diego, California, April 22-26, 1991:

J. R. VANVALZAH,* H. E. EATON, AND R. B DINWIDDIE, "Thermal Aging Behavior in Plasma-Sprayed Yttria Fully Stabilized Zirconia: Correlation of Thermal Conductivity Increase and Dimensional Shrinkage"

Seminar, University of Tennessee, Knoxville, Tennessee, April 23, 1991:

K. B. ALEXANDER* AND P. F. BECHER, "Characterization of Interfaces in SiC Whisker-Reinforced Alumina Composites"

Annual Meeting of International Group on Radiation Damage Mechanisms in Pressure Vessel Steels (IG-RDM), Raleigh, North Carolina, April 23-26, 1991:

M. K. MILLER* AND M. G. BURKE, "Characterization of Copper Precipitation in a 17/4 PH Steel: A Combined APFIM/TEM Study"

M. K. MILLER* AND M. G. BURKE, "Grain-Boundary Segregation and Ultrafine Carbide Precipitation: An Atom Probe Survey"

R. E. STOLLER, "Overview of the Key Issues Concerning the Use of dpa as a Damage Correlation Parameter"


Hahn-Meitner-Institut, Berlin, Germany, April 26, 1991:

J. H. SCHNEIBEL, "Mechanical Properties of Intermetallic Li2 Phases and Composites"

93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 28-May 2, 1991:

R. L. BEATTY, "Overview of Microwave Processing at Oak Ridge National Laboratory"


A. BLEIER, "Particle Size and Solvent Effects in the Processing of Silicon Slurries"

A. BLEIER,* P. F. BECHER, AND K. B. ALEXANDER, "Processing of α-Al2O3-ZrO2 Composites"


A. CHOUDHURY,* K. K. CHAWLA, J. FERNANDO, AND J. R. HELLMANN, "SIMS Characterization of Nextel 480/Glass Composites"


E. F. FUNKENBUSCH, R. H. PLOVNICK, AND P. F. BECHER,* "Structural and Mechanical Properties of Ternary ZrO2-CeO2-HfO2 Alloys"


C. H. HSUEH, "Residual Stress-Induced Interfacial Debonding and Sliding in Fiber-Reinforced Ceramic Composites"
C. R. HUBBARD, R. B. DINWIDIE, O. B. CAVIN, AND W. D. PORTER,* "Research Highlights from the X-ray Diffraction and Thermophysical Properties User Centers, High Temperature Materials Laboratory"


M. A. JANNEY,* C. L. CALHOUN, AND H. D. KIMREY, "Microwave Sintering of Yttria-Doped Zirconia"


J. O. KIGGANS,* H. D. KIMREY, C. E. HOLCOMBE, AND T. N. TIEGS, "Characterization of Silicon Nitride Synthesized by Microwave Heating"

H. E. KIM* AND A. J. MOORHEAD, "High-Temperature Environmental Stability of SiC Whisker-Reinforced Alumina"

H. E. KIM* AND A. J. MOORHEAD, "High-Temperature Oxidation of Injection-Molded Aluminum Nitride"


H. T. LIN,* P. F. BECHER, C. W. LI, C. J. GASDASKA, AND J. YAMANIS, "Creep Behavior of In Situ Reinforced Silicon Nitride Ceramics"

R. A. MCKEE, "Strained Layer Superlattice Oxide Ceramics in the Titanium-Oxygen System"

A. J. MOORHEAD* AND H. E. KIM, "Active Metal Brazing of SiC Whisker-Reinforced Alumina"

K. L. MORE, "Defect Analysis in CVD α-Si$_3$N$_4$"

O. O. OMATETE,* R. H. STREHLOW, AND C. A. WALLS, "Drying of Gelcast Ceramics"

M. A. SCHMIDT*, C. E. HOLCOMBE, JR., AND C. E. BAMBERGER, "Analysis of Surface-Modified B$_4$C Powders"


T. N. TIEGS,* M. K. FERBER, AND J. O. KIGGANS, "Microwave Annealing of Dense Si$_3$N$_4$"

J. R. VANVALZH, H. E. EATON, AND R. B. DINWIDDIE,* "Effect of Thermal Aging on the Thermal Conductivity of Plasma-Sprayed Yttria-Stabilized Zirconia"

L. WANG, H. WADA, AND L. F. ALLARD,* "Synthesis and Characterization of SiC Whiskers"

1991 Spring Meeting of the Materials Research Society, Anaheim, California, April 29-May 4, 1991:

R. A. MCKEE* AND F. J. WALKER, "Surface Structures and the Orthorhombic Transformation of Thin-Film BaSi$_2$ on Si(001)"

R. A. MCKEE,* F. J. WALKER, AND J. R. CONNER, "Surface Structures and the Orthorhombic Transformation of Thin-Film BaSi$_2$ on Silicon"


Department of Materials Engineering, University of Connecticut, Storrs, Connecticut, May 1, 1991:

S. A. DAVID, "Advances in Welding Science and Technology"

TEM 1991 Seminar, EG&G Mound Laboratories, Miamisburg, Ohio, May 1, 1991:

L. F. ALLARD* AND T. A. NOLAN, "Microcharacterization of Advanced Ceramics at the High Temperature Materials Laboratory (HTML), Oak Ridge National Laboratory"

Workshop on Coal-Derived Graphite, Coke, and Pitch, University of West Virginia, Morgantown, West Virginia, May 2-3, 1991:

T. D. BURCHELL,* E. L. FULLER, G. R. ROMANOSKI, AND J. P. STRIZAK, "Graphite for the Nuclear Industry"

Workshop on Processing and Mechanical Properties of Ceramic Materials, National Taiwan University, Taiwan, May 5-23, 1991:

C. H. HSUEH, "Toughening Behavior and Interfacial Properties of Fiber-Reinforced Ceramic Composites"
ASTM E24 Committee Meetings, Chevron-Notch Fracture Test Experience Metals and Non-Metals, Indianapolis, Indiana, May 6, 1991:


DOE Energy Research Contractor Self-Assessment Workshop, Gatlinburg, Tennessee, May 6, 1991:

J. O. STIEGLER, "Continuing Self-Assessment at the Working Level in a Research and Development Organization" 

University of Illinois, Urbana, Illinois, May 6, 1991:

G. E. ICE AND E. H. LEE* (SPEAKER ONLY), "Synchrotron Radiation Research by the ORNL Metals and Ceramics Division"

DOE Office of BES Workshop on Environmental, Safety, and Health Issues, Gatlinburg, Tennessee, May 7-10, 1991:

J. O. STIEGLER, "The Rationale Behind the Conduct of Operations Strawman Guidelines for Research and Development Activities"

ASTM Symposium on Metallography: 75 Years Later, Atlantic City, New Jersey, May 8-10, 1991:

M. A. SCHMIDT, "Applications for Confocal Reflected-Light Microscopy in Materials Science"


R. A. MCKEE, "RHEED Observations of Layer-by-Layer Growth of Oxide Ceramics"


D. J. ALEXANDER, V. K. SIKKA, AND R. K. NANSTAD* (SPEAKER ONLY), "Fracture Behavior of Fe-Al Alloy Fa-129"

J. H. DEVAN, "Environmental Effects on Iron Aluminides"

M. A. JANNEY* AND H. D. KIMREY, "Microwave Sintering of Lanthanum Chromite"

C. G. MCKAMEY* AND P. J. MAZIASZ, "Development of Iron Aluminides"

M. RAO, "Erosion Studies on an Fe₃Al-Based Iron Aluminide and 1100 Al"

D. P. STINTON,* R. A. LOWDEN, J. C. MCLAUGHLIN, L. RISTER, AND M. C. CLARK, "Fiber-Reinforced SiC-Matrix Composites"

R. W. SWINDEMAN, "Investigation of Austenitic Alloys for Advanced Heat Recovery and Hot-Gas Cleanup Systems"

P. F. TORTORELLI* AND J. R. KEISER, "The Measurement of the Mechanical Properties of Oxide Scales"

P. F. TORTORELLI* AND J. R. KEISER, "The Mechanical Properties of Oxide Scales"

ORNL Showcase Lecture, Oak Ridge National Laboratory, Oak Ridge, Tennessee, May 15, 1991:

P. F. BECHER, "Advanced Ceramic Materials: How Do We Overcome the Brittle Nature of Such Materials?"


J. O. KIGGANS,* T. N. TIEGS, AND H. D. KIMREY, "Microwave Sintering of Silicon Nitride"

M. K. MILLER, "3-D Characterization of Materials at the Atomic Level: Recent Advances in Atom Probe Design"

Z. L. WANG* and J. BENTLEY, "Reflection Electron Imaging and Microanalysis in TEM"

Physical Phenomena in High Magnetic Fields, Tallahassee, Florida, May 15-18, 1991:

J. S. FAULKNER* AND G. M. STOCKS, "Requirements for the Observation of Fermi Surfaces in Disordered Alloys"

ASNT's Industrial Computed Tomography II Topical Conference, San Diego, California, May 20-24, 1991:

B. E. FOSTER AND F. HOPKINS,* "High-Resolution Imaging of Ceramic and Other Materials with Computed Tomography"
The Advanced Aerospace Materials/Processes Conference, Long Beach, California, May 20-24, 1991:

J. A. HORAK, "Studies to Improve the Strength of Nb-1Zr for SP-100"


L. K. MANSUR, "Irradiation Creep by Climb-Enabled Glide Driven by Transient Point-Defect Processes"

Seminar at Georgia Institute of Technology, Atlanta, Georgia, May 21, 1991:

T. M. BESMANN, "Vapor-Phase Fabrication of Ceramic Coatings and Composites"

Adriatico Conference on Structural and Phase Stability of Alloys, Trieste, Italy, May 21-24, 1991:


Yearly Meeting of the Deutsche Gesellschaft fur Metallkunde, Graz, Austria, May 21-24, 1991:

J. H. SCHNEIBEL,* P. GRAHLE, AND J. ROSLER, "Mechanical Alloy of FeAl"

AT&T Bell Laboratories, Murray Hill, New Jersey, May 22, 1991:

R. MCKEE, "BaSi$_2$ and the Transition to Barium-Containing Epitaxial Oxides on Silicon"

The Workshop on Cost-Effective Ceramic Machining, Pollard Auditorium, Oak Ridge, Tennessee, May 22-23, 1991:

M. K. FERBER, "A New Ceramic Specimen Preparation User Center at the ORNL High Temperature Materials Laboratory"

1991 GRI/Penn State Workshop, Chicago, Illinois, May 22-23, 1991:

E. L. LONG* AND T. VOJNOVICH, "Material Solutions for Alternatively Fueled Engines"

Electrical Breakdown of Installing Ceramics in a High-Radiation Field, Vail, Colorado, May 26-June 1, 1991:


S. J. ZINKLE, "Anomalous Microstructural Effects Associated with Light Ion Irradiation of Ceramics"
American Nuclear Society 1991 Annual Meeting, Orlando, Florida, June 2-6, 1991:

T. M. BESMANN, "How to Put on an ANS Teacher's Workshop"

Colloquium, Riso National Laboratory, Roskilde, Denmark, June 4, 1991:

S. J. ZINKLE, "Microstructural Changes in Irradiated Copper"

Nondestructive Evaluation for Aerospace Requirements Meeting, Huntsville, Alabama, June 4-6, 1991:

F. M. SHELL, J. D. ALLEN, JR., AND C. V. DODD, "Image-Based Neural Network Eddy-Current Signal Analysis"

Workshop on Recent Advances on Mathematical Theory of Anisotropic Elasticity, Research Triangle Park, North Carolina, June 4-6, 1991:

J. K. LEE* AND M. H. YOO, "Elastic Strain and Homogeneous Nucleation of a Deformation Twin in Hcp Ti Single Crystals"

Second International Workshop on Long-Term Thermal Performance of Cellular Plastics, Niagara-On-the-Lake, Ontario, Canada, June 5-7, 1991:

D. L. MCELROY, R. S. GRAVES,* AND F. J. WEAVER, "Thermal Resistance of Prototypical Cellular Plastic Roof Insulations"

D. W. YARBROUGH, "Progress Report from the Advanced Modeling Group-MIT/NRC/ORNL"

1991 P/M Conference & Exhibition, Chicago, Illinois, June 9-12, 1991:

V. K. SIKKA* AND J. H. REINSHAGEN, "P/M Processing and Applications of Ni₃Al- and Fe₃Al-Based Intermetallics"

U.S./Japan Workshop on Structural Materials, University of Alabama, Huntsville, Alabama, June 10, 1991:

D. J. ALEXANDER* AND G. M. GOODWIN, "Thick-Section Weldments in 21-6-9 and 316LN Stainless Steel for Fusion Energy Applications"

Advanced Industrial Concepts (AIC) Materials Program Annual Review Meeting, Albuquerque, New Mexico, June 10-11, 1991:

4th International Symposium on Ceramic Materials and Components for Engines, Göteborg, Sweden, June 10-12, 1991:


K. C. LIU, H. PIH, C. O. STEVENS, AND C. R. BRINKMAN,* "Tensile Creep and Cyclic Fatigue/Creep Interaction of HIP (Si₃N₄)"

D. P. STINTON,* B. W. SHELDON, AND J. C. MCLAUGHLIN, "Fabrication and Testing of Corrosion-Resistant Coatings"

T. N. TIEGS, J. O. KIGGANS, M. K. FERBER, H. D. KIMREY, AND D. P. STINTON* (SPEAKER ONLY), "Microwave Processing of Si₃N₄ Ceramics"

C. S. YUST, "Material and Lubricant Relationships in the Tribology of Internal Combustion Engines"

1991 SEM Spring Conference on Experimental Mechanics, Milwaukee, Wisconsin, June 10-13, 1991:

M. G. JENKINS* AND M. K. FERBER, "Determination of High-Temperature Creep Parameters in Structural Ceramics from Constant-Displacement, Load-Relaxation Tests"

Lectures Given at the Department of Metal Science and Technology and to the Institute of Metal Science and Technology and to the Information Meeting on Intermetallics, Kyoto University, Kyoto, Japan, June 10-14, 1991:

M. H. YOO* AND C. L. FU, "Crystal Elasticity and Mechanical Behavior of Ordered Intermetallic Alloys"

International Cryogenic Materials Conference, University of Alabama, Huntsville, Alabama, June 11-14, 1991:

D. J. ALEXANDER* AND G. M. GOODWIN, "Thick-Section Weldments in 21-6-9 and 316LN Stainless Steel for Fusion Energy Applications"

13th Symposium on Applied Surface Analysis, Minneapolis, Minnesota, June 12-14, 1991:

J. C. BIRKBECK, L. S. KASTEN, D. S. FOOSE, W. E. MODDEMAN, AND L. F. ALLARD,* "Auger, XPS, EDS, and TEM Examinations of Chromate Conversion Coated Cadmium Metal"

Wright-Patterson Air Force Base, Ohio, June 13, 1991:

D. J. MCGUIRE, "NDE Capabilities at Oak Ridge"
ASTM Symposium on Standardization Terminology for Better Communication: Practice, Applied Theory, and Results, Cleveland, Ohio, June 13-14, 1991:


University of California, Santa Barbara, California, June 13-14, 1991:

E. H. LEE, "Ion-Beam Modification of Polymer Materials to Improve Surface-Sensitive Mechanical Properties"

Colloquium, Risø National Laboratory, Roskilde, Denmark, June 28, 1991:

S. J. ZINKLE, "Effect of Irradiation on the Microstructure and Electrical Properties of Ceramics"

Electric Power Research Institute, Palo Alto, California, June 16, 1991:

E. H. LEE, "Ion-Beam Modification of Polymer Materials to Improve Surface-Sensitive Mechanical Properties"

Meeting of Westinghouse Technology Board, Pittsburgh, Pennsylvania, June 17, 1991:

J. O. STIEGLER, "Technology Transfer Opportunities in the Department of Energy Laboratory System"

NKK Corporation Steel Research Center, Kawasaki, Japan, June 17, 1991:

G. E. C. BELL, "Current Research Activities on IASCC at Oak Ridge National Laboratory"

Workshop on the Closed-Cycle Gas Turbine Modular High-Temperature Gas-Cooled Reactor, Boston, Massachusetts, June 17-19, 1991:

T. D. BURCHELL, M. ETO, AND G. HAAG, "Graphite Technology for the MHTGR-GT"

B. F. MYERS, "Fission Product Behavior in the Gas Turbine High-Temperature, Gas-Cooled Reactor"

H. NABIELEK, K. FUKUDA, AND M. J. KANIA,* "Is Present-Day HTGR Fuel Good Enough for Gas Turbine Application?"

Sixth JIM International Symposium (JIMIS-6) on Intermetallic Compounds—Structure and Mechanical Properties, Sendai, Japan, June 17-20, 1991:

I. BAKER,* P. NAGPAL, S. GUHA, AND J. A. HORTON, "TEM In Situ Straining of B2 Compounds"
C. L. FU AND M. H. YOO* (SPEAKER ONLY), "The Role of Electronic Structure in the Mechanical Behavior of Aluminides"

C. T. LIU, "Environmental Embrittlement in Intermetallic Compounds"

C. T. LIU, "Environmental Embrittlement of Ordered Intermetallic Alloys at Room Temperature in Moist Atmospheres"

V. K. SIKKA, "Deformation Processing of Nickel and Iron Aluminides"

M. TAKEYAMA, C. T. LIU,* AND C. J. SPARKS, "Microstructures and Hardness of NiAl/ Ni_2Al_3 Two-Phase Ordered Intermetallic Alloys"

M. TAKEYAMA, C. T. LIU,* AND C. J. SPARKS, "Microstructures of NiAl/Ni_2Al_3 Two-Phase Ordered Alloys"

M. H. YOO, "Effects of Elastic Anisotropy on Mechanical Behavior of Intermetallic Compounds"

B. ZHOU, S. L. YU, Y. T. CHOU, AND C. T. LIU,* "Effect of Boron Addition on Recrystallization and Grain Growth in Ni_3Al Polycrystals"

ASTM 23rd National Symposium on Fracture Mechanics, College Station, Texas, June 18-20, 1991:

D. J. ALEXANDER* AND R. D. CHEVERTON, "Cleavage Fracture of Type A 508 Class 2 Pressure Vessel Steel"

B. GIESEKIE* AND A. SAXENA, "Understanding Transient Creep-Fatigue Crack Growth in a Model Alloy (Cu-1%Sb)"

D. E. MCCABE, "A Comparison of Weibull and Beta_c Analyses of Transition Range Data"

CHESS Users Meeting, Cornell University, Ithaca, New York, June 19, 1991:

G. E. ICE* AND C. J. SPARKS, "Mosaic Crystals for Synchrotron Radiation Research"

ADVMAT/91 First International Symposium on Environmental Effects on Advanced Materials, San Diego, California, June 19-21, 1991:

C. G. MCKAMEY* AND C. T. LIU, "Environmental Embrittlement of Iron Aluminides in Moisture-Containing Atmospheres"

Wright Research & Development Center, Dayton, Ohio, June 21, 1991:

R. E. CLAUSING, "Evolution of Bulk and Surface Structures of Chemically Vapor Deposited (CVD) Diamond"
ASME Pressure Vessel & Piping Conference, San Diego, California, June 23-27, 1991:


R. W. SWINDEMAN* AND T. H. KRUKEMYER, "Performance of a 22Cr-20Ni-18Co-Fe Alloy for Service to 870°C"

20th Biennial Conference on Carbon, University of California, Santa Barbara, California, June 23-28, 1991:


E. L. FULLER, "Chemistry and Structure of Coals: Oxidation and Water Sorption Studies Using Diffuse Reflectance Infrared Spectroscopy"

E. L. FULLER, "Chemistry and Structure of Coals: Systematic Evaluation of Surface Area and Porosity from Physisorption Isotherms for Carbonaceous Materials"


R. J. LAUF* AND C. HAMBY, "Graphite-Metal Thermal Storage Systems"

G. R. ROMANOSKI* AND T. D. BURCHELL, "Specimen Size Effect on Fracture Toughness of Nuclear Graphites"


J. P STRIZAK* AND T. D. BURCHELL, "The Effect of Stress Volume on the Tensile Properties of Graphite"

NATO Advanced Research Workshop on Ordered Intermetallics-Physical Metallurgy and Mechanical Behavior, Irssee, Germany, June 23-29, 1991:

C. L. FU AND M. H. YOO,* "Fundamentals of Mechanical Behavior in Intermetallic Compounds—A Synthesis of Atomistic and Continuum Modeling"
C. T. LIU, "Environmental Embrittlement in Intermetallic Compounds"

J. H. SCHNEIBEL* AND P. M. HAZZLEDINE, "Creep in U₂ Intermetallics"

M. H. YOO* AND C. L. FU, "Deformation Twinning and Cleavage Fracture in Ordered Intermetallic Compounds"

International Symposium on Superalloys 718, 625, and Various Derivatives, Pittsburgh, Pennsylvania, June 24-26, 1991:


M. G. BURKE* AND M. K. MILLER, "Precipitation in Alloy 718: A Combined AEM and APFIM Investigation"

6th International Conference on Radiation Effects in Insulators, Weimar, Germany, June 24-28, 1991:

P. A. THEVENARD, "Chemical Effects in Implanted Ceramics"

Workshop on Neutron Dose Rate Effects/Spectral Effects on Materials Irradiation Degradation, Atlantic City, New Jersey, June 26, 1991:


Properties of Irradiated Ceramics Conference, Los Alamos, New Mexico, June 27-29, 1990:

T. N. TIEGS,* M. K. FERBER, AND J. O. KIGGANS, "Microwave Annealing of Dense Si₃N₄"

Los Alamos National Laboratory Meeting on ES&H, Los Alamos, New Mexico, July 10, 1991:

J. O. STIEGLER, "The Conduct of Operations Guidelines and Their Implementation in a Research and Development Environment"

4th International Symposium on Ceramic Materials and Components for Engines, Goteborg, Sweden, July 10-12, 1991:


European Workshop on Ordering and Disordering, Grenoble, France, July 10-12, 1991:

F. C. CHEN, A. J. ARDELL,* AND D. F. PEDRAZA, "Disordering and Amorphization of Zr₂Al by 3.8-MeV Zr³⁺ Ion Bombardment"
Meeting on Technology Transfer, Scottsbluff, Nebraska, July 14-16, 1991:

J. R. WEIR, JR., "The Development and Commercialization of New Materials"

International Conference on Fracture Mechanics of Ceramics, Nagoya, Japan, July 15-17, 1991:

A. GHOSH, M. G. JENKINS,* M. K. FERBER, J. PEUSSA, AND J. A. SALEM, "Elevated-Temperature Fracture Resistances (K_c, R-Curves, γ_w0) of Monolithic and Composite Ceramics Using Chevron-Notched Bend Test"

M. G. JENKINS* AND M. K. FERBER, "Stress-Relaxation Tests Used to Determine the Elevated-Temperature Creep Parameters of Structural Ceramics"

M. G. JENKINS,* M. K. FERBER, W. S. HONG, J. M. SATER, AND M. A. RIGDON, "Effect of Exposure Time and Strain Rate on the Elevated-Temperature Tensile Strength of SiC Monofilaments"

7th Annual Coal Preparation, Utilization & Environmental Control Contractors Conference, Pittsburgh, Pennsylvania, July 15-18, 1991:


The Seventh International Conference on Ion Surface Modification of Metals by Ion Beams, Washington, D.C., July 15-19, 1991:

Y. LEE,* E. H. LEE, AND L. K. MANSUR, "Hardness and Wear Properties of Boron-Implanted PEEK and Ultem"


L. J. ROMANA,* P. S. SKLAD, C. W. WHITE, J. C. MCCALLUM, A. CHOUHURY, L. L. HORTON, AND C. J. MCHARGUE, "Formation and Annealing Behavior of an Amorphous Layer Induced by Tin Implantation Into Sapphire"

L. J. ROMANA, P. THEVENARD,* S. RAMOS, B. CANUT, L. GEA, M. BRUNEL, L. L. HORTON, AND C. J. MCHARGUE, "Formation of Small Metallic Precipitates of Niobium in α-Al2O3 Implanted with Nb Ions"

The Eighth International Conference on Composite Materials, Honolulu, Hawaii, July 15-19, 1991:

A. J. MOORHEAD* AND H. E. KIM, "Effects of Exposure to H₂-H₂O and Ar-O₂ and Air Atmospheres on the Strength of SiC Fibers"

Contractor's Review Meeting, Morgantown Energy Technology Center (METC), Morgantown, West Virginia, July 18, 1991:

R. W. SWINDEMAN AND R. H. MALLETT, * "Materials for Hot-Gas Filter Tubesheets"

Office of Industrial Technologies, Oak Ridge National Laboratory, Oak Ridge, Tennessee, July 19, 1991:


Technical Seminar, Center for Laser Application, University of Tennessee Space Institute, Tullahoma, Tennessee, July 24, 1991:

L. F. ALLARD, "The Ultrastructure of SiC Whiskers and Whisker-Reinforced Composites"

Argonne National Laboratory Fusion Power Program Seminar Series, Argonne, Illinois, July 29, 1991:


24th Annual Convention of International Metallographic Society, Monterey, California, July 29-August 1, 1991:

A. CHOUDHURY* AND C. R. BROOKS, "A Secondary Ion Mass Spectrometry (SIMS) Analysis of the Microstructure of an Embrittled 12% Cr Steel Bolt"

S. A. DAVID, M. J. GARDNER,* AND J. M. VITEK, "Application of Optical Metallography to Characterize Unique Features of Weld Microstructures"

Advanced Materials and Processing Technology Workshops, Alexandria, Virginia, August 1-3, 1991:

P. ANGELINI, "Advanced Industrial Concepts (AIC) Materials Program"

49th Annual Meeting of Electron Microscopy Society of America (EMSA), San Jose, California, August 4-9, 1991:

K. B. ALEXANDER* AND P. F. BEHCER, "Microstructural Evaluation of Silicon Carbide Whisker-Reinforced Alumina Fabricated with Carbon-Coated Whiskers"
L. F. ALLARD* AND T. A. NOLAN, "The Microstructure of Ceramic Whiskers"


N. D. EVANS,* S. J. ZINKLE, J. BENTLEY, AND E. A. KENIK, "Quantification of Metallic Aluminum Profiles in Al* Implanted MgAl2O4 Spinel by Electron Energy Loss Spectroscopy"


E. A. KENIK* AND K. HOJOU, "Radiation-Induced Segregation in Austenitic Stainless Steels"

K. L. MORE, AND L. ALLARD* (SPEAKER ONLY), "Defect Characterization in a CVD α-Si3N4"

G. M. MA, N. D. EVANS,* T. TACHIBANA, R. E. CLAUSING, AND J. T. GLASS, "Annealing Effects on the Microstructure of Titanium Electrical Contact on Diamond Films"

Z. L. WANG, "Multiple-Inelastic Phonon, Single Electron, and Valence Excitations in High-Energy Electron Scattering"

Z. L. WANG* AND J. BENTLEY, "Correcting Channeling and Diffraction Effects in Quantitative REELS Surface Microanalysis"

Z. L. WANG* AND J. BENTLEY, "In Situ REM Imaging of Surface Processes on Ceramic Bulk Crystals from 300 to 1670 K in a Conventional TEM"


38th International Field Emission Symposium, Vienna, Austria, August 5-9, 1991:


R. JAYARAM* AND M. K. MILLER, "An APFIM Analysis of Grain Boundaries in B-Doped NiAl"

R. JAYARAM* AND M. K. MILLER, "An APFIM/TEM Study of Crept Model Ni-Mo-Ta-Al Superalloys"
M. K. MILLER, "Implementation of the Optical Atom Probe"

M. K. MILLER, "Some Factors Affecting Surface Analysis in the Atom Probe"

M. K. MILLER* AND M. G. BURKE, "An APFIM Study of Composition in 17/4 PH Steels"


M. K. MILLER* AND K. F. RUSSELL, "Fractal Analysis of Field Evaporation Micrographs of Fe-Cr Alloys"

M. K. MILLER* AND K. F. RUSSELL, "Further APFIM Investigations of the Santa Catharina Meteorite"


Short Course on Preventive Maintenance Technology, the University of Tennessee, Knoxville, Tennessee, August 8, 1991:

C. V. DODD, "Eddy-Current Inspection of Steam Generator Tubing"

Pacific-International Congress on X-ray Analytical Methods, Honolulu, Hawaii, August 12-16, 1991:


C. R. HUBBARD,* G. ZORN, AND A. G. SIEMENS, "High-Temperature Diffraction"

B. J. REARDON AND C. R. HUBBARD,* "A Comprehensive Review of the XRD Data of the Primary and Secondary Phases Present in the BSCCO Superconductor System"


X-Ray Physics Gordon Conference, Brewster, New Hampshire, August 12-16, 1991:

E. D. SPECHT,* F. J. WALKER, AND R. A. MCKEE, "Determination of Interface Structure by Anomalous X-ray Diffraction"

D. P. STINTON, "Ceramic Fiber-Ceramic Matrix Hot-Gas Filters"

The Eighth International Conference on Composite Materials, Honolulu, Hawaii, August 15-19, 1991:

A. J. MOORHEAD* AND H. E. KIM, "Strength of SiC and Si-N-Ceramic Fibers Exposed to High-Temperature Gaseous Environments"

First International Conference on the Applications of Diamond Films and Related Materials—ADC'91, Auburn, Alabama, August 17-22, 1991:

C. J. MCHARGUE,* "Mechanical Properties of Diamond and Diamond-Like Films"

Z. L. WANG,* J. BENTLEY, R. E. CLAUSING, L. HEATHERLY, AND L. L. HORTON, "Growth Mechanisms of CVD Diamond Films Determined by a Novel TEM Technique"

30th Annual Meeting of the Canadian Institute of Metallurgists (CIM), Ottawa, Canada, August 18-21, 1991:

V. K. SIKKA, "Development of Nickel Aluminides and Their Applications"


J. E. BROWN, G. D. W. SMITH, AND M. K. MILLER,* "APFIM Characterization of the Spinodal Decomposition in Duplex Stainless Steels"

F. M. HAGGAG* AND R. K. NANSTAD, "Effects of Thermal Aging and Neutron Irradiation on the Mechanical Properties of Stainless Steel Weld Overlay Cladding"

F. M. HAGGAG,* R. K. NANSTAD, AND S. T. BYRNE, "Use of Miniature and Standard Specimens to Evaluate Effects of Irradiation Temperature on Pressure Vessel Steels"

A. J. JACOBS, C. M. SHEPERD, G. E. C. BELL,* AND G. P. WOZADLO, "High-Temperature Solution Annealing as an IASCC Mitigation Technique"

M. K. MILLER* AND M. G. BURKE, "Characterization of Copper Precipitation in a 17/4 PH Steel: A Combined APFIM/TEM Study"

American Chemical Society National Meeting, New York, August 25-30, 1991:

Workshop on Reviewing Energy Selective Neutron Irradiation Test Facility (ESNIT), Toaki-mura, Japan, August 26, 1991:

F. W. WIFFEN, "Materials Test Facilities for the Fusion Program—A U.S. Perspective on Neutron Sources"

SMIRT 11 Post-Conference Seminar No. 2, Assuring Structural Integrity of Steel Reactor Pressure Boundary Components, Taipei, Taiwan, August 26-28, 1991:

W. R. CORWIN,* R. K. NANSTAD, S. K. ISKANDER, F. M. HAGGAG, AND D. E. MCCABE, "Recent Results from the Heavy-Section Steel Irradiation Program"

Technology Transfer Conference and Workshop on Nickel and Iron Aluminides, Oak Ridge, Tennessee, August 26, 1991:

P. F. TORTORELLI, "The Measurement of the Mechanical Properties of Oxide Scales"

Japan Atomic Energy Research Institute, Tokyo, Japan, August 30, 1991:

W. R. CORWIN, "Recent Results from the Heavy-Section Steel Irradiation Program"

Mitsubishi Heavy Industries, Takasago, Japan, September 2, 1991:

W. R. CORWIN, "Recent Results from the Heavy-Section Steel Irradiation Program"

Diamond Films '91 Conference, Nice, France, September 2-6, 1991:


Workshop on Fusion Pilot Plants, Fusion Engineering Design Center, Oak Ridge, Tennessee, September 2-13, 1991:

E. E. BLOOM, "Materials Issues for a Fusion Pilot Plant"

Japan Power Engineering and Inspection Corporation, Tokyo, Japan, September 3, 1991:

W. R. CORWIN, "Recent Results from the Heavy-Section Steel Irradiation Program"

American Institute of Aeronautics and Astronautics (AIAA) Conference on Advanced Space Exploration Initiative (SEI) Technologies, Cleveland, Ohio, September 4-6, 1991:

R. H. COOPER, "Materials for Space Thermal Propulsion Systems"
R. H. COOPER* AND J. P. MOORE, "Refractory Alloys for Space Nuclear Electric Propulsion Systems"

First Canadian International Composites Conference and Exhibition, Montreal, Quebec, Canada, September 4-6, 1991:


D. P. STINTON,* R. A. LOWDEN, AND T. M. BESMANN, "Processing of Continuous Fiber-Reinforced Ceramic-Matrix Composites by Chemical Vapor Infiltration"

Seminar at United Technologies Research Center, East Hartford, Connecticut, September 5, 1991:

E. H. LEE, "Ion-Beam Modification of Polymer Materials to Improve Surface-Sensitive Mechanical Properties"

8th Meeting of EURO CVD at the University of Strathclyde, Glasgow, Scotland, September 8-13, 1991:

Y. G. ROMAN,* D. P. STINTON, AND T. M. BESMANN, "The Development of High-Density FCVI Silicon Carbide Composites"

Workshop on Advanced Thin Films, Los Angeles, California, September 9-10, 1991:


IAEA Specialists Meeting on Status of Graphite Development for Gas-Cooled Reactors, Japan Atomic Energy Research Institute, Tokai, Japan, September 9-12, 1991:

T. D. BURCHELL, "Graphite Development for Gas-Cooled Reactors in the USA"

T. D. BURCHELL, "A Microstructurally Based Fracture Model for Nuclear Graphites"

G. R. ROMANOSKI AND T. D. BURCHELL,* "The Effects of Specimen Geometry and Volume on the Fracture Toughness of Nuclear Graphites"

J. P. STRIZAK, "The Effect of Volume on the Tensile Strength of Several Nuclear-Grade Graphites"

J. P. STRIZAK, "Spatial Variability in the Tensile Strength of an Extruded Nuclear-Grade Graphite"
Electron Microscopy and Analysis Group '91, University of Bristol, Bristol, England, September 10-13, 1991:

J. BENTLEY,* Z. L. WANG, AND T. SHIRAISHI, *ALCHEMI* Measurements of Sublattice Occupancy in Cu_{50}Au_{50-x}Ni_x (0 x 12) L1_0 Ordered Alloys*

Z. L. WANG AND J. BENTLEY,* "Valence and Phonon Excitations in High-Resolution Electron Microscopy"

European Ceramic Society Second Conference, Augsburg, Germany, September 11-14, 1991:

K. C. LIU,* C. O. STEVENS, AND C. R. BRINKMAN, *High-Temperature Tensile and Fatigue Strengths of Silicon Nitride*

Technical University of Hamburg-Harburg, Germany, September 14-16, 1991:

P. F. BECHER, "Advances in the Design of Toughened Ceramics"

Virginia Professional Photographers' Association Fall Seminar, Roanoke, Virginia, September 16, 1991:

J. W. NAVE, "The Role of Research, Industrial, and Public-Relations Photography"

International Conference on High-Temperature Aluminides and Intermetallics, San Diego, California, September 16-19, 1991:

D. J. ALEXANDER* AND V. K. SIKKA, "Mechanical Properties of Advanced Nickel Aluminides"


F. CHEN, A. J. ARDELL,* AND D. F. PEDRAZA, *Fractography and Microstructure of Zr^{3+} Ion- and Proton-irradiated Zr_2Al*

C. L. FU, "First-Principles Calculations of Intrinsic and Extrinsic Defects in Ordered Intermetallics"

B. GIESEKE AND V. K. SIKKA,* "Influence of Test Temperature and Casting Temperature on Fatigue Properties of Cast Ni_2Al"


C. T. LIU, "Environmental Embrittlement and Alloy Design of Ordered Intermetallic Alloys"

P. J. MAZIASZ* AND C. G. MCKAMEY, "Microstructural Characterization of Precipitates Formed During High-Temperature Testing and Processing of Iron-Aluminide Alloys"

C. NISHIMURA* AND C. T LIU, "Factors Affecting the Tensile Properties of L12-Ordered (FeCo)3V Alloys"

J. H. SCHNEIBEL,* P. GRAHLE, AND J. ROSLER, "Mechanical Alloying of FeAl with Y2O3"

J. H. SCHNEIBEL,* J. A. HORTON, AND W. D. PORTER, "Bend Ductility and Physical Properties of Extruded Chromium-Modified Al3Ti"


P. F. TORTORELLI* AND J. H. DEVAN, "Behavior of Iron Aluminides in Oxidizing and Oxidizing/Sulfidizing Environments"

10th Pfefferkon Conference on Signal and Image Processing in Microscopy and Microanalysis, Cambridge, England, September 16-19, 1991:

Z. L. WANG AND J. BENTLEY,* "Diffraction and Imaging Theory of Inelastically Scattered Electrons—A New Approach"

First International Conference on Heat-Resistant Materials, Lake Geneva, Wisconsin, September 22-26, 1991:

S. A. DAVID* AND T. ZACHARIA, "Welding of Ductile Intermetallic Alloys"

J. H. DEVAN, "Corrosion Performance of Iron Aluminide Fe3Al in Coal Conversion Process Environments"

J. R. DISTEFANO AND J. H. DEVAN* (SPEAKER ONLY), "Corrosion of Refractory Metals in Liquid Metal and Gaseous Environments"

E. P. GEORGE,* J. J. LIAO, AND C. T. LIU, "The Effect of Boron on the Hall-Petch Slope of NiAl"

V. K. SIKKA, "Processing and Fabrication of Fe3Al-Based Alloys"

V. K. SIKKA,* C. G. MCKAMEY, AND D. J. ALEXANDER, "Mechanical Properties of Fe3Al-Based Alloys"

R. W. SWINDEMAN, "Mechanical Performance of an Alloy 3C - to Support Ceramic Barrier Filters in Hot-Gas Cleanup Systems"
R. W. SWINDEMAN AND P. J. MAZIASZ, "The Effect of MC-Forming Additions and 10% Cold Work on the High-Temperature Strength of 20Cr-30Ni-Fe Alloys"

Aerotech "91, Long Beach, California, September 23, 1991:

J. R. WEIR, JR.,* AND V. K. SIKKA, "Primary Fabrication Processes for Nickel and Iron Aluminides"

University of Tennessee, Graduate Seminar sponsored by the Department of Metallurgical Engineering, Knoxville, Tennessee, September 24, 1991:

R. E. PAWEL, "Aluminium Corrosion and Heat Transfer in the Advanced Neutron Source"

Polyurethanes World Congress 1991 Acropoli, Nice, France, September 24-26, 1991:


ORNL/Tractebel Meeting on Pressurized Thermal Shock, Brussels, Belgium, September 25, 1991:


Smoky Mountain Orchid Society, Knoxville, Tennessee, September 26, 1991:

R. J. LAUF, "In Vitro Propagation of Orchids from Seed"

University of Wisconsin, Madison, Wisconsin, September 26-27, 1991:


Graduate Student Seminar, Michigan Technological University, Houghton, Michigan, September 27, 1991:

A. CHOUDHURY, "Secondary Ion Mass Spectrometry and Applications to Microstructural Analysis"
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