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Energy Technology Division

Research Summary • 1997

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Energy Technology Division

Overview of the Energy Technology Division

The Energy Technology Division provides materials and engineering technology support to a wide range of programs important to the U.S. Department of Energy. As shown on the preceding page, the Division is organized into ten sections, five with concentrations in the materials area and five in engineering technology. Materials expertise includes fabrication, mechanical properties, corrosion, friction and lubrication, and irradiation effects. Our major engineering strengths are in heat and mass flow, sensors and instrumentation, nondestructive testing, transportation, and electromechanics and superconductivity applications. The Division Safety Coordinator, Environmental Compliance Officers, Quality Assurance Representative, Financial Administrator, and Communication Coordinator report directly to the Division Director. The Division Director is personally responsible for cultural diversity and is a member of the Laboratory-wide Cultural Diversity Advisory Committee.

The Division's capabilities are generally applied to issues associated with energy production, transportation, utilization or conservation, or with environmental issues linked to energy. As shown in the organization chart on the next page, the Division reports administratively to the Associate Laboratory Director (ALD) for Energy and Environmental Science and Technology (EEST) through the General Manager for Environmental and Industrial Technologies. While most of our programs are under the purview of the EEST ALD, we also have had programs funded under every one of the ALDs. Some of our research in superconductivity is funded through the Physical Research Program ALD. We also continue to work on a number of nuclear-energy-related programs under the ALD for Engineering Research. Detailed descriptions of our programs on a section-by-section basis are provided in the remainder of this book.

This Overview highlights some major trends. Research related to the operational safety of commercial light water nuclear reactors (LWRs) is funded by the U.S. Nuclear Regulatory Commission (NRC). In addition to our ongoing work on environmentally assisted cracking and steam generator integrity, a major new multiyear program has been initiated to assess the performance of high-burnup fuel during loss-of-coolant accidents. The bulk of the NRC research work is carried out in four ET sections: Corrosion; Mechanics of Materials; Irradiation Performance; and Sensors, Instrumentation, and Nondestructive Evaluation. The Transportation of Hazardous Materials Section is the other main contributor; staff from that Section have worked closely with NRC staff to draft a new version of the NRC Standard Review Plan that will be used to provide guidance to NRC reviewers of applications for the renewal of nuclear plant licenses.

ET Funding Sources for FY 1997

Technology development for the safe, long-term disposal of radioactive and hazardous waste is receiving major emphasis within the Department of Energy. The Ceramics Section has demonstrated a process for stabilizing hazardous, radioactive, and mixed wastes by using novel phosphate ceramics that set at room temperature. These materials, which have been named "Ceramcrete," can stabilize a wide range of waste streams. An R&D 100 award was given in 1996 for the development of these materials, and staff from the Section are now working with a number of DOE sites to demonstrate the effectiveness of these materials on their particular waste streams.

DOE recently selected a team, led by Air Products and Chemicals, that includes ANL and other partners for an eight-year, \$84 million project to commercialize the ceramic membrane technology, developed by the Ceramics Section, that allows air to be used in syngas-generation reactions.

The Tribology Section has recently developed a new technique for the production of diamondlike carbon films. The resulting films are extremely low in friction (the coefficient of friction is about 1/40 that of Teflon) and very hard, thus giving them exceptional wear resistance and durability. These films can be applied at lower temperatures and on a wider range of substrates than any current diamond coating.

The work of the Electromechanics and Superconductivity Applications Section has been particularly successful in developing devices that utilize the unique properties of high-temperature superconductors in ways that have great technological and industrial potential. Building on their comprehensive studies of low-loss superconducting bearings, Section staff have worked closely with Commonwealth Research Corporation (CRC) to develop an efficient flywheel system for diurnal utility energy storage. Supported by extensive funding and technical collaboration through CRC, this energy storage program provides an excellent example of how the Division can combine its expertise with that of private industry to move a new technology from the laboratory to near-commercial deployment.

A major strength of the Division continues to be its strong program in sensor development. The work of the Sensors, Instrumentation, and Nondestructive Evaluation Section is best characterized by the diversity of its sponsors and by how it is finding new and exciting applications for advanced systems in a variety of industrial areas. A noteworthy example of diverse deployment of a specific technology is provided by the Section's work on millimeter-wave devices. Initial work on the use of millimeter-wave sensors was supported by the arms control programs to detect effluent chemicals associated with chemical weapons production. That project led to a wide variety of innovative precompetitive commercial applications, including uses as diverse as the continuous monitoring of industrial effluents to systems for on-line, real-time detection of flaws in woven fabrics.

The Division has a long history of research and development of heat transfer and fluid flow systems for advanced energy systems. While much of this work grew out of nuclear reactor development programs, current emphasis has shifted to the chemical process and transportation industries. The programs of the Thermal, Mechanical, and Chemical Processes Section provide a good example of this transition. While the Section continues to work on a small program in flow-induced vibrations, which was once one of their major

activities, its largest recent effort has been to develop an understanding of the unique heat transfer problems associated with compact heat exchangers. Applications include high-performance condenser and evaporator tubes for automotive and residential air conditioning systems, applying compact heat exchangers to improve the efficiency and control of distillation columns, and developing a system to reform jet fuel to obtain a gaseous product with improved combustion characteristics.

Although many of the Division's efforts are best described as advanced technology development projects drawing on a specific engineering or scientific discipline, there are continuing programs that integrate a large number of specialized areas to provide a unique group capability for a DOE sponsor. The Transportation of Hazardous Materials Section is the best example of this ongoing integrated approach to a specific DOE need. Section staff provide direct technical support to DOE in reviewing Safety Analysis Reports for Packaging (SARPs) of radioactive material shipping packages to ensure compliance with DOE, NRC, and DOT regulations. This work spans technical fields such as structural and thermal analysis, shielding and criticality, and operations, maintenance and quality assurance. Working as a team on specific SARP submissions, staff provide DOE with the independent technical overview needed to allow safe and reliable shipment of a wide range of potentially hazardous nuclear materials.

The Division continues its long history of research into advanced methods of simulation and modeling of fluid systems through the work of the Analytical Thermal-Hydraulics Section. This is another example of a program that has shifted emphasis as funding priorities change. While once almost completely supported by the Nuclear Regulatory Commission, the Section now collaborates in projects with the auto industry and manufacturers of heavy equipment. A central focus has been to provide the auto industry with an advanced, general-purpose computational fluid mechanics code that can simulate the complex air flow and heat transfer associated with under-hood cooling and with passenger-compartment ventilation. In addition, work continues on computational models for casting and welding problems of interest to general manufacturing industries.

The Division's staff are active in national and international professional and scientific societies. They participate in joint programs, working groups, and national and international collaborations. They regularly attend meetings, present papers, organize conferences, and edit journals. Some even teach in local colleges and universities. Staff regularly receive awards and honors in recognition of their various achievements.

Engineering Technology

Analytical Thermal-Hydraulics

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Staff: M. Bottoni,¹ F. C. Chang, T. H. Chien, H. M. Domanus,

R. C. Schmitt, W. T. Sha²

Secretary: C. M. Vargas

The main research discipline of the Analytical Thermal-Hydraulic Section is computational fluid mechanics, with particular emphasis on development and implementation of mathematical and phenomenological models of single-phase and two-phase fluids applied to a variety of advanced energy systems. For much of its history, the Section has developed models related to components and safety systems of advanced nuclear reactors. In the past two years, however, work in this area has almost ended and several new classes of simulation have gained importance. Recent research has been directed toward modeling the fluid mechanics of casting and welding processes and to automotive applications such as prediction of air flow within the passenger compartment of an automobile.

Casting Modeling and Extensions of the CaPS Code

Casting Modeling is a research and development program that formally started in 1990 as a consortium comprising ANL, Caterpillar, and Teledyne Corporation. Early in 1992, Teledyne withdrew from the consortium for financial reasons. The program continued as a cooperative effort between ANL and Caterpillar, and most of the original objectives were achieved. The Casting Processes Simulator (CaPS) software package that resulted from this effort has been used by Caterpillar since 1995 and has resulted in improved gating and riser systems that lead to higher-quality castings and fewer rejections. The Section continues to interact with Caterpillar on the use of CaPS and on possible improvements in its capabilities; however, no formal R&D project is currently supported for this effort. In an attempt to interest a wider technical community in the use of CaPS, several extensions of the code have been considered during the last year. These include extensions to weld modeling and to die casting.

Welding

In developing the CaPS code and applying it to various problems of mold filling and solidification, it became evident that many of the technical issues being addressed for casting did not differ greatly from those associated with welding. In particular, formation and behavior of the weld pool could certainly be modeled by an extension of the simulation techniques employed when developing CaPS.

During discussions on welding simulation with various industrial representatives, it became clear that there are two coupled but distinct areas of concern. The first concerns local effects, that is, physical processes that are local to the weld pool and its solidification,

¹Leave of absence at Tokyo Institute of Technology.

²Resident Associate -- Guest Appointment.

and to evolution of final material properties and local stresses. The second area concerns more global features such as distortion of an entire structure due to the strains induced during welding. Because CaPS is well suited to the study of local effects, it was decided that only weld pool dynamics would be considered in the initial development. A link to global structural concerns and perhaps to other structural computer codes is being left for later development.

Most weld materials are alloys or mixtures of several constituents. While the constituents are uniformly distributed in the object part and filler wire, they are usually of different compositions. As the filler wire melts and combines with the molten portion of the object part and begins to solidify, the alloying constituents separate due to solubility differences between liquid and solid phases. This macroscopic segregation creates an inhomogeneous distribution of the constituents in the final weld due to variations of mechanical properties that, in turn, affect weld performance. An initial binary macrosegregation model has been developed and implemented in CaPS.

The CaPS modification that allows simulation of the welding process provides detailed information on weld geometry and time histories of all points within and external to the weld pool. Comparison of these results with data to be obtained from detailed experiments at ANL will provide the basis for code validation. The illustration below shows a section through a weld bead and indicates regions where melting of the base metal has occurred, it also indicates the extent of the weld pool. The section is through the V-groove between two plates being welded together. The light-shaded line is the boundary between molten metal and the solidified weld material. One can observe the deep pool directly below the weld wire and note how the molten area extends to the left of the wire (the weld proceeds from left to right).

Die Casting

As a part of the 1995 conference "Modeling of Casting, Welding, and Advanced Solidification Processes," ANL participated in a fluid simulation of a specific test casting.

*Section through weld showing bead surface
and depth of weld pool*

The CaPS result was excellent and, of the nine submitted simulations, CaPS was probably second in overall performance. To test the feasibility of extending CaPS from modeling of gravity-fed systems to high-velocity injection molding or die casting, it was decided that the same flat plate geometry used in the 1995 blind simulation would be the test case. Of particular interest would be the changes in observed fill pattern as one moved from a fill time of 2 s to one where the fill was complete in 20 ms. For this test case, the goal was to ensure that the calculation remained stable at the high velocities involved and that the fill pattern seemed to be physically reasonable. A typical result is shown below. The gravity-fed fill pattern forms the background of this figure, and the injection pattern is superimposed on that background. The gravity feed has filled approximately half of the plate in 1.50 s, while the injection filling produces a jet of material that has struck the top surface and spread downward, taking only 10 ms. The computations appear stable and physically reasonable.

Application of High-Performance Computing to Automotive Design and Manufacture

The Section has participated with members of several other ANL divisions in a collaborative project with the Big Three automakers to develop a general-purpose computational fluid mechanics (CFD) code applicable to problems in combustion, underhood cooling and HVAC, and exterior aerodynamics. This activity has built on the Los Alamos developed code, CHAD, which is an acronym for Computational Hydrodynamics for Advanced Design. During the past year, the ANL focus has been on passenger compartment air flow and windshield defrosting and on developing CHAD modifications that would model radiator and other component behavior through use of a porous media formulation.

*Comparison of transient surface
shape of plate casting with
equivalent injection-filled
surface*

Several components in automotive systems have characteristic structures smaller than can be practically resolved through conventional means. Examples include filters, vanes, heat exchangers (radiator, heater core, evaporator, etc.), mufflers, catalytic converters, and screens. These components are present in fuel, lubrication, cooling, and exhaust systems. Current computing hardware is not able to handle these components with the conventional approach present in CHAD. Consider a CFD model for a catalytic converter: while theoretically possible to model the full flow field, it is not practical to assume that the detailed flow, temperature and concentration fields of the exhaust gases flowing around the multitude of spheres comprising the catalytic medium can be modeled by accounting for each of the spheres with complete boundary conditions. To handle such situations, the porous media formulation is being considered.

The porous medium formulation involves the parameters of volume porosity, directional surface permeability, distributed mass source or sink, distributed resistance, and distributed heat source or sink. With this formulation, an anisotropic flow domain with stationary structures can be modeled. The porous medium formulation with the additional parameter of directional surface permeability represents a unified approach to thermal-hydraulic analysis. Volume porosity is the ratio of the volume occupied by fluid in a control volume to the total control volume. Surface permeability is similarly defined as the ratio of fluid flow area through a control surface to the total control surface area. Accurately accounting for these geometrical aspects reduces the required accuracy for the correlations needed in the distributed mass, momentum, and energy source terms. The distributed mass source or sink accounts for the effect on species concentration due to the fluid interacting with the immersed solid structures. The distributed resistance is a drag force opposing the flow and is due to the fluid flowing around the immersed solid structures. The distributed heat source or sink represents the thermal interaction with the immersed solid structures.

It has been determined that a complete implementation of the porous medium formulation is beyond the scope of the current automotive project. Much of CHAD, as well as preprocessors, would have to be modified or rewritten to account for the geometrical aspects. A partial implementation consisting of only the concept of distributed resistance is being considered as an alternative.

The usual form of the instantaneous momentum equations are modified by simulation of the pressure drop through a porous medium using a Darcy's law model with a resistance force $\bar{F}_R$ of the form

$$\bar{F}_R = \nabla p_D = -\bar{\beta} \cdot \bar{u} ,$$

where $\bar{\beta}$ is a symmetric second-order tensor. Through a relatively straightforward process, a discrete equation can be written for the nonlinear residuals of the velocity vectors to reduce these to small values during the solution procedure.

Replacement of the momentum equations at porous medium nodes by Darcy's law $\nabla p = \bar{F}_R$ would require extensive modifications to the current formulation and solution scheme, but these are beyond the current scope of the project.

Regions within the configuration are identified and associated with specific flow resistance characteristics. Each region may have different flow resistance characteristics. The flow resistance characteristics include a unit direction vector, $\hat{n} = (n_x, n_y, n_z)$ and correlations in that direction, f_n , and for the crossflow direction, f_t . The flow resistance coefficient tensor, $\bar{\beta}$, is symmetric. The diagonal and off diagonal terms are given by

$$\bar{\beta} = \frac{1}{2} \frac{\rho |\bar{u}|}{L} \left[f_t \bar{I} + (f_n - f_t) \hat{n} \hat{n} \right],$$

where L = characteristic length and both f_n and f_t can be functions of the local velocity magnitude. Three important limiting situations are:

- Isotropic resistance. This situation may occur in the media of a catalytic converter or a seat cushion. Here $f_n = f_t$ and the off-diagonal terms vanish.
- Flow in the direction specified by $\hat{n}$. This situation occurs when multiple parallel flow paths are available, such as air flow through a radiator. Here $f_n \ll f_t$ as the resistance is much larger in crossflow than in the preferred direction $\hat{n}$.
- Flow in a plane normal to the direction specified by $\hat{n}$. This situation occurs between parallel plates. Here, $f_n \gg f_t$ as the resistance is much larger in the preferred direction than in crossflow.

The above formulation has been implemented in CHAD, and an initial test computation has been performed.

Applications of COMMIX in the Chemical Industry

For many years, the Section has been concerned with models of complex flows within large vessels and between interconnected vessels of various geometry. Almost all of these simulations were related to nuclear reactor safety problems; however, in terms of the generic class of components for which the COMMIX code was designed, such simulations could apply equally well to process simulation problems in the chemical industry. In an attempt to have that industry consider COMMIX as a tool for process simulation, the Section has performed several sample calculations related to flow and mixing in chemical reaction vessels. The figure on the next page, typical of these simulations, shows the flow distribution within a specific crystallizer of interest to a potential industrial collaborator.

The issue of interest in this simulation was how certain internal geometry changes such as the distance between the lower head of the vessel and the downcomer would alter the upflow of the product stream. In particular, there was concern that internal jetlike flows would not be created, thus altering the composition of the product being drawn off the vessel at a point near the lower head. These detailed 3-D plots of the flow field were used by the industrial partner to select the proper geometric parameters for an optimal process flow.

Instantaneous flow field in crystallizer tank

Recent Work by the Analytical Thermal-Hydraulics Section

Publications

Enhancement and Suppression of Turbulence in Particulate Flow

M. Bottoni, F.C. Chang, J. Ding (Energy Research Corp.), and R. W. Lyczkowski
Presented at 1996 ASME Fluids Engineering Division Summer Mtg., San Diego,
July 7-11, 1996.

Thermal Diffusivity Imaging for Processing Evaluation of Continuous Fiber Ceramic Composites

W. A. Ellingson, T. E. Easler (Dow Corning), A. Szveda (Dow Corning), J. G. Sun,
J. B. Stuckey, and S. Thompson (Iowa State U.)
INVITED presentation at Nondestructive Evaluation Techniques for Aging
Infrastructure and Manufacturing, sponsored by International Society for Optical
Engineering, Scottsdale, AZ, Dec. 2-5, 1996.

Laser-Based Optical Scattering Detection of Surface and Subsurface Defects in Machined Si₃N₄ Components

J. G. Sun, M. Shirber, W. A. Ellingson, and M. H. Haselkorn (Caterpillar Inc.)
Presented at 21st Annual American Ceramic Society Conference, Cocoa Beach, FL,
Jan. 12-16, 1997.

Application of Optical Scattering Methods to Detect Damage in Ceramics

J. G. Sun, W. A. Ellingson, J. S. Steckenrider, and S. Ahuja
Chapter in Machining of Ceramics and Composites, Marcel Dekker, Inc. (1997).

Physical Modeling and Numerical Simulation of Subcooled Boiling in One- and Three-Dimensional Representations of Bundle Geometry

M. Bottoni, S. Ahuja, and R. Lyczkowski
J. Nucl. Sci. & Technol., Vol. 34, No. 1, pp. 30-40 (Jan. 1997).

Two Phase Pressure Drop Multipliers and ICE Technique Applied in a Multibubble Slug Ejection Model

M. Bottoni and S. Ahuja
J. Nucl. Sci. & Technol., Vol. 33, No. 12, pp. 964-972 (Dec. 1996).

Development of Nondestructive Characterization Methods - Task 2.3.3

W. A. Ellingson, J. G. Sun, J. B. Stuckey, S. Thompson, and T. A. K. Pillai
INVITED presentation to CFCC Review Mtg., Washington, DC, Dec. 18, 1996.

Development of Nondestructive Characterization Methods - Task 2.3.3

W. A. Ellingson, J. G. Sun, J. B. Stuckey, D. A. Creech, and T. A. K. Pillai
INVITED presentation to Annual CFCC Working Group Mtg., San Diego,
Oct. 16-17, 1996.

Nondestructive Characterization of Ceramic Joints - Some Initial Results

W. A. Ellingson, J. G. Sun, J. B. Stuckey, M. A. Ortner (DOE), D. A. Creech, and
S. Thompson

Presentation to Annual BES/CFCC Working Group Mtg., San Diego, Oct. 18, 1996.

Correlation of NDE Data with Polymer Impregnation Processing

W. A. Ellingson, T. E. Easler (Dow Corning Corp.), A. Sweda (Dow Corning Corp.),
J. G. Sun, J. B. Stuckey, and S. Thompson

Presentation to 21st Annual Conference on Ceramic Composites, Materials and
Structures, Cocoa Beach, FL, Jan. 26-31, 1997.

Electromechanics and Superconductivity Applications

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The Electromechanics and Superconductivity Applications Section conducts research and development on new applications involving magnetics, electromagnetics, electro-dynamics, superconductivity, and their support technologies. The work is both experimental and analytical.

Section projects are funded by the U.S. Department of Energy and industry sponsors, with most projects funded through cooperative R&D agreement (CRADA) relationships. The Section is presently receiving substantial funding from Commonwealth Research Corporation (CRC) to develop a flywheel energy storage device that uses superconducting magnetic bearings. Several of the Section's projects are funded by the Superconductivity Technology Center at ANL; such projects are conducted with industrial collaborators, typically with each institution funding its own work but with staff from each institution working on the same project. Section members are thus frequently involved with industrial scientists and engineers in these collaborative programs. The Section collaborates with the ET Ceramics Section and with the Materials Science Division in the superconductivity program.

Flywheel Energy Storage

Past work in the Section explored the use of high-temperature superconductors (HTSs) in magnetic bearing applications, and a number of techniques were discovered in order to realize an HTS bearing with extremely low rotational loss. We have demonstrated such a bearing with a rotational drag that is more than two orders of magnitude lower than that of a conventional magnetic bearing and several orders of magnitude lower than that of mechanical bearings. With this bearing, an energy-efficient flywheel could be constructed with a bearing loss of <2%/day. Such a flywheel has potential for use in diurnal utility energy storage or as a replacement for batteries in an electric vehicle. Several patents on

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this bearing/flywheel concept have been granted and several more are in process. The HTS bearing work, as a result of substantial funding from and collaboration with CRC, has evolved into a flywheel energy-storage program. As part of this project, collaborations have been developed with a number of companies that are potential subcontractors for commercial manufacturing.

An important vacuum-chamber apparatus in this program is a glass bell-jar test chamber with an oil-diffusion pump. This apparatus is capable of testing bearings with rotating mass of up to ≈ 1 kg. In the past year, this apparatus has been used to test an Evershed-type HTS bearing, i.e., an HTS bearing in which most of the levitation is accomplished by an attractive force between a stationary permanent magnet and the permanent magnet rotor, while the HTS is used to provide stiffness and stability. A coefficient of friction of 10^{-8} has been obtained with this bearing. Experiments with this type of bearing have shown that velocity-dependent losses in such systems can be almost completely eliminated. Another novel HTS bearing, called the mixed- μ bearing, was tested in this apparatus and has demonstrated coefficients of friction of 10^{-9} , two orders of magnitude lower than the conventional HTS bearing.

A second apparatus, shown in the adjacent figure, is a stainless steel vacuum chamber, equipped with a turbomolecular vacuum pump, that is capable of testing rotating masses of >100 kg. A steel-reinforced concrete pit has been modified to safely test flywheels with rotational energies of 1 kWh and higher. We have spun 10-kg rotors to 20,000 rpm in this chamber. We also test laboratory prototypes of commercial-size flywheels in this apparatus and are presently testing rotors with mass of 170 kg.

A third vacuum-chamber apparatus is a burst chamber, in which we use HTS bearings to spin small rotors and components of larger rotors to destruction. These experiments allow us to find the failure modes for various flywheel designs, help determine the maximum safe speed for our larger rotors, and help to validate various stress models that have been employed to analyze the flywheel rotors.

Stainless steel vacuum chamber for testing rotating masses of more than 100 kg at high rotational speeds

A fourth apparatus, presently under construction, uses part of the burst chamber to form a stand-alone flywheel unit. This apparatus has a heavy steel shell capable of confining a failure of a 5-kWh flywheel. The intent of this apparatus is to form a fieldable flywheel energy storage unit that can be tested on the utility grid.

Advanced HTS Bearings

Using DOE funds, we are exploring advanced bearing concepts in the bell-jar test chamber. The first is a new geometry in which a thin-film HTS is placed above a bulk HTS. The thin-film HTSs have higher current densities than bulk HTSs and should shield the bulk HTS and therefore reduce rotational losses. The second concept is to use the low rotational loss to make a low-friction platform on which to accurately measure the gravitational constant $G = 6.67 \times 10^{-11} \text{ Nm}^2/\text{kg}^2$. We believe that this concept has the potential to measure this fundamental constant with an order-of-magnitude greater accuracy than other methods.

Superconducting Motor/Generator

As part of our effort in the DOE superconductivity program, we have investigated novel motor and generator topologies that take advantage of the properties of HTSs. In particular, designs should acknowledge the decrease in effectiveness of iron in reducing magnetic reluctance at the high magnetic fields expected to be used in practical HTS devices. In addition to conventional superconducting motor and generator designs, we examined permanent magnet motors and hysteresis motor work, especially recent work in HTS hysteresis motors. From these studies, we developed a novel concept that can be used as either a motor or generator. The concept uses a stator with HTS windings, preferably without iron. The rotor consists of bulk HTS used in the hysteresis or trapped-field mode. The novelty of the concept is derived from its special geometry, and the concept appears to have advantages in specific power over other superconducting motor and generator designs, in that a maximum torque is produced in motor mode for a given amount of superconductor. The concept also has the lowest demagnetization field for the bulk HTSs. A patent has been filed for this invention, and development is continuing via a collaboration with Boeing Defense and Space Group, Everson Electric Corp., and Superconducting Components, Inc.

A general model of bulk HTS magnetization behavior has been developed that takes into account hysteresis behavior. The model applies a concept analogous to that of thermal annealing to the HTS in order to predict where the current density will flow inside the bulk HTS. This model will be essential in optimizing the HTS configuration for the motor. In FY 1997, the basic features of this model were developed and demonstrated. In FY 1998, the model will be applied to the motor design.

To identify many of the problems associated with the HTS motor/generator, we developed a working prototype of an HTS hysteresis motor. The rotor, consisting of a sintered BSCCO tube was submerged in liquid nitrogen. The stator was a copper winding with iron yoke from a conventional synchronous motor. The bearings were at room temperature.

Fault-Current Limiter

HTS fault-current limiters act as nonlinear resistors and reactors and have diverse applications in electric power systems. Previously, in collaboration with Illinois Superconductor Corporation, we investigated a resistive fault-current limiter design. In a separate effort, the Section has also investigated several inductive-limiter designs in which the HTS is used to magnetically shield a ferromagnetic material. Several small-scale models of this type of limiter have been demonstrated.

Electromagnetic Casting

Previously, the Section worked with Inland Steel Corporation through a CRADA to model the use of electromagnetic (EM) edge dams in steel casting. The computer code ELEKTRA was used to calculate the eddy currents induced in the molten metal, and the results were then used as input to another ANL computer code (CaPS) that predicts fluid flow with free surfaces. Flow velocities and shape of the free surface are the primary results. The basic idea of the EM edge dam is the use of an EM field to support a wall of liquid metal at the ends of the rolls. Conventional mechanical dams have significant problems that have prevented the use of twin-roll technology for some time. Using patented technology originally developed at ANL, Inland has developed the EM edge dams to near-commercialization. We successfully modeled the EM fields and fluid flows of a static test rig, with excellent agreement found between the model predictions and experimental data provided by Inland. This work has evolved into a Work-for-Others program, completely funded by Inland, in which the model is being further developed, and predictions for new experimental apparatus are provided. The development will include solidification and the dynamics introduced by the moving twin rolls.

Electromagnetic Forming of Aluminum

A new technology that is being pursued by the Section is the use of pulsed EM fields to enable high-velocity forming of sheet aluminum for automobiles, containers, and other commercial shapes. High-velocity EM forming holds significant promise for making complex sheet aluminum components. Key advantages of this forming method include forming limits that are improved over those of quasi-static forming, use of one-sided dies, minimization of springback, rapid cycle times, and reduction of environmental impacts. When induction heating is included, it is believed that EM forming can be used to produce magnesium parts. The Section has written a number of proposals in collaboration with The Ohio State University to pursue this technology.

Recent Work by the Electromechanics and Superconductivity Applications Section

Publications

Experimental and Analytical Investigation of Dissipation and Heating in a YBCO Filament
S. Y. Seol (Chonnam Natl. U., Korea), T. R. Askew (Kalamazoo College), and Y. S. Cha
Applied Superconductivity, Vol. 4, No. 5/6, pp. 231-240 (1996).

Flux-Flow Resistivity of Three High-Temperature Superconductors
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Sensors, Instrumentation, and Nondestructive Evaluation

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The Sensors, Instrumentation, and Nondestructive Evaluation (SI&NDE) Section conducts research and development for a broad range of energy-related technologies. Major areas of responsibility are the development of instruments and NDE techniques for materials under development for fossil energy, conservation, and nuclear technologies. Another strong area for the Section has been the development of sensors and technologies for arms control and treaty verification. We have been actively involved in a number of initiatives, including AMTEX, AUTO, Manufacturing, Industries of the Future, and others related to the general competitiveness of American industry. The projects described below give a clear indication of the breadth of our work.

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Arms Control

Pulse Compression Radar for Detection of Subsurface Anomalies

Under an advanced concept project funded by the DOE Office of Nonproliferation and National Security, a feasibility study is being carried out to determine the potential for a specialized ground penetrating radar system to detect buried anomalies. The proposed system utilizes a novel pulse-modulated transmitter and a matched filter receiver for optimal detection of reflected energy from subsurface targets. A computer code has been developed to simulate electromagnetic wave propagation in a multilayered media (e.g., earth's crust) and to estimate the radar return (back-scattered) signals of buried targets of different geometries and depths as a function of incidence angle and pulse waveform. Based on this model, modern signal processing techniques are also being examined to determine optimal signal compression and detection methods.

Millimeter-Wave Detector

This program is funded by the DOE Office of Nonproliferation and National Security, Office of Research and Development, and is in its seventh year of development. The objective is to develop a millimeter-wave (mm-wave) sensor for standoff detection of effluent chemicals associated with the production of chemical and nuclear weapons of mass destruction. The technique uses a swept-frequency radar technique to measure the unique molecular (rotational) absorption signatures. Unlike optics, the mm-wave technique is highly immune to interference from common atmospheric species such as water vapor, oxygen, and carbon dioxide and offers better transmission properties under adverse atmospheric conditions.

In this project, we have developed a unique open-path mm-wave spectroscopy system in the 225-315 GHz atmospheric window. The system is essentially a monostatic swept-frequency radar consisting of a mm-wave sweeper, hot-electron-bolometer detector, and a trihedral reflector. The heart of the system is a Russian backward-wave oscillator (BWO) tube (model OB-30) that is tunable over 225-350 GHz. A mm-wave sweeper has been built with the BWO tube for sweeping the entire frequency range within 10 ms. The chemical plume to be detected is positioned between the transmitter/receiver and the reflector. Millimeter-wave absorption spectra of the plume chemicals are determined by measuring the radar return signals with and without the plume in the beam path. Because of power supply noise and thermal instabilities of the BWO structure with time, the BWO frequencies fluctuate between sweeps which can cause errors in baseline subtraction. To mitigate the frequency jitter problem, a quasi-optical Fabry-Perot cavity has been built in conjunction with the radar system for on-line calibration of sweep traces, which allow excellent baseline subtraction and signal averaging.

The mm-wave system was field-tested in May 1997 in a wind tunnel at the Nevada Test Site, with a standoff range of 60 m. The wind tunnel is capable of producing well-characterized open-air plumes (2 m diameter at the exit point) with various concentrations of chemical vapors. Four chemicals (methyl chloride, butanol, HCl, and dodecane) were selected by DOE and released at different concentrations, individually as well as in combinations. Two national laboratories, Argonne with the mm-wave sensor and

Lawrence Livermore with a passive infrared sensor, participated in the tests. In terms of absorption properties, the four chemicals can be characterized as follows: methyl chloride is highly absorbing with narrow spectral lines; butanol is weakly absorbing with broad spectral features; HCl has frequency spectra outside the system range, and dodecane is practically nonabsorbing. Consequently, we concentrated on detecting only methyl chloride and butanol.

The mm-wave sensor was set up in a trailer (see figure below) 60 m from the wind tunnel in the cross-wind direction. A 0.9 m corner cube was mounted behind the plume for return of radar signals. For safety, the trailer was not manned during the release, leaving the radar system operating autonomously during the entire test duration (typically 6 hr). Data were collected remotely from the wind-tunnel control room 1.7 km away via an RF link between a master computer in the control room and the data-acquisition (slave) computer in the trailer, in a master-slave fashion. The system worked very well in the desert field environment of high winds (up to 10 m/s), heat ($\approx 95^{\circ}\text{F}$), and dust clouds. While detailed analysis of the data is still ongoing, the preliminary data suggest that both methyl chloride and butanol were detected to concentration levels of 40 and 400 ppm, respectively.

*Preparing for the field test of the millimeter-wave remote
chemical sensor at the DOE Nevada Test Site*

Millimeter- and Submillimeter-Wave Sweeper and Gas Analyzer

This project is a Cooperative Research and Development Agreement (CRADA) between Argonne National Laboratory and the Clean Air Engineering Company, in partnership with two Russian institutes (Institute of Applied Physics and Istok Research and Production Corporation), under the auspices of the DOE Initiatives for Proliferation Prevention (IPP) program. The objective is to develop a mm- and sub-mm-wave gas analyzer using the unique Russian backward wave oscillator (BWO) technology. Gas analyzers are typically used to quantify molecular constituents in gas samples, thus having broad commercial value in the environmental compliance monitoring and pollution control industry. A fast-scan millimeter-wave gas analyzer, proposed in this project, will allow better sensitivity and specificity for molecular detection than possible with current systems.

For determining the gas analyzer requirements, a laboratory system was assembled and demonstrated in September 1995 at the Clean Air Engineering company in Palatine, Illinois. The company built a test rig with a gas cell in which the mm-wave system was tested with premixed gas samples. Based on the results, requirements specifications were defined for the operating pressure range, detection sensitivity, molecular specificity, and speed of analysis. A prototype system is being built, along with a multicomponent gas analysis software. The components of the analyzer include a fast-scanning BWO sweeper in the 220-350 GHz range, a quasi-optical frequency measurement system, a gas cell with vacuum pump and gas feed lines, and data acquisition and gas analysis software.

Passive Millimeter-Wave Imaging of Thermal Signatures

This is an advanced concepts project funded by the Office of Nonproliferation and National Security. The purpose of this project is to investigate the use of millimeter-wave radiometer technology for passively imaging thermal and chemical signatures of unannounced weapons of mass destruction (WMD) production facilities. The technique senses temperature and molecular emissions of effluents released to the ambient. Brightness temperatures of hot effluents such as groundwater and gaseous plumes will be measured remotely and mapped against terrestrial backgrounds. The mm-wave results will be compared with that of an infrared system. The passive mm-wave system is compact, battery powered, and can operate under all-weather conditions from a remote platform such as an aircraft or a satellite.

A passive focal-plane imaging system has been built in the 160-168 GHz range. The system consists of a 160-168 GHz radiometer with 16 channels of 500 MHz bandwidth each. Brightness temperatures of objects in the far field were imaged by scanning with a conical horn antenna at the focal plane of a 6-in. lens. Clear images of test scenes with different objects such as sand, a metal plate, and water were obtained with the system. The image contrast of these objects, which were all at ambient temperature, is due to differences in emissivity. The ability to detect thermal signatures such as that of hot water effluents was tested by imaging cups of cold and hot water. Increased signal intensity was observed with increase in water temperature. The laboratory system will be mounted on a cart next to test the effect of sky radiation, atmosphere, and target range on the remote detection and imaging capability of terrestrial targets.

Micro-Mass Spectrometer

This program is funded by the DOE Office of National Security and Nonproliferation NN-20. The goal is to develop several mass spectrometers to support the intelligence community's efforts to combat the proliferation of weapons of mass destruction and to safeguard national security from terrorist threats. Under this program, several time-of-flight mass spectrometer (TOF-MS) systems are being developed. Each system meets unique requirements and scenario objectives requested by the intelligence community.

Under the TOF-MS program, ANL is developing two highly miniaturized MS systems that are specifically designed to meet specific portability and utility requirements for in-field applications while detecting materials associated with nuclear production, enrichment, and proliferation. These systems are highly miniaturized, linear, or reflectron

TOF mass spectrometers. They will ultimately be highly portable, weighing 15-18 lb and occupying no more space than a notebook (approximately 4 x 9 x 11 in.). The spectrometer will be capable of multiphoton ionization, direct sampling of volatile, membrane inlet sampling of solutions, and photodesorption of low volatile chemicals.

The unique features of this system include a highly miniaturized (20 cm long) linear or linear reflectron ion flight tube and advanced ion detector technologies. Ionization is via an Nd-YAG laser system for MPI. Despite the radical departures from conventional mass spectrometer technologies (which may require 1 m flight tube designs), we have demonstrated that these designs operate with performance specifications similar to those of their much larger laboratory counterparts. Currently, we are developing a fieldable demonstration unit. This system will be employed for on-site inspections or placed on-site for remote automated monitoring.

In a cooperative venture with the Pacific Northwest National Laboratory (PNNL) and Los Alamos National Laboratory (LANL), ANL is also developing two additional TOF-MS systems. These systems will be integrated with other components to be developed at PNNL and LANL to provide two unique conjugated analytical instruments. For example, in conjunction with PNNL, ANL is developing a rapid TOF-MS system for application to a supercritical extraction-GS-GS-TOF-MS system. This system is being designed to monitor the development of chemical weapon precursors and agents at ultra-trace levels and extreme molecular specificity. In addition to the specificity achieved through the extraction-GC-GC front end components, the MS system is being designed to provide multidimensional MS capabilities that employ fragmentation analysis of individual molecular signatures as a function of variable electron ionization energy. This technology is useful for detecting the manufacturing and application of agents from extreme distances.

In association with researchers at LANL, ANL is beginning to develop a TOF-MS system for application to a new ultra-high-efficiency ion generator currently under development at LANL. This system is ideal for isotopic analysis of Pu and U metals at trace levels. This technology is designed to meet the rapid intelligence communities requirements for rapid in-field analysis of complex metal system.

Tracking Treaty-Limited Components with Commercial Technology

This project is developing a system to monitor the movements of treaty-limited items and transport vehicles by determining and transmitting the locations of treaty-limited-item movements throughout eastern Europe and the former USSR. The commercial aspect of this technology provides an openness that will contribute to acceptance of this system for movement surveillance by the treaty participants. Analysis of the surveillance is used to verify treaty compliance. Supporting systems are being utilized to complete the technology. Secure tags for treaty-limited components have been developed by Lawrence Livermore National Laboratory and are being adapted for this project through a cooperative effort. When a tag attached to an item is tampered with, information about the item and its location is sent by the system operators to DOE TRANSCOM headquarters in Washington, DC, via established commercial service for satellite communication.

Arms Control and Treaty Verification

Civilian reactors could be used to generate weapons-grade fissile material and to carry out deceptive practices in the dismantling of weapons in order to conceal fissile material. Detecting such clandestine activities is very difficult, and DOE is playing an important role in developing methods for checking compliance with the Nuclear Non-Proliferation Treaty. The SI&NDE section is addressing this issue by applying the use of pulsed neutrons to isotopic analysis by monitoring neutron-energy-dependent prompt gammas during irradiation. Resonance-resolved prompt-neutron-capture gamma ray spectroscopy is a relatively untried technique in which prompt capture gamma ray spectra are recorded simultaneously with TOF neutron energy analysis. The wide energy spectrum of ANL's Intense Pulsed Neutron Source (IPNS) allows the gamma spectrum from many different neutron energies to be studied. Experiments were carried out during the past year to study prompt gammas generated by an IPNS neutron beam.

Environmental Management

Ultrasonic Sensors for In-Situ Monitoring of Physical Properties

This project was initiated in FY 1996 and it is funded under characterization, monitoring, and sensor technologies of EM-50 for high-level nuclear waste treatment. The objective of this project is to develop ultrasonic sensors for in-situ monitoring of physical properties of radioactive tank waste. Initial focus of this project is on development of sensors for fluid viscosity and solid concentration measurements. A feasibility study was completed in FY 1996. In FY 1997, a prototype instrument for measuring viscosity, density, and solid concentration will be built and tested during the sensor comparative evaluation test that will be conducted at ORNL.

Development of a Magnetic Resonance Monitor for ^{99}Tc Column Breakthrough

In support of the U.S. Department of Energy's efforts to privatize the cleanup of nuclear material processing facility waste sites at Hanford and Savannah River, efforts are underway to develop a ^{99}Tc on-line processing sensing and control system. The sensor, based on magnetic resonance spectroscopy of the ^{99}Tc nucleus, will monitor feedstocks and elutants from ion exchange and electrolytic preconcentration processes. This program is funded by DOE's Environmental Management Cross-Cutting Technologies area.

Magnetic resonance spectroscopy of ^{99}Tc is ideal for on-line monitoring of waste tank cleanup processes. The technique is intrinsically nonintrusive, generally immune to contaminants, is capable of operating over large deviations in pH and salinity, and is immune to high background radiation. Additionally, the ^{99}Tc nucleus is 100% isotopic-abundant, is the fifth most sensitive magnetic resonance isotope, and provides approximately 40% of the sensitivity of the proton. The isotope also has a fast nuclear relaxation time, allowing analysis measurements in near real time. Moreover, the high isotopic spin number of 9/2 provides spectroscopic peaks with narrow line-widths that are

easily detected. Furthermore, the nucleus has an exquisite chemical shift dispersion (over 6000 parts per million), thus providing excellent speciation among ^{99}Tc oxidation states and molecular structures.

The sensor will be modeled after ANL's on-line flow-through magnetic resonance sensor technology that is currently being developed for on-line sensing and quantification of organic components. When complete, the spectrometer will incorporate a permanent magnet, a highly miniaturized electronic package, an intelligent operating system, a remote setup and operation panel, and be enclosed in a 19-in. NEMA 4 instrument rack. The system will be capable of monitoring ^{99}Tc at $10\ \mu\text{Ci/L}$ (beta decay with a half-life of 2.14×10^5 is equivalent to 15 micromolar). Efforts in FY 97 include (a) modification of ANL's magnetic resonance process controller, (b) demonstration of sensor performance on ^{99}Tc , and (c) issuance of a deployment plan for full-scale operation. FY 98 efforts will include (a) interaction with the user to develop operation procedures, (b) deployment of the sensor system, (c) performance validation, (d) technology transfer, and (e) report documentation.

Fossil Energy

Development of Nondestructive Characterization Methods for Ceramic Materials

This work is part of the overall materials technology development effort within the Fossil Energy Advanced Research and Technology Development (AR&TD) area. This part of the work is focused on development of NDE methods for a broad range of ceramic materials under development for fossil energy systems. Of current technological interest are (a) continuous fiber ceramic matrix composites (CFCCs) for filters used in hot-gas stream clean up, combustor liners for gas-fired high-temperature turbines and high-temperature aggressive-environment heat exchangers; and (b) thermal barrier coatings (TBCs) for protection of metal blades and vanes in hot sections of gas-fired high-temperature turbines.

We are developing several NDE methods to address issues relative to utilization of these materials systems in fossil energy systems. First, we are developing 3-D X-ray computed tomographic imaging (CAT scanning) for mapping axial and radial density variations in CFCC materials under development for hot-gas filters. This is important to process development. Known as X-ray densitometry, this technique allows total nondestructive measurement of density variations along the length of filters, as well as radially at any axial position. Because filter effectiveness is controlled by porosity, knowledge of the closure state of the porosity provides information on filter effectiveness. The method under development at ANL thus allows study of back-purging necessary to maintain clean filters in use. Second, we are developing methods to evaluate the integrity of ceramic-ceramic joints. For many applications, the CFCC materials must be joined to either monolithic ceramics or to metals. We are developing NDE methods to assess the quality of the initial joint, as well as the state of the joint after operation in service. Acoustic resonance, thermal imaging, acoustic emission, air-coupled ultrasonics, and other approaches are being evaluated for this application. Third, as part of our thermal barrier

coating (TBC) development, we are evaluating elastic-optical scatter methods using low-power visible-wavelength lasers and special optical detectors to characterize the TBC materials.

On-Line Nuclear Magnetic Resonance Monitor

This is a CRADA funded by DOE Fossil Energy (Bartlesville Program Office) between Amoco Corporation, Oxford Instruments USA Inc., and ANL. The purpose is to develop, demonstrate, and transfer technology for use with on-line nuclear magnetic resonance (NMR) sensing and control technology in upgrading light oil residuum and heavy oil. Goals include (a) development of on-line NMR sensor technology (NMR pulse techniques and prototype), (b) implementation of the NMR sensing and control system prototype in a plant environment, (c) implementation of this technology for a variety of upgrading processes and characterization needs (for both residuum and heavy oil) over the lifetime of the proposal, and (d) transfer of the technology to Oxford for commercialization.

The NMR sensing technology provides genuinely enabling near-term technology for on-line characterization of residuum and heavy oil properties, in order to predict processability and improve upgraded products yields, as well as for efficient process control. Unlike other process-control sensing systems (such as gas chromatography, laser chromatography, Fourier transform infrared, near-infrared, and mass spectroscopy), the NMR technology can be used to monitor the chemical and physical properties of optically diffuse liquids, semisolids, and solids. This technology is directly applicable to many of Amoco's heavy oil upgrading needs. For example, in residuum and heavy oils, NMR measurements can be quantitatively correlated with a variety of properties such as viscosity, molecular weight distributions, aliphatic/aromatic ratio, and presence of impurities.

The feasibility of applying NMR spectroscopic techniques to process sensing and control is being evaluated via laboratory experiments. To date, we have seen excellent sensitivity to impurities (e.g., water) and to specific gravity variations in the residuum. Additionally, results have indicated that sufficient signal-to-noise ratio will be available for both on-line ^1H and ^{13}C NMR spectroscopic monitoring in a plant environment. We are developing a prototypical instrument (now nearly complete) for in-plant operations. The unit is contained in an environmentally safe industrial 19-in. rack unit. Final testing of the spectrometer is in process. We will soon evaluate the performance of the sensor system through field trials at the Amoco facilities.

Energy Conservation and Renewable Energy

Part I: Office of Advanced Transportation Technologies

Development of Nondestructive Characterization Methods for Ceramics (Office of Heavy Vehicle Technologies)

The work in this project is directed toward two NDE research areas: (a) development of fast, reliable, 3-D X-Ray CAT scanning for mapping density variations in chopped carbon fiber/silicon nitride (Si_3N_4), which is under development as a material system for new

low-friction valve guides in diesel engines, and (b) development of a fast on-line elastic-optical scatter method to detect machining-induced damage in monolithic Si_3N_4 . Both projects are cooperative efforts with the Caterpillar Inc. Research Center in Peoria, IL. The X-ray densitometry work is also cooperative with AlliedSignal Ceramic Components of Torrance, CA.

New high-dynamic-range X-ray detectors (12 to 16 bit) and new digital image processing methods have allowed totally nondestructive densitometry studies that suggest a variation in density of <2% can be mapped in ceramic components (see figure below). For the valve guides, the chopped carbon fiber loading ranges from 10-12 vol.%. Destructive measurements carried out by AlliedSignal Ceramic Components have been correlated to the nondestructive measurements made at ANL. These correlations verify the 2% detection sensitivity.

New high-speed data acquisition systems coupled with new digital image processing developments here at ANL have allowed the fast elastic-optical system to be greatly advanced. Our work is establishing the correlation between the NDE data with microstructure and mechanical properties. Two Si_3N_4 materials are being studied. The machining parameters being varied include material removal rates and grinding wheel composition. Also, high-speed rotary ultrasonic machining is being studied for very fast material removal rates. The elastic-optical scatter method (next figure) has shown clear detection of subsurface machining damage as a function of machining parameter, and now determination of the proper elastic-optical scatter parameter to use for correlation with mechanical properties is underway. Correlation with microstructure has been initially established.

X-ray computed tomographic imaging (CAT scan) used for densitometry analysis on complex-shape ceramic turbine rotors. Graph shows image gray scale as a function of volume percent organic binder.

(a)

(b)

(c)

(d)

Elastic-optical scattering system: (a) system diagram; and typical scattering images for diamond-ground specimens: (b) ratio image, (c) sum image, and (d) surface plot of sum image in (c)

NMR Spectroscopy and Imaging of Advanced Ceramics and Ceramic Composites

The objective of this work is to develop and evaluate advanced NDE technology for characterization of the chemical and physical properties of advanced ceramics and ceramic composites. Working with Dr. O. O. Omatete of the Materials and Ceramics Division at Oak Ridge National Laboratory, we will evaluate the use of NMR in aiding the process development and on-line production of gel-cast composites. Nondestructive NMR imaging and spectroscopic studies will enable visualization of the polymerization process, calculation of polymerization rates, and measurement of viscosity within the component. Additionally, imaging methods will be employed for flaw determination, and the results will be correlated with those obtained by destructive analysis and fracture mechanics.

Part 2: Office of Industrial Technologies

Development of Nondestructive Characterization Methods for Ceramics Advanced Turbine System (ATS)/Ceramics in Stationary Gas Turbines

As part of the Office of Industrial Technologies (OIT) Program on Advanced Gas Turbines (ATS), ANL is a subcontractor to Solar Turbines, Inc. (a wholly owned subsidiary of Caterpillar Inc.) of San Diego, CA, which is developing and evaluating ceramics for application to stationary gas turbines. The ANL effort is directed toward new NDE methods for the ceramic materials being developed and tested for the Centaur H gas turbine.

The evaluated ceramic materials include monolithic materials for turbine blades and nozzles and ceramic composite (CFCC) materials for combustor liners and transition ducts. The monolithic materials are Si_3N_4 , and the CFCC materials are both nonoxide (SiC/SiC) and oxide ($\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$). The ANL NDE effort has included the development of (a) methods to determine thermal properties (thermal diffusivity) of the CFCC materials and conduct full-field mapping of thermal properties, (b) methods to detect delaminations of the CFCC materials, (c) methods to detect surface cracks and subsurface cracks in the monolithic materials, and (d) determining whether NDE data can be correlated with predicted component performance.

We have made significant progress on developing NDE methods for this project. The thermal imaging method based on measurement of thermal diffusivity is now the NDE method being used to accept/reject components for use in the gas turbine. Full-sized liners (13 and 30 in. in diameter and 8 in. long) have been successfully studied by ANL with the thermal imaging method. Tests have been conducted before service and after 100 hr of engine test time. The components are now scheduled for 1000 hr of testing. By use of a new, very-high-speed data acquisition system (up to 1900 frames/sec), including a new 12-bit dynamic range camera, one-sided data acquisition is being developed for complex-shaped components. Correlation of infrared imaging with new air-coupled ultrasonic technology has been also demonstrated and will be implemented. We continue to develop the dye penetrant method for examination of monolithic ceramics and this additional effort has led to significant digital image processing that allows new detection sensitivity. Work also continues on acoustic resonance for determining the specific damping capacities of some components.

Continuous Fiber Ceramic Matrix Composite (CFCC) Program

The work in this DOE OIT project, which is part of the Task 2 support technologies, is directed toward development of NDE methods for three specific activities: (a) information for process development/reliability, (b) information for assessment of joint quality and joining process development, and (c) information to assess impact damage. The NDE developments in this project are being directly implemented on the CFCC materials being used on the Solar Turbines project. Work in this project, focused on development of thermal imaging methods and air-coupled ultrasonic development, is highly interactive with the industrial suppliers (Dow-Corning, DuPont Lanxide Composites, Textron, Amercom, and Babcock & Wilcox). In the case of Dow Corning and process development, several special specimens that have been prepared allow study of the thermal imaging and air-coupled ultrasonic relative to processing variables in the polymer impregnation process. Successful correlations were established. Dow Corning and DuPont Lanxide Composites have each prepared and supplied joined specimens to allow NDE development on systematic joining variables; this is new work now underway. Impact damage to SiC/SiC 2-D laminated CFCC material, induced by instrumented pendulum impactors, is being studied by thermal imaging and air-coupled ultrasonic analysis for sensitivity to damage levels; initial correlations are excellent. Joining studies have now begun, with Dow Corning and DuPont Lanxide Composites preparing lap-joint specimens in cooperation with INEEL. ANL has studied thermal imaging methods, air-coupled ultrasonic methods, and acoustic resonance to date. Regions of poor joining have been determined, but correlations of NDE data with mechanical properties must still be determined.

NDE of High-Temperature Ceramic Superconducting Tapes

Measurement of ultrasonic velocity has been used to monitor the fabrication (powder and tube process) of silver-clad superconducting ribbons. This approach is being employed to indicate the presence of a liquid phase during sintering and changes in crystal structure during fabrication. Knowledge about the presence of the liquid phase is very important in optimizing the fabrication process so that the highest possible critical currents can be handled. The system has been automated and continues to be used routinely for characterizing superconducting tapes.

Energy Research

Development of Rapid Prototyping Technology for Ceramics

This CRADA is a joint project with three small businesses; its purpose is twofold: (a) to develop technology that will allow the fabrication of net shape or near-net-shape ceramics with rapid prototyping (RP) technology, and (b) to develop technology to allow CAT scanning image data to be used in reverse engineering for dimensional verification and quality verification of RP-fabricated parts.

Significant developments have been made in this project. First, in cooperation with Ford Advanced Manufacturing in Dearborn, MI, an agreement was reached to allow testing of RP-produced ceramics in injection molding facilities. Net shape ceramic inserts were

produced as part of a special die designed to allow testing of inserts. The complexity of the part shape and the finish of the surfaces necessitated that green ceramic machining be developed for RP-produced ceramics. A precision three-axis milling machine was set up, software selected, and machining parameters established. Machining of both green as-cast and sintered ceramics was required. Two parts were sent to Mexico for full-scale injection mold testing. The ceramic inserts performed well enough to suggest that they should be certified for production, but this was not done. The parts were tested at full production rates for 25 min and no deterioration was observed. Second, work progressed on reverse engineering. The key to reverse engineering lies in the development of an algorithm that will allow data taken from a measurement system, e.g., a CAT scanner, a coordinate measuring machine (CMM), or laser range finder to be formatted for use as the input to solid-model CAD software programs. If automatable, this would complete the ability to replicate parts or obtain CAD designs from some measurement system. One such approach is constructive solid geometry (CSG). An approach was formulated at ANL using the CSG system for the algorithm. At present, the input to the algorithm is an algebraic boundary representation (as opposed to a parametric boundary representation). Algebraic boundary representations can be obtained from measurements with existing software. While not yet coded, the basic algorithm has been developed.

Development of Rapid Prototyping for Bioceramics

This CRADA project is a joint effort with two large businesses, Zimmer, Inc., of Warsaw, IN, the world's largest producer and supplier of orthopaedic devices, and Midwest Orthopaedics of Chicago, a leading orthopaedic surgical group. The purpose is twofold: (a) develop and evaluate RP technology for production of bioceramic prosthesis devices, e.g., small hand bones, and (b) determine the limitations of reverse engineering technology by using high-spatial-resolution CAT scanning). At present, two specific prosthetic devices, small hand bones and a selected forearm bone segment, have been identified for fabrication. Aluminum oxide has been formulated for rapid prototyping, and initial hand bones have been made. New materials of choice are hydroxyapatite/tricalcium phosphate (HA/TCP) because of its potential for surface-porosity control, which in turn should control engraftment time. Problems exist because of surface finish of currently produced materials, but may be overcome via five-axis machining or other surface modification methods. A high-density polymer skeleton model is being used to acquire 3-D X-ray CAT scan data. Using the hand and the forearm bone, we have obtained CAT scans with very high ($<60\text{ }\mu\text{m}$) spatial features. These files have been subjected to digital image processing to allow computer files of the proper format to be electronically transferred to the RP machine, and initial bioceramic components have been produced. We have thus demonstrated almost all parts of the complete reverse engineering loop, leaving only reacquisition of the parts in order to compare dimensional fidelity.

Rapid Prototyping for Heat Engine Components

This is a CRADA project with two small businesses, Advanced Ceramics Research (ACR) located in Tucson, AZ, and BPM Technologies, Inc., of Greenville, SC. The purpose of this CRADA is to (a) establish whether near-net-shape functional structural ceramics (e.g., Si_3N_4) can be produced by RP, and (b) establish whether ceramic cores can be developed for metal turbine blade casting by using high-part-definition RP (e.g., ink jet

printing) technology). ACR is developing a special material delivery system for fused-deposition-type RP machines. The advantages of this material delivery system is that it uses screw feeders for mixing binder and powder, thus avoiding the need for filament-type feedstock that has high development costs and storage problems. Initial feedstock has been prepared. A CAD file of a turbine blade has been received and an initial Si_3N_4 turbine blade has been built. Mechanical properties, surface finish, and dimensional fidelity all must be developed. BPM Technologies is developing an RP machine based on very-high-precision ink jet printer technology. Because ceramic cores used for cast turbine blade production require very high precision, ink jet technology is the clear choice. Remaining to be developed for the BPM machine is the feedstock, an alumina-silica binder system material used for cores. ANL has a BPM machine now and initial development work is proceeding. A special lab benchtop setup has been established to allow development of feed rates and mechanisms. Because of the technology used in ink jet printing, various aspects of the nozzle and piezoelectric transducers, pulse shape, pulse repetition rate, etc. must to be studied relative to material delivery for materials other than the multicomponent polymer normally used with these printers.

Staff Exchanges: CC/TA&T

This is a new project funded by ER/LTT as part of an industrial staff exchange activity. Ceramic Composites of Technology Assessment and Transfer (CC/TA&T) of Annapolis, MD, is developing continuous fiber ceramic matrix composite materials, and this project will evaluate different NDC methods under development at ANL to measure distributed properties, e.g., density, or to detect defects such as delaminations. Staff from CC/TA&T have visited ANL and have evaluated several NDE methods that have been beneficial to CC/TA&T.

Evaluation of Elastic-Optical Scatter for Detecting Damage in High-Removal-Rate Machining of Monolithic Ceramics

This project is funded by ER/LTT and is part of the Industrial Staff Exchange activity. The industrial partner is Caterpillar Inc., which is interested in fast-removal-rate ceramic machining. The purpose of this staff exchange is to give Caterpillar staff "hands-on" experience with the ANL-developed elastic optical scattering system that was developed to detect subsurface defects in monolithic ceramic materials. Caterpillar supplies specimens of various machining conditions and typical surface characterization data. SI&NDE Section and Caterpillar staff then conduct elastic-optical scatter analysis of the specimens. Correlations between the elastic optical scatter data and fracture behavior are then determined.

Computer-Aided Fabric Evaluation

We are helping the U.S. textile industry improve the quality of its products. The national AMTEX program is a collaborative effort between many textile companies and several national laboratories to improve the competitiveness of the U.S. textile industry. This Section is using the expertise and technology developed through other programs to

help increase the technology base for this industry. The most recent effort has been the development and testing of nonvisual systems to detect fabric flaws generated by knitting machines.

Ultrasonic Imaging System for Fabric-Defect Inspection

This work was also carried out under a CRADA between ANL and the AMTEX companies to develop Computer Aided Fabric Evaluation (CAFE) sensors. The objective was to develop an on-loom ultrasonic imaging system for on-line, real-time detection of defects in as-woven fabrics. Using air-coupled transducers, the ultrasonic system is a noncontact instrument that operates in a pitch-catch mode and detects the fabric defects from the variation of the transmitted signals.

A prototype system was developed and successfully demonstrated during an alpha test in FY 1995. Subsequently, development of the required electronics and defect-detection algorithms was completed and the system integration was planned for FY 1996. An industrial prototype was scheduled to be built and tested in FY 1996. The lack of funding in FY 1996 caused the test to be postponed.

Millimeter-Wave Inspection of Fabric Defects

This work was carried out under a CRADA between ANL and the AMTEX companies to develop computer aided fabric evaluation (CAFE) sensors. The objective was to develop an on-loom mm-wave sensor for on-line, real-time detection of defects in as-woven fabrics. Based on dielectric property changes, the mm-wave sensor detects and maps fabric defects in a noncontact manner.

Development of the mm-wave sensor was realized in two phases: (a) proof-of-concept evaluation in the laboratory and (b) proof-of-principle evaluation on a loom, called the alpha test. The proof-of-concept evaluation used a 94-GHz mm-wave imaging system that employed a reflection measurement configuration with a reflector plate behind the fabric being examined. The proof of concept used industry-supplied test fabrics that contained various types of defects and that represented different defect categories, fabric types, and manufacturers.

A prototype mm-wave sensor was designed, built, and field tested on a test loom. It consisted of a mm-wave transmitter, a pair of near-field waveguide probes, and a differential receiver. The sensor was mounted on a scanning platform that provided 10-in.-wide coverage. A plug-in digital signal processor board (DSP) in a personal computer was used to acquire, process, and display both the scanned raw images and the detected flaw regions, all in real time. The system successfully imaged and detected fabric flaws in real time. This sensor was chosen as one of the recipients of the 1996 R&D 100 award.

Sensors for Agile Manufacturing

The purpose of this project is to develop sensors for automation of the servicing processes associated with garment assembly; the results will be improved product quality

and process productivity. The major deliverable will be demonstrations of the sensor technologies and controls as they apply to identified industry needs. Demonstrations will be carried out in both the laboratory and the production environment.

Advanced In-Car Sensing and Control

This is a CRADA project under the United States Council on Automotive Research (USCAR) initiative. The goal is to develop low-cost sensor systems for tailpipe or engine-out emission monitoring. Two types of gas sensors, the surface acoustic wave (SAW) chemical sensor and micro mass-spectrometer (MS) are under development in this project. The initial focus of the project was on evaluation of SAW coating materials and on design and testing of a laboratory micro-MS system. A sensor test facility is being constructed to evaluate sensitivity and sensor performance evaluation. Three polymer films were tested for the SAW sensor. The results showed a lack of sensitivity and repeatability in detecting hydrocarbons, and also indicated that the films cannot survive the high-temperature environment. New high-temperature microsensor technology is now being investigated.

Ultrasonic Viscometer

This is a new CRADA project with Brookfield Engineering Laboratory, Inc. (BEL of Stoughton, MA). The goal is to transfer the ANL ultrasonic viscometer technology to industry. Under this program, ANL will provide technical assistance to BEL in developing an industrial prototype. ANL will also complete the design of the required electronics, evaluate wedge materials for better sensitivity, and optimize the instrument performance. The control electronics have been developed and tested. A spool piece of the sensor assembly has also been built and is being tested.

Work for Others

Application of Neutron Diffraction to Determination of Residual Strains in Engineering Materials

Neutron diffraction techniques using ANL's Intense Pulsed Neutron Source (IPNS) and General Purpose Powder Diffractometer have been employed to measure residual strains in crystalline particles embedded in otherwise amorphous materials, including fiber reinforced composites, with an accuracy sufficient to validate analytical models used to predict mechanical properties. The particles act as "strain gauges" for these materials. The stress-free temperature has also been measured using neutron diffraction by determining bulk strain as a function of temperature (stress-free temperature is needed to predict mechanical properties, and the only practical way to obtain this experimental value is by neutron diffraction). In addition, the coefficient of thermal expansion as a function of crystallographic direction has been measured in composites by neutron diffraction to characterize anisotropic effects. The most recent effort has involved the study of fabrication-induced strain in high-temperature superconducting powder-in-tube wire.

Component Leak Testing

Funded by The National Center for Manufacturing Science (NCMS) in Ann Arbor, Michigan, this new joint project is between ANL and Ford, Chrysler, Vacuum Instrument, the University of Michigan, and AlliedSignal, Inc. The objective of this project is to develop advanced technologies for locating and quantifying leaks as small as 10^{-6} scc/sec in manufactured components such as air conditioning components. Three technologies are proposed from ANL; they are surface acoustic wave (SAW) sensor, millimeter wave (MMW) technique, and time-of-flight (TOF) mass spectrometry. The first-year project goal is to demonstrate the leak detection/locating capability of the proposed technologies. An alpha test will be conducted in the first year (FY 1997) to select two promising technologies for further development. Significant progress has been made for each of the ANL tasks.

Acoustic Leak Detection and Location in District Heating Systems

A collaborative effort continues between DOE, Consolidated Edison of New York, and NRG Energy (a subsidiary of Northern States Power) to develop an acoustic system for locating leaks in underground piping. Results of field testing with acoustic sensors placed on bare pipe indicate that information is present in the acoustic signal that could help locate leaks. However, the use of in-stream sensors appears to have more potential for leak location. Laboratory experiments have been completed in the program to develop an in-stream acoustic microphone. Placing the sensors directly in the stream significantly improves the chances of accurately locating leaks in buried pipes. A valve and sensor system for inserting microphones into underground piping has been developed. Valve and sensor systems have been delivered to both Consolidated Edison and NRG Energy for insertion in their district heating systems. Divisional staff will analyze acoustic signals after these systems have been installed.

Development of Nondestructive Characterization Methods for Ceramic Joints

As part of the DuPont Lanxide Composites (DLC) effort to develop continuous fiber ceramic matrix composites, joining technology becomes important. The purpose of this small project was to determine initially whether ANL-developed technologies for NDE of CFCC materials could be useful for certain aspects of joint interrogation. The ANL NDE methods of infrared imaging, acoustic resonance, and X-ray CAT scanning were evaluated in special test specimens. Results were provided to DLC.

ASME Task Group on Digital Imaging Applications for the Nuclear Industry

In cooperation with the Nuclear Regulatory Commission (NRC), the American Society of Mechanical Engineers (ASME) has organized a task group to develop a code for digitizing industrial radiographic film. Computer storage of the hundreds of thousands of radiographs associated with the construction of a nuclear power plant will save the utilities considerable money and time and will preserve the aging films. The SI&NDE Section

continues to participate in this effort to develop the specifications and standards necessary to faithfully reproduce the ASME-code-acceptable radiographs. The proposed code was approved by ASME in May 1997.

NRC: Steam Generator Tube Integrity Program

A new NRC program has begun on the integrity of nuclear reactor steam generator tubing. Degradation of steam generator tubes is due to corrosion and wastage, pitting, denting, stress corrosion cracking, and intergranular attack. Cracking has been experienced on both the primary and secondary side of the steam generator tubes. Because knowledge about tube degradation in operating plants is derived from in-service inspections, the nondestructive test results and parameters from these inspections must be related directly to the failure pressures and leak rates or to the flaw characteristics that control these pressure and leak rates. The purpose of this program is to produce and/or update information, data, and predictive modeling (see figure below) for the degradation and inspection methods of current interest, i.e., various forms of cracks, multifrequency eddy current test equipment, modern probes and equipment, voltage parameters, and complementary inspection methods such as ultrasonic testing. Cracked tubing samples will be nondestructively tested before being subjected to pressure and leak rate testing.

Classical multifrequency and advanced eddy current tests will be conducted. This new research will allow evaluations of various defect specific management schemes and will support current NRC rulemaking needs and activities. Part of this effort involves the fabri-

*Predictive modeling of eddy current probe response for NDE of steam generator tubing:
distribution of current density with differential bobbin coil in Inconel 600 tube*

cation of a steam generator mock-up that will be used for round robins and assessment of emerging technology to help establish the reliability of inservice inspection and accuracy of sizing defects in steam generator tubing.

Laboratory Directed Research and Development

Millimeter-Wave Sensor for Environmental Monitoring

This program is funded by ANL's Laboratory Directed Research and Development program. The objective is to investigate the potential of the millimeter-wave technique for continuous emissions monitoring (CEM) of industrial effluents. With the enactment of the Clean Air Act amendments in 1990, the need for CEM devices has taken on added importance for a variety of manufacturing industries and waste treatment facilities. Emphasis is on commonly occurring pollutants such as combustion products, volatile organic chemicals, and auto exhaust emissions.

A theoretical analysis was made to compare the mm/sub-mm-wave technique with optical techniques. Using a data base called HITRAN, we calculated detection sensitivities in the mm/sub-mm and infrared (IR) ranges for several environmentally hazardous molecules. For molecules with strong dipole moments, mm-wave detection sensitivity is comparable to or better than that of the IR technique, but for weak-dipole-moment molecules, it can be lower than the IR technique by two to three orders of magnitude. The main advantages of the mm/sub-mm-wave technique, however, are wide-area monitoring, simultaneous measurement of several target chemicals in real time, ease of spectral interpretation, and operability under hostile environments and all weather conditions.

The proof of principle of the active mm-wave technique was tested in air. Using a sweeper in the 75-110 GHz range and a frequency tripler, we generated mm waves at 225-330 GHz. A plume was created by heating D₂O in a beaker and releasing the vapor into the path of the mm-wave beam. A corner reflector on the far side of the plume returned the radiation to a hot-electron-bolometer receiver. Lenses were used to collimate and focus the mm-wave beam. The measurement results showed the D₂O absorption lines clearly, and the line height decreased as the release rate of vapor decreased with time, thus confirming the detection.

Microwave Tomographic Imaging

Under an individual LDRD program, applied research studies are being carried out on implementation of a stepped frequency microwave/mm-wave tomographic imaging system for NDE of dielectric materials. This work involves theoretical electromagnetic modeling and wavefront processing in conjunction with laboratory-based experimental studies. A computer-controlled translation stage and data acquisition apparatus is used to record multiaspect monostatic backscatter (reflection) data from test specimens. Wave-front processing and back projection algorithms are currently being implemented to construct tomographic images samples with varying permittivity profiles.

Individual Investigator Laboratory Directed Research and Development

The objective of this research project is to develop a highly miniaturized (i.e., beverage-can-size) ultrasensitive nanoliter-volume nuclear magnetic resonance (NMR) spectrometer. The attributes of this spectrometer include a high-homogeneity miniaturized permanent magnet system, a magnetic-susceptibility-matched micromachined radio frequency coil and magnetic field shimming stack, and highly integrated control and data acquisition electronics. Designed for a wide variety of in-field sensing applications, the total system (i.e., the entire spectrometer) will weigh less than 5 lb, while providing chemical sensitivity similar to that of many high-magnetic-field laboratory-based NMR spectrometers. Due to size reductions and micromachining of components, the estimated cost per unit is ~\$2500. Applications of this system will include downhole monitoring of groundwater pollutants and flow, in-tank spatial composition profiling, in-field spectroscopic analysis of waste materials, and real-time in-process monitoring of waste remediation activities. Other applications may include medical monitoring systems, industrial process sensing and control, and possibly near-vehicle monitoring at refueling stations. Specific research to date has concentrated on developing the appropriate magnet system through electromagnet modeling. Results indicate that appropriate geometries can be developed to provide the required magnet characteristics while keeping overall magnet dimensions compact.

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Rapid Infrared Characterization of Thermal Diffusivity in Continuous Fiber Ceramic Composite Components

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Thermal, Mechanical, and Chemical Processes

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The charter of the Thermal, Mechanical, and Chemical Processes (TMCP) Section is to (a) perform applied research with the overall objective of developing new and/or improved correlations, prediction methods, and experimental techniques for the design and evaluation of energy-efficient and environmentally acceptable components and processes; (b) facilitate technology transfer to industry via workshops, short courses, cooperative research and development agreements (CRADAs) and work-for-others contracts; (c) perform consulting activities, including diagnostics and resolution of mechanical problems in existing energy systems components, such as flow-induced vibration of nuclear reactor components; and (d) provide technical support to Laboratory and Division activities such as the NRC Steam Generator Tube Integrity Program.

Staff areas of expertise include heat transfer and fluid flow (including phase-change heat transfer, multiphase flow, transient flow and mixing, thermally driven flows, advanced heat transfer fluids, and flow visualization), and structural dynamics and flow/structure interaction (including flow-induced vibration, vibration control, and measuring/monitoring vibration-sensitive structures). Staff expertise and experience are primarily in experimentation and testing, but they also extend to modeling. A particular strength of the Section is its array of general-purpose test facilities and smaller, single-purpose test apparatuses for experiments and evaluation of component designs. These include small-channel phase-change heat transfer test apparatuses (flow boiling, condensation, and flow visualization), water flow test facilities (8,000-gpm water flow loop, 2,200-gpm flow and heat transfer test facility, 500-gpm water flow loop, and a 2- x 4-ft low-velocity water channel), and special-purpose test facilities (shell-and-tube research heat exchanger, ice-water slurry test facility, distillation research column, and endothermic fuels heat transfer test apparatus).

Current and/or recently completed activities include applied research related to the application of compact heat exchangers for the transportation and process industries (development of high-performance condenser and evaporator tubes for automotive and residential air conditioners, improved efficiency and control of distillation, and development of a heat exchanger/reactor for reforming jet fuel to obtain gaseous fuels with improved combustion characteristics), and research in the areas of advanced heat transfer fluids, flow-induced vibration of nuclear plant steam generator tubes, ice slurries for district cooling, and a multiport dryer for the paper industry. A Section staff member is also providing support to the NRC Steam Generator Tube Integrity Program in the areas of

¹Special Term Appointee.

²Serves two sections.

design, fabrication, and commissioning of special-purpose test facilities for the rupture and leak-rate testing of flawed and unflawed tubing under a wide range of conditions.

Flow-Induced Vibration

Because of the existing test facilities and extensive experience in flow-induced vibration at Argonne, the Electric Power Research Institute (EPRI) funded a recently completed three-year program to investigate nuclear plant steam generator tube vibration. The key elements necessary to predict fluidelastic instability of tube arrays in crossflow are fluid-damping and fluid-stiffness coefficients. The objectives of this EPRI-sponsored program were to develop accurate, experimental, motion-dependent fluid-force data and to utilize these data to improve the accuracy and applicability of the fluid-force calculation technique for predicting tube vibratory response and fluidelastic instability. Motion-dependent fluid forces were measured and the unsteady-flow theory for fluidelastic instability of tube arrays in crossflow was completed. Three tube arrangements were tested: tube row, triangular array, and square array. Fluid-damping and fluid-stiffness coefficients were obtained as a function of the reduced flow velocity, excitation amplitude, and Reynolds number. We also developed a mathematical model, based on the unsteady-flow theory, to provide additional insights into fluidelastic instability of tube arrays in crossflow, and performed detailed analysis with mathematical models for various conditions of tube arrays in crossflow. The results, documented in a final report to EPRI, represent the most comprehensive experimental data base that has been published on motion-dependent fluid-force coefficients for tube arrays in crossflow. The results can be applied to model the various types of flow-induced vibration typically encountered in practice, including fluidelastic instability, turbulence-induced vibration, and vortex-induced vibration, and to resolve issues related to steam generator and heat exchanger tube vibration and instability.

Compact Heat Exchangers for Transportation and Process Industries

The Section continues to have an active program in compact heat exchangers for transportation and process industry applications. High-performance condenser tubes for automotive air conditioning applications are being studied in CRADA projects with Modine Manufacturing Company. Another CRADA project with Modine is focused on small-channel evaporator tubes. The application of compact evaporators and condensers as integral reboilers and condensers in a distillation column is being investigated under a CRADA with Xytel Corporation.

High-Performance Condensers

A three-year CRADA project with the Modine Manufacturing Company was successfully completed. Project results provided data on the effects of flow passage geometry and size on condensation heat transfer. The testing was performed in a small-channel heat transfer test apparatus designed and fabricated by Argonne for this project shown in the figure on the next page. A total of 14 different tube assemblies were tested with refrigerant R-12.

Two separate follow-on research projects were subsequently developed for direct funding from Modine. The objective of the first follow-on research project, which has been completed and reported, was to measure average condensing heat transfer coefficients and two-phase pressure drop, in a variety of multiport condenser tubes, with refrigerant R-134a (the replacement for R-12). Data were obtained as functions of mass flux, for mass flux values in the range dictated by the application. In the second research project, condensation heat transfer and two-phase pressure drop data are being determined for three different refrigerants at three saturation temperatures. These CRADA projects have resulted in new/improved design correlations for heat transfer and pressure drop and have led to an improved understanding of automotive air-conditioning systems that use Modine's high-performance multiport condenser tubes.

Small-Channel Evaporator

The focus of our small-channel evaporator research was on a one-year CRADA project with Modine. The work represents an extension of the research sponsored by the DOE Office of Energy Efficiency and Renewable Energy, Thermal Sciences Program, on compact heat exchangers. The goal was to improve our understanding and characterization of the dominant evaporative heat transfer mechanism(s) in small-channel flow boiling, including the effect of flow passage geometry and size. An existing small-channel flow boiling test apparatus was modified for the test phase. Testing was performed with R-134a in a 2.46-mm circular brass tube, and in a 3.23-mm square brass tube. Extensive sets of boiling heat transfer coefficient and two-phase pressure drop data were obtained for three different values of saturation pressure. Data trends were used to identify the dominant heat transfer mechanisms and the ranges over which they are dominant. Nucleation was shown to be the dominant mechanism over a large range of heat flux ($>8 \text{ kW/m}^2$) and wall superheats ($>2.7^\circ\text{C}$). The results of the project were made available to Modine for use in the design of new high-performance evaporators for residential air conditioning and heat pump applications. Previously generated data sets from other tubes and different refrigerants (see table on next page for a summary of this test data base) were also used as the basis for the development of a new correlation for nucleate flow boiling. The new correlation includes a "confinement number," and, as shown in the following figure, predicts the majority of the experimental data from all the data sets (including three different refrigerants) within 15%.

Improved Efficiency/Control of Distillation

The application of compact heat exchangers in the process industries is an objective of the TMCP Section. Conventional distillation, in which heat is input to the column through a reboiler at the base of the column and is removed in a condenser at the top of the column, is inherently very energy-inefficient. Column efficiency can be improved by selectively distributing the heating and cooling along the length of the column. We have partnered with Xytel Corporation on a CRADA project to demonstrate the use of compact heat exchangers as integral heat and mass transfer devices in distillation. The device is located within the column and can serve as an integral condenser in the rectifying section or as an integral reboiler in the stripping section. An important feature of the device is that it provides mass transfer surface area, in addition to providing the local cooling or heating required to reduce the thermodynamic irreversibilities. A 12-in.-diameter, 15-ft-tall

Summary of Test Data Base for Nucleate Flow Boiling
($\Delta T_{sat} > 2.75^\circ\text{C}$ and $x > 0.2$)

Parameter ^a	Test Series			
	1	2	3	4
Refrigerant	R-113	R-12	R-12	R-134a
Channel Material	Stainless steel	Brass	Brass	Brass
Channel Geometry	Circular	Circular	Rectangular	Circular
D (mm)	2.92	2.46	2.40 ^b	2.46
P_R	0.045	0.12, 0.20	0.20	0.10, 0.15, 0.20
G (kg/m ² s)	50-400	63-832	44-505	92-476
q'' (kW/m ²)	8.8-90.8	7.5-59.5	7.7-129	7.9-49.8
Bo	0.00075-0.0023	0.00020-0.0017	0.00028-0.0016	0.00039-0.00081
ΔT_{sat} ($^\circ\text{C}$)	7.2-18.2	2.8-6.6	2.8-8.2	2.8-7.1
No. of Tests	27	104	118	189

^aWhere D is channel hydraulic diameter, P_R is reduced pressure, G is mass flux, q'' is heat flux, Bo is boiling number ($= q''/i_{fg}G$), $\Delta T_{sat} = T_w - T_{sat}$, i_{fg} is latent heat of evaporation, T_w is wall temperature, and T_{sat} is saturation temperature.

^bActual channel dimensions are 1.70×4.06 mm.

*Predicted Nusselt number vs.
experimental Nusselt number from
four test series summarized in the
table (438 data points)*

research column has been designed and fabricated by Xytel, and installed at Argonne; see the photograph on the next page. An integral heat exchanger (heat and mass transfer device), shown below, has also been designed and built. Plans are for the heat exchanger to be installed as an integral condenser, located above the feed point on the column, and to demonstrate the application with methanol-water separation. Industrial partners will be sought to participate in follow-on research and in demonstration of the device and associated energy savings.

Endothermic Fuel Heat Transfer

A supercritical fuel-reforming system for reducing emissions of industrial gas turbine engines is being developed under a United States Industrial Coalition (USIC) CRADA with United Technologies Research Center (UTRC), and under a contract with the Russian Central Institute for Aviation Motors (CIAM). Key to the development of such a system are the supercritical fuel injectors/combustors and the compact heat exchanger/catalytic reactor in which the liquid fuel is reformed to obtain gaseous products with improved combustion characteristics. Successful development of these key components requires applied research in the following areas: supercritical fuel injection and combustion, catalysts, supercritical heat transfer during endothermic chemical reactions, heat transfer enhancement using passive and/or active devices, mitigation of coking, and heat exchanger/reactor materials and fabrication techniques.

Argonne's work scope is focused on the heat exchanger/reactor. The project objectives are to perform experimental measurements of supercritical heat transfer and pressure drop in small-diameter tubes, both bare-wall and catalyst-coated; develop design

*Combined heat and mass
transfer device*

Distillation research column

Endothermic fuel heat transfer test apparatus

correlations for supercritical heat transfer and pressure drop, based on the generated experimental data base; collaborate with CIAM on the application of these correlations to design a prototype heat exchanger/reactor; collaborate with CIAM and UTRC on materials selection and fabrication of the prototype heat exchanger/reactor; and collaborate with UTRC on evaluation of heat exchanger/reactor performance in the UTRC injector/combustor tests.

The project was initiated in January 1997. A special-purpose Endothermic Fuels Heat Transfer Test Apparatus, shown schematically in the figure above, has been designed and fabricated. In this test apparatus, jet fuel at supercritical pressures (>600 psia) is heated to 800 to 1200°F by passing DC current through the test channel. Local heat transfer coefficients will be determined to include the effects of the catalytic chemical reactions taking place within the channel.

Nanofluid – An Advanced Heat Transfer Fluid

Nanofluids are a new class of advanced heat transfer fluids that are engineered by suspending metallic nanoparticles in conventional heat transfer fluids. Demonstration of feasibility is being performed under a Science and Technology, Laboratory-Directed Research and Development (LDRD) project, with the Materials Science Division as our partner. Efforts within the TMCP Section are focused on measuring and characterizing the heat transfer and flow properties of nanofluids as a function of particle material, size, and loading. A test apparatus based on the transient hot-wire technique was designed and fabricated for use in measuring the thermal conductivity of nanofluids. The thermal

conductivities of four oxide nanofluids were measured. In particular, water and ethylene-glycol-based nanofluids containing copper and aluminum oxide particles were tested. As shown in the figure below, thermal conductivity increases linearly with volume fraction, but with different slopes for each nanofluid.

The measured thermal conductivity was shown to be a function of the thermal conductivity of the base fluid as well as that of the nanocrystalline particles. Heat transfer tests to be conducted will allow us to back-calculate, for a particular nanofluid, the effective values for specific heat and viscosity.

It remains to investigate the effect of particle shape and size. Pure metallic particles versus oxides and the effect of flow should also be studied. This follow-on research, and the scale-up of production methods, will be performed under a CRADA project with Heatcraft and Nanophase Technologies as industrial partners.

Ice Slurries for District Cooling

Argonne, under U.S. DOE funding, pioneered the development and fostering of ice slurries for district cooling. Because of the high energy content of ice slurries, which is due to the phase-change (i.e., melting) of the ice particles at a cooling load, the energy content of a slurry pumped to a load can be up to five times greater than that associated with chilled water delivered at the same mass flow rate. This high energy content also allows slurry transmission piping and storage tank sizes to be reduced significantly over what would be required with conventional, chilled-water cooling systems. Today both domestic and foreign interests have considered adopting this technology for the cooling of large loads.

The storage of ice slush in tanks, however, is complicated because under certain not well understood conditions, the ice crystals will progressively grow together, or agglomerate, making pumping of the slurry from the tank very difficult. It is desirable to

*Enhanced thermal
conductivity of oxide
nanofluid system*

store the slurry at the highest packing density possible, in order to most effectively utilize the available tank size. Therefore, even if the ice does not agglomerate in the tank, it must be carefully diluted to a packing density that is compatible with pumping it through a pipe delivery network. No reliable means has been developed for tank-slurry extraction or ice-loading control. A research project, initiated in July 1997 in collaboration with NKK Corporation of Japan, is being conducted to reconfigure the Argonne Slurry Test Facility and perform experimental studies on the factors influencing ice crystal agglomeration in ice slurry storage tanks. The studies will also address developing methods for minimizing agglomeration and for improving the efficiency and controllability of tank extraction of slurry for distribution to cooling loads. These studies will address the remaining engineering issues that are impediments to the utilization of the ice slurry cooling concept.

Nuclear Steam Generator Tube Integrity

Argonne's Energy Technology Division is conducting, for the NRC, a program on nuclear steam generator tube integrity in an effort to quantify the importance of corrosion-flawed steam generator tubes to the safe operation of nuclear plants that operate with flawed tubes. The TMCP Section is contributing to the program, leading the efforts to design and fabricate two new large-scale facilities to furnish unique data supporting an improved understanding of the corrosion mechanisms causing flawed tubes, and the nature of the leakage flows and rupture conditions that can occur in steam generators. Both facilities are designed to operate under prototypical pressurized-water-reactor conditions.

Leak/Rupture Facility

The ANL tube-rupture and leak-rate blowdown test facility will be used to obtain data on burst pressures, failure modes, and leak rates of flawed tubing at temperatures up to 343°C (650°F), pressures of 21 MPa (3000 psi), and pressurized-water flow rates up to 760 L/min (200 gal/min). The important features of the facility are a large blowdown-vessel water inventory to ensure high, stable flow rates and permit full-range testing of initially stable leaking cracks to instability; piping and valves of a size appropriate to minimize pressure drop in the supply line to a flawed tube and thereby permit high flow rates; the use of a downstream back-pressure regulator valve to control tube secondary-side pressure, thereby minimizing nonprototypical two-phase flow from entering the tube; and a computer feedback valve control to allow programmed ramps of the pressure differential across the tube. The tubes to be tested in the facility will have either laboratory-grown cracks produced by chemical means or machined flaws. Both axial and circumferentially oriented flaws will be examined. Crack sizes will range from very small cracks, which are stable under main steam line break (MSLB) conditions, to larger cracks, which will first propagate through the wall and then eventually grow unstable as the pressure is increased to that corresponding to MSLB.

Model Boiler Facility For Corrosion Cracking Tubes

Steam generator tube secondary-side stress corrosion cracking is a major aging degradation mechanism in nuclear power plant steam generators. The Model Boiler Multi-Tube Corrosion Cracking Facility will be used to simulate near-prototypical steam generator thermal-hydraulic and chemistry conditions for tube/crevice geometry as a means of

creating corrosion-flawed tubes. The flawed tubes will be used in leak-rate/rupture characterization and NDE testing at ANL. In this apparatus, heat transfer across the tube wall (from primary/inside to secondary/outside) into a crevice surrounding the tube produces chemical concentration/hideout of certain chemicals residing at trace levels in the secondary water to detrimental levels in the crevice and causes stress corrosion cracking on the outside of the tube. The facility was designed to achieve prototype boiling conditions on the secondary side of a steam generator tube in the crevice area formed by the tube and its interface with a crevice simulator. Both tube and crevice simulators are of prototype materials and the crevice region is subject to prototype temperature, pressure, and heat flux. The secondary-side water chemistry can be typical plant chemistry, or enhanced corrosion-inducing chemistry to accelerate tube degradation and allow the exploration of parameter influences. Without crevice boiling, chemical concentration would not occur in the crevices. The apparatus offers excellent control over the thermal conditions that influence boiling and hence a good opportunity to study the influence of thermal conditions on the corrosion of steam generator tubes. The facility is designed for unattended operation and will run continuously for periods of 2 to 5 weeks, the estimated time range required for various degrees of stress-corrosion cracking to occur.

Multiport Cylinder Dryer

DOE's Office of Industrial Technologies, as part of its *Forest Products - Vision 2020 Initiative*, has approved funding for an TMCP Section proposal to develop and demonstrate a multiport cylinder dryer concept. In the pulp and paper industry, increased productivity with existing paper mills is limited by the dryer. The concept of a multiport cylinder dryer involves flowing steam through multiport passages that are close to the cylinder dryer surface. Relative to a conventional steam-drum dryer, the new design is expected to significantly increase the heat transfer surface area and also to minimize the "condensate rim," which is a major contributor to heat transfer resistance. As a result, drying rates can be increased in existing cylinders, or the diameters of new dryers can be made smaller. It is expected that the project will be performed with the Beloit Corporation, a major dryer manufacturer, as an industrial partner.

The first year of the project will focus on laboratory-scale heat transfer tests, with the objective of evaluating various design concepts, including the use of heat transfer enhancement devices, that will break up the condensate rim and increase the heat transfer coefficient. Working closely with Beloit engineers, we will develop in the first year an overall design concept that will allow retrofitting of existing systems. The end of the first year will represent a "go/no-go" decision point. If the first-year research shows the multiport dryer concept to be feasible from a performance, cost, and maintainability standpoint, in the second year of the project we will perform limited pilot-scale tests in the Pilot Test Dryer and Pilot Paper Machine at the Beloit Corporation's R&D Center.

Recent Work by the Thermal, Mechanical, and Chemical Processes Section

Publications

Flow-Induced Vibration of Tubes in Cross-flow

S. S. Chen, Y. Cai, and S. Zhu

Proc. 14th Intl. Conf. on Offshore Mechanics & Arctic Engr., Copenhagen, June 18-22, 1995; in J. of Offshore Mechanics and Arctic Engr., Vol. 118, pp. 253-258 (1996).

Small Circular- and Rectangular-Channel Boiling with Two Refrigerants

T. N. Tran, M. W. Wambsganss, and D. M. France (U. of Illinois at Chicago)

Intl. J. Multiphase Flow Vol. 22, No. 3, pp. 485-498, 1996.

Dynamic Characteristics of Magnetically Levitated Vehicle Systems

Y. Cai and S. S. Chen

Submitted to Applied Mechanics Review.

Dynamic Stability of Repulsive-Force Maglev Suspension Systems

Y. Cai, D. M. Rote, T. M. Mulcahy, Z. Wang, S. S. Chen, and S. Zhu

ANL-96/18.

Dynamic Response of a Stack/Cable System Subjected to Vortex-Induced Vibration

Y. Cai and S. S. Chen

Journal of Sound and Vibration (1996) 196(3), 337-349.

A Review of Dynamic Characteristics of Magnetically Levitated Vehicle Systems

Y. Cai and S. S. Chen

ANL-95/38 (1996).

Steam Generator Tube Rupture and Leak-Rate Blowdown Test Facility

K. Kasza

Presented to USNRC-EPRI Steam Generator Research Coordination and Cooperation Meeting, Argonne National Laboratory, April 24-25, 1996.

Steam Generator Tube Model Boiler Test Facility

K. Kasza

Presented to USNRC-EPRI Steam Generator Research Coordination and Cooperation Meeting, Argonne National Laboratory, April 24-25, 1996.

Bubble Research Helps Establish Compact Heat-Transfer Designs

V. Comello and K. E. Kasza

R&D Magazine, Vol. 38, No. 9, p. 50 (1996).

Application of Metallic Nanoparticle Suspensions in Advanced Cooling Systems

S. Lee and S. U. Choi

Proc. 1996 Intl. Mech. Engr. Conf. Session on Application of Metallic Matls. in Adv. Engr. Systems, Atlanta, GA, Nov. 17-23, 1996; in Recent Advances in Solids/Structures and Application of Metallic Materials, eds. Y. Kwon, D. Davis, and H. Chung, ASME, New York, PVP Vol. 342/MD-Vol. 72, pp. 227-234 (1996).

Nuclear Power Plant Generic Aging Lessons Learned (GALL): Vols. 1 & 2

K. E. Kasza, D. R. Diercks, J. W. Holland, S. U. Choi, J. L. Binder, W. J. Shack, O. K. Chopra, D. C. Ma, and A. Erdemir; J. L. Edson, L. C. Meyer, E. W. Roberts (INEL) NUREG/CR-6490, ANL-96/13 (Dec. 1996).

Microscale Flow Visualization of Nucleate Boiling in Small Channels: Mechanisms Influencing Heat Transfer

K. E. Kasza, T. Didascalou, and M. W. Wambsganss

Proc. Intl. Conf. on Compact Heat Exchangers for the Process Industries, Snowbird, UT, June 22-27, 1997, R. K. Shah, ed., Begell House, Inc., New York, 1997, pp. 353-364.

A Correlation for Nucleate Flow Boiling in Small Channels

T. N. Tran (Texas Tech U.), M. W. Wambsganss, M. C. Chyu (Texas Tech U.), and D. M. France (U. of Illinois, Chicago)

Proc. Intl. Conf. on Compact Heat Exchangers for the Process Industries, Snowbird, UT, June 22-27, 1997, R. K. Shah, ed., Begell House, Inc., NY, 1997, pp. 343-352.

Steam Generator Tube Integrity Program: Semiannual Report August 1995-March 1996

D. R. Diercks, S. Bakhtiari, O. K. Chopra, K. E. Kasza, D. S. Kupperman, S. Majumdar, J. Y. Park, and W. J. Shack
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Enhanced Thermal Conductivity through the Development of Nanofluids

J. A. Eastman, S. U. Choi, S. Li, L. J. Thompson, S. Lee

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D. R. Diercks, S. Bakhtiari, K. E. Kasza, D. S. Kupperman, S. Majumdar, J. Y. Park, and W. J. Shack
NUREG/CR-6511, ANL-97/3, Vol. 2.

Transportation of Hazardous Materials

Section Manager: Y. Y. Liu

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Secretary: J. L. Fisher

The Transportation of Hazardous Materials (THM) Section primarily provides direct technical support to DOE in reviewing Safety Analysis Reports for Packaging (SARPs) of radioactive material shipping packages for compliance with DOE Orders and with NRC and DOT regulations. In addition, the Section provides technical assistance to DOE on a variety of generic waste management and transportation issues, including the production and distribution of training and public information videos.

In response to decreasing DOE support for the review of SARPs, the THM Section has secured NRC program support. The Section provides technical support to NRC on issues related to aging that can affect the function of nuclear power plants. The review expertise is similar to that required for reviewing SARPs.

Review of Aging Issues for License Renewal

A substantial number of older nuclear power plants will reach the end of their initial 40-year licensing periods in the next 10-15 years, and the NRC is developing criteria for license renewal. An important consideration in developing these criteria is the proper accounting for component aging and time-dependent degradation processes. Accordingly, the NRC is sponsoring a program at ANL to provide technical support for generic aging issues that can affect the function of nuclear power plants during their period of extended operation.

Under one activity in this program, ANL is revising and updating the NRC's "Standard Review Plan for License Renewal." This activity will result in a Standard Review Plan that is based on the latest NRC Rule for license renewal contained in 10 CFR Part 54, as well as information from industry reports and lessons learned during the past years of license renewal activity. Over the years, ET Division engineers, primarily in the Mechanics of Materials Section, have worked with the NRC in this license renewal activity on a project that exhaustively catalogued the mechanisms that could lead to plant performance degradation and affect the technical justification for granting plant life extension licenses. In this effort, technical evaluations of the industry reports supporting license renewal have been carried out by ANL, comparing industry findings against the results of ANL research

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and of that at other national laboratories. The project included documenting and cataloging the industry and the NRC positions for all aging mechanisms for all components related to aging and degradation.

In a second activity under this NRC-sponsored program, proposed aging-management strategies for specific plant designs are being reviewed. Owner's groups for Westinghouse and Babcock & Wilcox pressurized water reactors (PWRs) have submitted a series of technical reports to the NRC addressing the aging of major reactor components and systems and the impact of this aging on license renewal. Similar reports from specific utilities are also being reviewed. Technical experts from ANL's Energy Technology and Reactor Engineering divisions provide comprehensive reviews of these reports in the areas of materials and mechanical/structural engineering to verify that the significant aging issues are properly identified and addressed. Using a protocol that is similar to that described below for the evaluation of radioactive material packaging safety analysis reports, THM section staff have carried out thorough reviews of the "Aging Management Review Reports" for approximately 20 specific nuclear power plant component systems, such as the reactor containment vessel, the containment building, and emergency diesel fuel oil system, for example, and have generated questions to be issued to the licensee organization that prepared the reports. These questions are then presented to the licensee by NRC, leading eventually to revisions of the reports and further review by ANL staff. This process is ongoing and expected to continue through the coming year.

SARP Review Group

This Group operates independently of other Section programs and comprises a staff that includes the disciplines necessary to review the entire range of information provided in a SARP. On the average, more than 10 SARPs are under consideration by ANL at any one time. The SARP is a voluminous document that describes the package technically and justifies why the package meets all applicable regulations.

Each SARP consists of nine chapters: General Information and Drawings, Structural, Thermal, Containment, Shielding, Criticality, Operating Procedures, Acceptance Tests and Maintenance Program, and Quality Assurance.

Selected Review Activities

The ANL SARP Review Group provides DOE with an independent review and evaluation of the information contained in a SARP that describes the packaging used to transport radioactive materials. The review is conducted to apprise DOE of the ability of the packaging to meet all DOE orders and federal regulations for safe transport. This process ensures that a consistent and independent review is performed, while maintaining the authority granted to DOE by the U.S. Department of Transportation for the packaging evaluation and certification process.

The function of the ANL review group is to perform an interdisciplinary, in-depth assessment of the proposed packaging based on the SARP submitted by the applicant to DOE. The SARP contains detailed information on the design, construction, and contents of the packaging. The assessment of the packaging includes detailed confirmatory analyses of

the structural, thermal, containment, radioactive shielding, and nuclear criticality aspects of the packaging design. In addition, construction drawings, fabrication procedures, operating procedures, maintenance and test programs, and quality assurance plans are evaluated.

The primary goal of the review is to establish that the packaging will perform as intended under both the normal conditions expected in transport and the hypothetical accident conditions. These transport conditions are detailed in the regulations contained in Title 10 of the Code of Federal Regulations, Part 71 (10 CFR 71). The regulations contain specific environmental and mechanical loading conditions that the package must be able to accommodate during transport.

Because of the hazardous nature of the package contents, the regulations are necessarily severe. Consequently each element of the review is essential and, to be successful, requires a team effort from the SARP review group. For example, the General Information and Drawings Section is reviewed to ensure that there is sufficient information for each reviewer to understand the type, quantity, and form of the radionuclides or fissile materials; the proposed use of the packaging; the operational and safety features; and the materials used in construction. The construction drawings and engineering sketches should provide sufficient details to evaluate the methods of fabrication and allow development of appropriate analytical models.

Materials and Design Criteria (D. T. Raske)

Radioactive material packaging must be designed and constructed in compliance with the requirements of DOE Order 460.1 (Packaging and Transportation Safety), Title 10 of the Code of Federal Regulations (Packaging and Transportation of Radioactive Material: 10 CFR 71), and Code of Federal Regulations 49 CFR 173 (Shippers General Requirements for Shipments and Packagings). These regulations specify the performance during transport, but actual design criteria are not addressed by these regulations.

The provisions of 10 CFR 71 state that a package "... must be evaluated by subjecting a sample package or scale model to test, or by other method of demonstration acceptable" This statement is generally interpreted as meaning that a package can be qualified by either physical testing or analysis. Qualification by analysis requires that the package be designed to criteria suitable for the environment and structural loadings unique to high-level, Category I, radioactive materials transport packagings. In the structural area, there are no design criteria specific to radioactive materials packaging. However, both DOE and NRC identify parts of the ASME Boiler and Pressure Vessel Code as acceptable design criteria.

The regulations allow a packaging to be qualified by subjecting a sample package to specified tests. Qualification by testing may not necessarily involve a detailed stress analysis of the containment vessel's structural components. Nevertheless, as with qualification to the ASME Code, the construction materials must be rigorously pedigreed. Qualification by testing must be supported by a comprehensive quality assurance plan to provide materials traceability, thorough documentation of construction methods, and documented evaluation of test results.

The regulations also state that a scale model may be subjected to the qualification tests to demonstrate that a packaging satisfies the requirements. One problem area associated with the use of scale models is in modeling a containment vessel seal assembly. Although the literature contains many studies on the design of bolted gasketed joints, there are at present no known scaling laws relating leak rates in scale models to those in full-size joints. Consequently, a bolted gasketed joint must be qualified by analysis and/or physical acceptance testing of a prototype.

In summary, qualification by analysis, physical testing, or scale models requires that the materials and methods of construction be sufficiently documented to prove that all packages are identical, and that future replacement or repairs do not alter the original packaging. A quality assurance plan covering the structural materials, methods of construction, and physical testing is essential for all packages. The use of scale models for physical testing is complicated by uncertainties in scaling laws; the lack of documented scaling laws for seal assemblies with bolted gasketed joints, and the time-dependent mechanical behavior of materials, make the use of scale models difficult for packaging qualification. All structural materials should be Code-approved or otherwise qualified by authoritative specifications. Certification, fabrication, and examination of the structural materials should be equivalent to the requirements of the applicable ASME Code.

Structural (P. Turula)

The structural review of SARPs is a process that interacts in a complex manner with various other analyses because there are very few regulatory provisions that directly specify structural performance requirements. However, evaluation of the compliance of the packaging with almost any of the requirements specified in the regulations is dependent on a characterization of the structural performance of the packaging. The regulations specify a series of normal operating condition tests and hypothetical accident condition tests, many of which are structural in nature, that indicate a structural evaluation corresponding to a physical test and then state requirements that are related not directly to structural performance but to other metrics such as leakage, radiation, or subcriticality.

The regulatory tests that are specified in structural terms are the various drop and impact tests such as the "normal conditions" drop test, which may range from 1 to 4 ft, the stacking test, the 30-ft hypothetical accident drop test, the puncture test and the impact test. Others, such as the various specified thermal tests, result in thermally induced structural loadings in addition to the direct material thermal performance effects that they impose. With a few exceptions, the required performance measures under these imposed tests are not structural in nature. That is, the regulations do not specify maximum deformations or maximum stresses or minimum safety margins, but rather a minimum level of containment integrity or a level of external radiation in terms of a maximum activity release, or they limit "the loss of effectiveness" or require that a margin to subcriticality be maintained.

Thus, in general, structural evaluation and analysis plays a supporting role in the overall evaluation of a packaging. The structural analyst must understand the loadings being applied, as well as how the performance under these loadings affects other regulated parameters. Simply determining what structural deformations and stresses the loadings

produce is not sufficient. The exceptions to this generalization relate to specific factor of safety measures specified in the regulations for lifting and tie-down devices, and some specific requirements on overall deformations that affect spacing of components and the size of openings, related to packagings that are designed to carry fissile materials.

The structural designs of radioactive materials shipping packages change substantially from one packaging design to another. The general shape will vary from package to package even when some design features may be repeated. However, it is the design details that control the structural behavior, and therefore it is impossible to generalize the appropriate analysis procedures and produce a standardized code for packaging structural analysis. Several attempts to do this have been made by major government agencies involved in this field, without any real success. Such general purpose packaging evaluation programs fail to perform an acceptable evaluation because each packaging will have some key details that the standardized code is incapable of recognizing and dealing with properly. Therefore the structural evaluation performed by the Argonne SARP Review Group is unique for each packaging and generally takes one of two (and sometimes both) directions. One approach is to develop a simplified model that treats the behavior of the packaging structures on a macro scale, as a single-mass system or a multiple-mass system. The other approach treats the structure in detail with finite-element modeling. Frequently, both approaches are applied selectively to different parts, or to different loading evaluations, of the same packaging.

A typical example of a structural review task is the simulation of a 30-ft drop of a package that has some impact-limiting hardware, such as a foam cylinder or several layers of wood on its end. Although sophisticated computer programs are available for this type of dynamic analysis, they are not suitable for our purposes. These general-purpose dynamic analysis programs require extensive and time-consuming effort to set up the details of the geometry and the properties of the various materials involved. The material models must characterize the nonlinear large strain behavior of the material reasonably well or the results will be meaningless. The running time of these program is still a problem, even with the computing power currently available, because the same solution algorithm is typically employed for all parts of the model, regardless of the need to treat any particular part with the minimum time-step needed in some key aspect of the analysis.

Specialized packaging programs have been found lacking because typically they treat the impact limiting feature as a single element and analyze the relatively rigid part of the package with a detailed elastic model, considering its dynamics and recovering its stresses, but ignoring the subtleties of the highly inelastic large deformation behavior of the limiter. Such essential material behavior as the unloading during strain reversal, is not considered. Moreover, these programs require the detailed specification of the behavior of the impact limiter as a single node component, rather than accepting as input the behavior of the constitutive limiter material and impact limiter geometry. Determining the global impact limiter behavior becomes a major part of the problem to be solved, and the specialized programs are of no help in solving this problem.

The package drop simulation analyses performed by the ANL SARP Review Group have consisted of three types: First, single body dynamic, energy balance type calculations, in which the container is treated as a simple body loaded by the impact limiter and the impact

limiter is treated as a massless inelastic member; Second, one-dimensional models with detailed simulation of the limiter geometry and its nonlinear material properties, and third, selected efforts with large finite-element codes.

Much conventional linear elastic analysis is also used. Typical examples include consideration of tie-down and support hardware under the unusual loadings postulated by 10 CFR 71.45. Both "hand calculation" solutions and conventional finite-element model solutions have been developed. Another example is the solution of linear elastic models for stresses in cask vessels under pressure and thermal loadings. Handbook analyses and simple linear finite-element solutions have been used here with particular attention to the interaction between a vessel and its lid.

Thermal (V. L. Shah)

The thermal review evaluates the thermal operating characteristics and performance of the package under normal transport and hypothetical accident conditions, taking into account the extreme environmental conditions, minimum and maximum decay heat loads, and damage caused by the drop and puncture tests as specified in 10 CFR 71. The review also includes confirmatory analyses; comparison of the analytical results with allowable limits of temperature, pressure, and thermal stresses for the package components; and determination of the effect of thermal performance on the structural, containment, and shielding areas of the package.

To determine that a package complies with the CFR requirements under all transport conditions, the confirmatory analyses are focused on modeling and evaluating package performance under worst-case conditions. An example of a review performed recently of the Remote-Handled Transuranic package, Model 72-B, (RH-TRU-72B), is described below. The HEATING6 module of the SCALE Computer Code was used for the analysis. The SARP Review Group selected HEATING6 because this module is designed specifically for thermal analysis of nuclear shipping casks. Furthermore, this module has been thoroughly tested on a set of casklike problems, has been compared with other thermal analysis codes such as TRUMP, TEMPEST, Q/TRAN, CINDA-3G, etc., and is well documented.

The RH-TRU-72B package, shown in the next figure, is designed to transport remote-handled waste material contaminated with transuranic radionuclides. The packaging comprises a waste container that confines the waste, an inner vessel that provides an inner containment boundary, an outer cask with a thermal shield that provides an outer containment boundary and acts as an environmental barrier, and energy-absorbing impact limiters at each end of the outer cask. The inner vessel, fabricated primarily of type 304 stainless steel, consists of a 1.5-in.-thick bottom plate; a 0.375-in.-thick, 32-in.-outer-diameter shell; and a 6.5-in.-thick lid. The outer cask, fabricated primarily of type 304 stainless steel, consists of a 5-in.-thick bottom plate; a 1-in.-thick and 32.375-in.-inner-diameter inner shell; a 1.5-in.-thick and 41.125-in.-outer-diameter outer shell; and a 6-in.-thick lid. The annular gap between the outer and inner shell is filled with lead. The thermal shield of 10-gauge stainless steel is spaced from the outer shell of the outer cask by a 12-gauge wire wrap on a 3-in. pitch spacing. The impact limiters (76-in. in outside diameter and 46 in. long) are filled with polyurethane foam and have skins fabricated from 300 series stainless steel.

Geometry of RH-TRU-72B Package

The containment and shielding review staff must know if the thermal shield and impact limiters will provide sufficient protection so that during the hypothetical accident thermal event, the containment vessel O-ring temperatures will stay below the allowable limits and the lead shield in the outer cask will not melt. Confirmatory analysis of the hypothetical accident thermal event used a three-dimensional axisymmetric model of the package. The numerical model of the package is shown on the next page. To evaluate the performance under the worst-case condition, the package was assumed to be damaged from the hypothetical accident drop and puncture tests. Damage from the 30-ft vertical and side drop tests was modeled by assuming that the top and bottom ends of the impact limiters were crushed by 13 in. and the outer radii of the limiters were reduced by 14 in. The damage from a 40-in. side drop on a 6-in. puncture bar may cause the thermal shield to come into contact with the outer surface of the outer cask. To account for this possible

thermal contact and to make the prediction conservative, a solid thermal contact, (6 in. wide and 6 in. long) between the thermal shield and the outer surface of the outer cask, was assumed in the model.

The maximum temperatures of the O-ring and lead shield predicted in the analysis, together with the allowable limits, are presented in the table on the following page. The analysis shows that the RH-TRU-72B packaging can provide adequate thermal protection during the hypothetical accident thermal event to maintain the containment boundary and lead shielding below the maximum allowable temperature limit.

Maximum temperatures (°F) for RH-TRU-7B package

Component	Analysis	Allowable
Outer Cask O-ring	219	400
Inner Vessel O-ring	203	400
Lead shield	590	620

Containment (J. J. Oras, Jr.)

The containment review evaluates the (a) integrity of the containment boundary, (b) radioactive-leak-rate requirements for normal and hypothetical accident conditions, and (c) special double-containment requirements for plutonium shipments. The containment review is intimately connected with the structural and thermal analyses for impact on the environment and integrity of the containment boundary. On occasion, the reviewer finds that the Code of Federal Regulations (CFR) or the American National Standard for Radioactive Materials - Leakage Tests on Packages for Shipment (ANSI N14.5-1987) are unclear in an area of concern. The reviewer then proposes a position or interpretation to the certifying office in Washington so that the containment review can proceed. The following discussion covers ANL concerns for leakage testing of packagings with three-O-ring closure seals that have been submitted to the certifying office in Washington.

Most Type B packagings used to transport radioactive contents employ a set of two nested O-rings in the closure seal to facilitate the leakage rate testing of the containment boundaries as recommended in ANSI N14.5 to demonstrate compliance with Title 10, Part 71 of the Code of Federal Regulations. A small annular volume is created between the outer O-ring and the containment O-ring being tested. Recent large packagings, such as the TRUPACT II Shipping Package and the RH-TRU 72-B Waste Shipping Package incorporate three O-rings in their closure seals for the inner containment boundaries. The additional inner O-ring is used either to protect the containment O-ring from grit from the contents (which could prevent the O-ring from sealing properly during transport) or to limit the amount of helium required to conduct a leakage rate test by defining a volume much smaller than the contents cavity. The concerns caused by the addition of the inner O-ring to the more common set of two nested O-rings will be discussed together with possible means of addressing these concerns in a Safety Analysis Report for Packaging (SARP).

Schematic diagrams of a typical closure and the upper seal test port arrangement, the gas sampling port arrangement, and the backfill port arrangement (next three figures) are useful in understanding leakage testing concerns. The gas sampling port is used when loading a shipment for ease of lid installation and as a sampling port on receiving a shipment. The backfill port is used to backfill with helium the region between the containment O-ring and the inner O-ring. The seal test port is used to pull a vacuum on the annular volume between the containment O-ring and the outer O-ring of the closure seal.

The evacuated gas is sampled by a mass spectrometer to determine the helium leakage rate. Failure of either the inner O-ring of the closure seal or the inner O-ring of the gas sampling port would release the helium backfill into the package cavity (where the contents are located) diluting the helium with air. The gas sampling port insert and the backfill port insert are designed for leakage rate testing of the seal of the containment boundary, specifically the outer O-ring of the gas sampling port and the O-ring of the backfill port, once the port closure bolts are tightened. The outer O-ring of the gas sampling port is the containment seal, while the inner O ring provides a barrier between the contents cavity and the containment O-ring of the lid closure.

Upper seal test port arrangement

Gas sampling port arrangement

Backfill port arrangement

The concern is that the procedures in the SARP do not preclude false positive results of the leakage rate tests of the containment O-ring, i.e., that the measured leakage rate appears to pass the acceptance criteria because either no helium is detected or because the helium detected has mixed with an unknown quantity of air, resulting in a much lower indicated leakage rate than is actually present. The leakage test procedures require that the region between the containment O ring and the inner O-ring of the lid closure be evacuated to either 1 or 52 mm of Hg for the preshipment or acceptance leakage tests, respectively. Then, the procedures require the backfill of helium to 1 atm, closure of the backfill port and evacuation of the region outside the containment O-ring of the lid closure seal by a roughing pump for the helium leak detector prior to actual leakage testing. False positive results can be caused in two ways:

- *A large leakage in the containment O-ring.* If there is a large leak in the containment seal, evacuation by the roughing pump could pull the limited amount of helium in the annular volume between the containment and inner O rings, past the containment O-ring before the vacuum required by the helium leak detector is established and the leak detector is functioning. This concern could be eliminated if the source of helium was the entire contents cavity or if the leak detector was operating before the helium backfill. Filling the entire contents cavity may be impractical because of the time and resulting cost associated with pulling a vacuum on the large cavity volume before backfilling with helium.
- *Leakage in either the inner O-ring of the closure seal or in the inner O-ring of the gas sampling port.* If there is a leak in either the inner O-ring of the closure seal or the inner O-ring of the gas sampling port, the limited amount of helium could mix with the air in the package cavity (where the contents are located). Thus, when the leak detector is pulling a vacuum outside the containment seal, it would see a mixture of air diluted with a small amount of helium instead of pure helium. The leak detector is sensitive to helium and thus would indicate a leak that is smaller than the actual air/helium mixture leakage. This concern could also be eliminated if the source of helium was unlimited, e.g., if the whole cavity inside containment is filled with helium. Once again, filling the entire contents cavity may be impractical. This concern could also be eliminated if the leakage rate past the inner O-ring was measured and determined to be negligible before the containment O-ring is tested. This could be accomplished by backfilling helium into the annular region between the containment O-ring and the inner O-ring and pulling a vacuum on the contents cavity via the gas sampling port. However, the large contents cavity would have to be evacuated, and this may also be time-consuming and therefore costly. The applicant for a packaging certification should justify in the SARP that the inner O-ring will maintain an essentially pure volume of helium tracer in the annular region between the containment O-ring and the inner O-ring/contents cavity during leakage rate testing of the containment boundary O-ring.

Frequently, SARP writers indicate that if qualified leak testers, e.g., Level II or Level III, perform the leakage rate tests, the tests will somehow always be done correctly. We have examined Section V of the ASME Boiler and Pressure Vessel Code, ANSI/ASNT CP-189, the Nondestructive Testing Handbook and the Annual Book of ASTM Standards for the specific training required of Level II or Level III qualified leak testers, but could not find training that would address the specific concerns presented in this paper. An argument is also frequently presented that evacuation of the annular seal region to 52 mm Hg demonstrates that the seals are functioning properly. However, all this actually shows is that the vacuum pump can evacuate the annular region at a rate higher than the leakage rate through the O-rings forming the annular region. Thus, the length of time required for conducting the leakage rate tests may be important for the particular packaging design. The SARP should provide justification that the requirements for leakage rate testing procedures, including the length of time needed to conduct the tests, will ensure that the containment closure seal is properly tested.

Shielding (Y. Y. Liu)

The objective of the shielding evaluation of a SARP is to verify that the packaging design satisfies all radiation shielding requirements and that the packaging will perform as intended under normal conditions of transport and hypothetical accident conditions defined in 10 CFR 71. For nonexclusive-use shipment under normal conditions of transport, the radiation level must not exceed 2 mSv/h at any point on the external surface of the package (≤ 10 mSv/h if the shipment is made in a closed transport vehicle; also, the package must be secured within the vehicle and no loading or unloading is allowed during transportation). If the package is shipped in an open vehicle, the 2 mSv/h radiation level applies to any point on the vertical planes projected from the outer edges of the vehicle. In addition, the radiation level must not exceed 0.1 mSv/h at any point 2 m from the above surfaces, and also must not exceed 0.02 mSv/h in any normally occupied position in the vehicle. Under hypothetical accident conditions, the radiation level must not exceed 10 mSv/h at 1 m from the package surface.

The shielding evaluation typically involves analysis to confirm that the package shielding design described in the SARP meets the regulatory requirements. Depending on the complexity of the design, the tools used in the analysis can vary from a point-kernel code (e.g., MICROSIELD) running on a personal computer, to three-dimensional Monte Carlo codes (e.g., MORSE-SGC/S and MCNP) running on the Section's dedicated IBM and SUN workstations. One of the shielding designs examined in detail last year was the Beneficial Uses Shipping System (BUSS), a packaging developed for shipping cesium chloride (CsCl) or strontium fluoride (SrF_2) capsules to facilities such as sewage, food, and medical-product irradiators. The primary purpose of the BUSS cask is to provide shielding and confinement, as well as impact, puncture, and thermal protection for its certified contents under both normal transport and hypothetical accident conditions. The figure on the next page is a schematic diagram of the BUSS cask; the following figure is a cutaway view of the three-dimensional MCNP4A geometrical model of the BUSS cask. This model was constructed to evaluate the measured photon streaming through the upper drain port, which had caused concern when it was first reported during the SARP review.

Diagram of BUSS cask

The MCNP4A geometry model of the BUSS cask consists of a total of 236 cells and 118 surfaces. Smaller cells were used near the upper drain port to enhance the efficiency of Monte Carlo calculations by allowing photon importance to be specified in the general direction of leakage. The drain plug, bore plug, and thermal cover in the upper drain port were modeled as separate cylindrical cells, with each cell material specified as either 304SS or void (when the component is absent) in the simulation of the particular configuration in the radiation survey. The radiation detector was modeled as a volume detector (Cell 51) with a diameter (8.26 cm) and volume (400 cm³) corresponding to the diameter and active volume of the actual ion chamber detector. In the majority of MCNP4A calculations, Cell 51 is used in conjunction with a track length estimator for flux (i.e., the F4 tally in MCNP), which keeps score by counting the average number of photon particle tracks traversing the volume of the detector. (The F2 tally in MCNP, which is a surface current estimator that counts the number of particles crossing a surface, is used in only two cases to verify that MCNP4A correctly calculated the dose rates measured on the general body of the cask.) The bulk of the MCNP4A calculations, therefore, consisted essentially of simulating each of the five radiation survey configurations for a given detector location and orientation. Altogether, 15 MCNP4A simulations were performed in a total CPU time of ~750 h. Because of the intense nature of the computation, some of the final production runs were performed on the IBM SP system in ANL's High Performance Computing Research Facility.

As noted earlier, photon importance has been specified in the small cells to aid particle transport in the general direction of leakage. Additional source-particle biasing techniques, such as source axial biasing, source radial biasing, source directional biasing,

MCNP4A geometry model of BUSS cask showing volume detector (Cell 51) and components in upper drain port: thermal cover (Cell 143), drain plug (Cell 144), and bore plug (Cell 145).

and source energy biasing, were also used to improve the efficiency of MCNP4A simulations. However, even with all of these variance-reduction techniques, the simulations still required a large number of particle histories and relatively long computing time (≈ 50 h in some cases) to generate reasonable statistics with relative errors that are comparable to the nominal $\pm 15\%$ experimental uncertainty. The simulations have yielded results that are in reasonably good agreement with the radiation survey data. The simulations have also provided insight into modeling radiation detection, notably on location and orientation of the radiation detector with respect to the photon streaming paths, and on techniques used to reduce variance in the Monte Carlo calculations. Additional details of the MCNP4A simulation of the gamma photon streaming in the BUSS cask can be found in the paper "Monte Carlo Simulation of Radiation Streaming from a Radioactive Material Shipping Cask," by Y. Y. Liu, R. A. Schwarz, and J. S. Tang (Proceedings of 1996 Topical Meeting on Radiation Protection and Shielding, North Falmouth, MA, April 21-25, 1996).

Criticality (Y. Y. Liu)

The objective of the criticality evaluation of a SARP is to verify that the packaging design satisfies all of the criticality requirements and that the package will perform as intended under normal conditions of transport and hypothetical accident conditions defined in 10 CFR 71. Under normal conditions of transport, a package used for shipment of fissile material must be so designed and constructed and its contents so limited that it would be

subcritical if water were to leak into the containment system or its liquid contents were to leak out. In addition, the maximum reactivity of the fissile materials is to be attained with the most reactive credible configuration, with moderation by water to the most reactive credible extent, and with close reflection by water on all sides. Under hypothetical accident conditions, the package must remain subcritical with the fissile material in the most reactive credible configuration consistent with the damaged condition, with water moderation to the most reactive credible extent, and with reflection by water on all sides as close as is consistent with the damaged condition.

The new edition of 10 CFR 71 that became effective on April 1, 1996, eliminated the three fissile class designations and established a single set of criteria for all packages of fissile materials. To enable control of fissile packages, a number "N" shall be derived on the basis of satisfying all of the following conditions and assuming packages are stacked together in any arrangement and with full reflection on all sides of the stack by water: (a) Five times "N" undamaged packages with nothing between the packages would be subcritical; (b) two times "N" damaged packages, if each package were subjected to the tests specified in 10 CFR 71.73 (Hypothetical accident conditions) would be subcritical with optimal interspersed hydrogenous moderation; and (c) the value of "N" cannot be less than 0.5.

One of the criticality evaluations performed last year was for the ATR fresh fuel container, which is being used for shipping unirradiated ATR fuel elements containing uranium enriched to a maximum of 94 wt.% ^{235}U . The previous Certificate of Compliance allows a maximum of 12 ATR shipping packages, each containing 4 unirradiated ATR fuel elements, to be shipped together as Fissile Class II. The elimination of fissile classification in the new edition of 10 CFR 71 did not change the above evaluation criteria, and "N = 12" was still the limiting case derived for an array (2 x 1 x 12) of damaged ATR packages for which the effective neutron multiplication factor (k_{eff}), calculated by KENO V.a, was ≤ 0.95 ($k_{\text{eff}} \leq 0.95$ is a conservative criterion widely used for ensuring criticality safety). The modeling of the damaged ATR package was conservatively based on the results of an actual ATR package following the drop and fire tests. Because the wood in the outer container burned in the fire test, the inner container was assumed to be able to move anywhere on the bottom steel surface of the outer container. Cadmium and polyethylene sheets in the inner container were excluded from the model, because both materials melted in the fire test. Even though the trapezoidal wooden spacers between the fuel elements were only charred in the inner container, a much smaller spacing, approximately the width of the coolant channels (2 mm) in the fuel plates, was assumed for the fuel elements, thus bringing them much closer than the spacing (27.4 mm) observed in the fire-damaged ATR shipping container.

In addition to the 2 x 1 x 12 configuration of the damaged ATR packages, other configurations, such as 3 x 1 x 8, 4 x 1 x 6, or 2 x 2 x 6, etc., have also been studied and shown to have lower calculated values of k_{eff} than that of the 2 x 1 x 12 configuration. Thus, based on the criticality calculations performed under a set of very conservative assumptions, up to 12 ATR packages may be shipped in a single, nonexclusive-use shipment, and up to 24 ATR packages may be shipped in a single, exclusive-use shipment. An array of such packages in either shipment would satisfy the 10 CFR 71 requirements on criticality safety. This has been the technical basis for the criticality control for the ATR package, which is currently at the final stage of the SARP review.

Quality Assurance (R. R. Fabian)

The objective of the quality assurance (QA) review is verification that all applicable QA requirements for Type B and fissile material packaging are included in the SARP. The QA chapter includes provisions for control of design, procurement, fabrication, handling, cleaning, assembly, inspection, testing, operation, maintenance, repair, and modification activities. Quality Assurance requirements are specified in DOE Order 460.1, 10 CFR 71, and 49 CFR 173. Guidance for implementation of DOE Order 460.1 is included in the Implementation Guide, G-460.1, Rev. 0, while guidance for 10 CFR 71 is contained in NRC Regulatory Guide 7.10 (Establishing Quality Assurance Programs for Packaging Used in the Transport of Radioactive Material).

In order to provide guidance to DOE and DOE contractors that design and use radioactive material packagings, the ANL SARP Review Group, along with Lawrence Livermore National Laboratory, authored the QA Chapter of the draft *Packaging Handbook*. The document has the primary goal of providing sufficient information and guidance to designers of packagings and authors of SARPs. The handbook provides guidance on the design, testing, certification, and operation of packaging for transportation of irradiated fuel and spent fuel.

In addition to reviewing SARPs, the ANL SARP Review Group presents a three-day "QA Training Course for Radioactive Material Packaging" at DOE field office sites to train DOE contractors in techniques to develop a QA Plan at the onset of a project. Through development of an effective QA Plan early in the life of a project, potential problems can be avoided and the result will be a quality packaging that is designed and fabricated within cost and schedule projections. The QA training course provides valuable information for developing, establishing, and implementing an effective QA Plan and a QA Chapter for a specific packaging. Since 1987, ANL has conducted 26 QA training courses for more than 600 DOE and DOE-contractor personnel.

Recent Work by the Transportation of Hazardous Materials Section

Publications

DOE QA Training Course for Radioactive Material Packaging

R. R. Fabian, J. J. Oras, and G. F. Popper.

Overview of the DOE Packaging Certification Process

Y. Y. Liu, R. D. Carlson, R. W. Carlson (Lawrence Livermore National Laboratory), and A. Kapoor (U.S. Dept. of Energy)

Proc. 11th Intl. Conf. on Packaging and Transportation of Radioactive Materials (PATRAM '95), Las Vegas, Dec. 3-8, 1995, Vol. I, pp. 473-479.

Progress Toward NuPack, the ASME Code for Type B

P. Turula

Proc. 11th Intl. Conf. on Packaging and Transportation of Radioactive Materials (PATRAM '95), Las Vegas, Dec. 3-8, 1995, Vol. IV, pp. 1631-1637.

Drop Test Energy Balance by Computer-Graphics Analysis of High-Speed Videotape

T. R. Bump and R. D. Carlson

Proc. PATRAM '95 Conf., Las Vegas, Dec. 3-8, 1995, Vol. I, pp. 178-184.

A Simplified Analysis of the Regulatory Puncture Test

T. R. Bump and Y. Y. Liu

Proc. PATRAM '95 Conf., Las Vegas, Dec. 3-8, 1995, Vol. I, pp. 407-414.

Chemical Reactions of UF₆ with Water on Ingress to Damaged Model 48X 10 Ton Cylinder

A. B. Rothman

Proc. PATRAM '95 Conf., Las Vegas, Dec. 3-8, 1995, Vol. IV, pp. 1571-1578.

A Guide for the ASME Code for Austenitic Stainless Steel Containment Vessels for High-Level Radioactive Materials

D. T. Raske

Proc. PATRAM '95 Conf., Las Vegas, Dec. 3-8, 1995, Vol. IV, pp. 1675-1682.

Analysis of Radiation Measurement Data of the BUSS Cask

Y. Y. Liu and J. S. Tang

Proc. PATRAM '95 Conf., Las Vegas, Dec. 3-8, 1995, Vol. IV, pp. 1831-1838.

Monte Carlo Simulation of Radiation Streaming from a Radioactive-Material Shipping Cask

Y. Y. Liu, R. A. Schwarz (Westinghouse Hanford Co.), and J. S. Tang (Oak Ridge National Laboratory)

Proc. Amer. Nucl. Soc. Mtg. on Radiation Protection and Shielding, Falmouth, MA, April 21-25, 1996.

Estimation of Package Temperatures During Hypothetical Accident Thermal Test Conditions

V. L. Shah

Proc. PATRAM '95 Conf., Las Vegas, Dec. 3-8, 1995, Vol. II, pp. 633-640.

Design and Analysis of Lid Closure Bolts for Packages Used to Transport Radioactive Materials

D. T. Raske and A. Stojimirovic

1995 ASME/JSME Pressure Vessel and Piping Conf., Honolulu, July 23-27, 1995; in Transport and Storage of Radioactive Materials 1995, PVP-Vol. 307, p. 77.

How to Avoid Errors in the Design and Fabrication of Transportation Packages

D. T. Raske

Proc. 1996 ASME Pressure Vessel and Piping Conference, Montreal, July 1996; in Transport and Storage of Radioactive Matls, PVP-Vol. 334, pp. 67-69 (1996).

Criticality Review of DOE Transportation Packages

M. K. Sheaffer (Lawrence Livermore National Laboratory), Y. Y. Liu, R. W. Carlson (LLNL), L. E. Fischer (LLNL), and A. K. Kapoor (LLNL)

Trans. American Nuclear Society 1996 Annual Meeting, Reno, NV, June 16-20, 1996; Report No. UCRL-JC-122818-ABS, Jan. 1996, pp. 210-211.

Quality Assurance

R. R. Fabian, T. R. Bump, G. F. Popper, and E. W. Russell (LLNL)

Chapter 11 in "DOE Packaging Handbook" (DRAFT), to be published by Oak Ridge National Laboratory; ORNL/M-5003, pp. 11-1 to 11-37.

Monte Carlo Simulation of Radiation Streaming from a Radioactive Material Shipping Cask

Y. Y. Liu, R. A. Schwarz (Westinghouse Hanford Co.), and J. S. Tang (Oak Ridge National Laboratory)

Proc. Amer. Nucl. Soc. Topical Mtg. on Radiation Protection and Shielding, Falmouth, MA, April 21-25, 1996; in Advances and Applications in Radiation Protection and Shielding, Vol. 2, pp. 1053-1060 (1997).

Technical Review Report for the ATR Fresh Fuel Shipping Container, Docket No. 91-29-9099, Jan. 21, 1997

R. D. Carlson, R. R. Fabian, Y. Y. Liu, J. J. Oras, D. T. Raske, V. L. Shah, and P. Turula
Prepared for U.S. Dept. of Energy by ANL SARP Group.

Technical Review Report for the RH-TRU 72-B Waste Shipping Package, Docket No. 95-19-9518, Dec. 19, 1996.

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Prepared for U.S. Dept. of Energy by ANL SARP Group.

Experimental Qualification of Containers to Transport Radioactive Materials

D. Raske

Presentation to Society for Experimental Mechanics Mtg., Dept. of Theoretical & Applied Mechanics, Urbana, IL, March 10, 1997 (video of testing methods included).

QO Questions for the Steel Banded Wooden Shipping Container

R. D. Carlson, R. R. Fabian, Y. Y. Liu, J. J. Oras, D. T. Raske, V. L. Shah, and P. Turula
Docket No. 96-39-5467, ANL/DOE Certification Review, Argonne National Laboratory, PC-2604, Dec. 1996.

Structural Design of Containment Vessels for Highly Radioactive Materials under the Proposed ASME NuPack Rules

P. Turula

Presented at ASME Pressure Vessel and Piping Conf. (PVPC '96), Montreal, July 1996.

Leakage Test Concerns for Packagings with Three-O-Ring Closure Seals

J. J. Oras, R. H. Towell (Eagle Research Grp.), and M. E. Wangler (USDOE)
Presented at 1997 ASME Pressure Vessels and Piping Conf., Orlando, July 27-31, 1997; in Transportation, Storage, and Disposal of Radioactive Materials, PVP-Vol. 348, pp. 97-99 (1997).

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Method of Estimating the Leakage of Multiple Barriers in a Radioactive Materials Shipping Package

R. H. Towell (Eagle Res. Grp.), A. Kapoor (USDOE), S. B. Moses (ORNL), and J. J. Oras
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An Investigation of the Relationship between Graphitization and Frictional Behavior of DLC Coatings

Y. Liu (Louisiana State U.), A. Erdemir, and E. I. Meletis
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ANL Report for the USNRC, Argonne National Laboratory, PC-2561, Aug. 1996.

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ANL Report for the USNRC, Argonne National Laboratory, PC-2565, Aug. 1996.

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D. T. Raske, Y. Y. Liu, and D. C. Ma

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Profits Are Taken Out of The Box Marked Safety

D. T. Raske

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Estimation of Maximum Temperature in a Package Subjected to Hypothetical Accident
Thermal-Test Conditions

V. L. Shah

ANL-96/12.

Method of Estimating the Leakage of Multiple Barriers in a Radioactive Materials Shipping
Package

R. H. Towell (Eagle Research Group, Inc.), A. Kapoor (USDOE), S. B. Moses (ORNL), and
J. J. Oras

Presented to ANL/DOE/LLNL Reviewers' Mtg., Livermore, CA, June 18-20, 1997.

Foaming and Foam in Industrial Applications

Y. Y. Liu

INVITED lecture, Foam Euroconference, Arcachon, France, May 28-June 2, 1996.

Materials Technology

Ceramics

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The Ceramics Section investigates the synthesis, fabrication, and characterization of ceramic materials to optimize their properties for practical engineering applications. In most cases, the applications are intended to facilitate generation, storage, or distribution of energy. For example, the goal of the superconductor program is to develop ceramic superconductors for more efficient generation, storage, and distribution of electrical energy. Ceramic membranes are being developed to convert methane to more valuable hydrocarbons and to separate hydrogen. The cementitious properties of phosphate-based materials are used to stabilize radioactive and chemical wastes. In the structural ceramics/composites program, more reliable structural ceramics and composites are developed for use in advanced, high-efficiency gas turbines. Other programs in the Ceramics Section focus on improving the structural reliability of ceramics and composites used in applications such as hot-gas filters. New programs in energy storage focus on improving thin-film capacitors and the performance of cathodes for molten carbonate fuel cells.

Since the last review, there have been several changes in the Section's staff. A new postdoctoral, Beihai Ma, was hired to work on development of ceramic membranes. Seven of our Laboratory Graduate Participants received graduate degrees and took up positions in industry or academia.

¹Special Term Appointee.

²Serves two sections.

³Northwestern U.

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⁶Tri-State U.

⁷Alfred U.

⁸U. of Illinois at Chicago.

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¹³Loyola U.

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¹⁵California State-Fresno.

¹⁶New Mexico Tech.

¹⁷Florida State U.

Our facilities have been upgraded through expansion of our laboratory space and several purchases. A 400-MPa cold isostatic press was obtained to produce parts with high green-state densities. This capability has proved to be especially important for fabrication of many superconductor components. Several new pieces of mixing equipment were obtained for our work on phosphate ceramics.

Superconductor Development

Superconductor development remains the largest program in the Ceramics Section. The goal of this program is to develop ceramic superconductors for applications in the generation, storage, and distribution of electrical energy. The general target is to produce, in collaboration with industrial partners, long lengths (≈ 1000 m) of conductor capable of carrying $>10^4$ A/cm² in magnetic fields of >2 T at temperatures > 35 K. Program focus is on the two systems that currently appear to be the most promising as practical engineering materials: Y-Ba-Cu-O (YBCO) and Bi(Pb)-Sr-Ca-Cu-O (BSCCO). However, to maintain its position in the forefront of high- T_c superconductor research, the program also investigates promising new materials as they are developed.

To be used in applications such as motors, generators, large magnets, and power transmission lines, superconductors must have high critical current density (J_c) and, equally important, they must be available in long lengths. Powder-in-tube (PIT) processing of BSCCO materials remains the most promising method for fabricating long superconductors with high J_c . In PIT processing, precursor powder is sealed in a silver tube, then mechanically worked and heat treated to form a thin tape of silver-sheathed superconductor.

The Section collaborates with two world leaders in PIT processing of BSCCO superconductors: American Superconductor Corporation (ASC) and Intermagnetics General Corporation (IGC). In the work with ASC, the focus is on optimizing the microstructure and properties of tapes made by the two-powder process, a synthesis procedure developed by the Ceramics Section and found to yield improved superconductor phase purity. As part of this collaboration, effects of stoichiometry and heat treatment conditions on phase-evolution kinetics are studied. Collaboration with IGC is directed more at issues associated with fabricating long lengths of superconductor. It has resulted in a 1260-m-long multifilament conductor with a J_c of up to 12,000 A/cm² at 77 K. From such a conductor, a magnet was constructed that generated a world-record field of 4.2 T at 4.2 K. We recently fabricated a BSCCO tape that carried 55 A at 77 K, a record for a PIT tape.

In collaboration with General Atomics, the Section is developing a modified PIT process (called the coated-wire-in-tube process) in which fine continuous silver wires are embedded in the superconductor core to increase the silver/superconductor interface area. This approach is based first on the widely made observation that well-aligned regions of superconductor form at interfaces with silver, and second on the widely held belief that most electrical transport in PIT samples takes place in these well-aligned regions.

Gold contact wires being attached to a bicrystalline YBCO superconductor sample in order to study electric current transport across an individual grain boundary

Because impediments to intergranular currents known as weak links dominate the behavior of YBCO, its J_c generally drops dramatically in weak magnetic fields (<0.01 T). We are working on understanding weak-link behavior and on minimizing its effects. For this work, as shown in the figure above, we have fabricated and studied unique samples containing engineered single grain boundaries. YBCO is useful for magnetic levitation because it exhibits relatively high resistance to flux creep, the motion of quantized magnetic flux through the superconductor. A collaboration involving the Ceramics Section, the Electromechanics and Superconductivity Applications (ESA) Section, and Commonwealth Research Corporation utilizes the levitation properties of YBCO in demonstrating flywheel energy storage. In this project, the Ceramics Section fabricated the YBCO levitators used in the flywheel designed and built by the ESA Section.

Many of the envisioned applications of high-temperature superconductors require greater resistance to the motion of magnetic flux, i.e., improved flux pinning. To improve flux pinning, nanophase oxides such as TiO_2 , ZrO_2 , and Al_2O_3 were added to $\text{YBa}_2\text{Cu}_3\text{O}_x$, $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$, and $\text{TlBa}_2\text{Ca}_2\text{Cu}_3\text{O}_x$. These additions resulted in formation of nanometer-scale compounds such as BaTiO_3 and BaAl_2O_4 and increased pinning by factors of 2 to 10 at temperatures below 50 K. In another approach, sol-gel methods were used to form intragranular nanophase inclusions directly during the synthesis reaction.

Methane Conversion and Utilization

In Alaska's far-north slope and beneath the deep waters of the Gulf of Mexico, natural gas resources remain untapped because costs of transportation are prohibitive. Over the past several years, extensive research efforts have focused on converting methane to value-added products, especially certain easily transportable fuels and gasoline additives that minimize air pollution from automobile exhaust gases. The first step in natural gas conversion is generation of so-called "syngas" (short for synthesis gas, a mixture of CO and H₂). Conventional processes require two or more steps to produce syngas and expensive cryogenic separation plants to produce the required high-purity oxygen. Another approach is steam reforming, but it is also energy- and cost-intensive.

As a cost-effective alternative to present-day approaches, the Ceramics Section has developed ceramic membranes that allow air to be used in syngas-generation reactions. The ceramic separates oxygen from air. The oxygen can then be used for partial oxidation of the methane (the chief constituent of natural gas) to form syngas. In principle, air flows on one side of the membrane while methane flows on the other side. Because the membrane is permeable to oxygen, but not to nitrogen, the oxygen diffuses through the membrane and reacts with the methane to generate the product gas. No external electrical source or circuitry is required because the membrane is a mixed ionic/electronic conductor. Maintaining the mechanical integrity of the membrane under the severe reactor operating conditions required that we develop a new material based on the Sr-Fe-Co-O system.

Reactor tubes extruded from the new Sr-Fe-Co-O composition have operated without failure for >1000 h at $\approx 900^{\circ}\text{C}$ with a methane conversion efficiency of >98%. While characterization of the microstructure and electrical, physical, and mechanical properties is continuing, results to date suggest that the material can withstand the normal operating stresses of a commercial reactor. Current efforts include development of the cold and hot seals required for commercial applications, development of alternative membrane materials with improved mechanical properties, design and fabrication of different reactor configurations, and finite-element analysis of reactor tubes to predict stresses and fracture under actual service conditions.

Based on the success of our work, DOE issued a solicitation for cost-shared proposals to take this technology forward to commercialization. From the responses to this solicitation, DOE recently selected a team, led by Air Products and Chemicals, that includes ANL and other partners for an eight-year, \$84 million project to further develop this revolutionary technology.

Waste Stabilization

The Ceramics Section has demonstrated a process for stabilizing hazardous, radioactive, and mixed wastes by using novel phosphate ceramics that set at room temperature. Chemically bonded ceramics based on magnesium, magnesium/potassium, iron, and zirconium phosphates can stabilize a wide range of DOE waste streams, such as contaminated ashes, salt cakes, and sludges, and hold promise for treating waste streams generated by the utility, chemical, and defense industries. The process stabilizes problem

wastes, such as secondary waste streams from thermal treatment processes (such as vitrification and plasma arc treatment) and other chemical processes for destroying organics. The process can also be used to stabilize wastes containing volatile species and pyrophorics, which cannot be treated by thermal stabilization.

Stabilization of these materials involves both chemical solidification and physical encapsulation of wastes, resulting in superior waste forms. Optimization of the phase compositions, microstructures, and mechanical and physical properties has led to the development of dense and durable waste forms. The figure below shows that our processes can produce waste forms at a commercial scale. Because of the high waste loading and low-temperature operation, chemically bonded phosphates are a low-cost alternative to other stabilization technologies. The process has been scaled up to stabilize actual wastes; collaborations with private industry are ongoing to demonstrate the stabilization of DOE waste streams.

*55-gallon ceramic waste form fabricated from ANL's
phosphate binders and contaminated soil*

In addition to stabilizing hazardous and radioactive wastes, the chemically bonded phosphate ceramics have been shown to bind various high-volume wastes such as contaminated plastic gloves and light bulbs. In addition, innocuous wastes such as plastics, cellulose fibers, ashes, and lumber can be made into inexpensive value-added construction products. The waste materials are mixed in powder or shredded form with the phosphate binder and fabricated into a variety of forms such as blowable or pumpable insulation, particle boards, and bricks. Because the phosphate binder is inorganic, the resulting products are nonflammable. Relevant properties of all resulting materials have been evaluated to establish their applicability for storage or for lightweight, fire-resistant, environmentally benign construction materials for energy conservation.

Structural Ceramics and Composites

In ongoing studies, monolithic ceramics and composites with fiber, whisker, and particulate reinforcements are being developed for high-temperature structural applications. Recent efforts have focused on continuous fiber-reinforced ceramic-matrix composites (see figure below) and have involved interactions with the Dow Corning Corporation. Effects of fiber strength and architecture and fiber/matrix interfacial characteristics on the mechanical properties of composites have been evaluated at ambient and elevated temperatures. These results are being used by ET's SI&NDE Section to guide its development of testing methods.

At ANL's state-of-the-art facility for testing composite materials, researchers are evaluating fiber damage that occurs during composite fabrication and service.

Fibers on fracture surface are shown at left; at very high magnification, fracture origins can be identified (right).

The strength of fibers in composites is measured by fractographic techniques after processing and after exposure to service environments. Comparing these strengths to those in the as-fabricated condition provides information on the nature of flaw generation and fiber damage during processing and in service. Fiber/matrix interfacial properties are evaluated by fiber pushout testing and electron microscopy on composites fabricated under various processing conditions. The flaw population and characteristics, fiber strength, and interfacial properties are then correlated with measured mechanical properties to gain critical information for producing composites with improved properties.

In collaborations with various hot-gas filter manufacturers (3M, Babcock & Wilcox, Industrial Filter and Pump, Pall Corporation, and DuPont-Lanxide), the objectives are to develop test protocols, evaluate microstructures and mechanical properties, and identify damage and failure modes of hot-gas filters. These studies provide much-needed information for materials selection and process optimization for hot-gas ceramic/composite filters, and help to ensure structural reliability of the filters in service environments. In one program, the state of the art in mechanical and microstructural evaluation and understanding of thermal barrier coatings is being assessed. Mechanical test procedures and protocols have been established, and coatings from several sources are being characterized to predict failure and, ultimately, to ensure structural reliability in service.

Fuel Cells

The goal of one collaborative program with ANL's Chemical Technology (CMT) Division is to develop improved cathodes for molten carbonate fuel cells (MCFCs). Because the lifetime of present-day MCFCs is limited by dissolution of the NiO cathode into the molten carbonate, new cathode compositions are being explored, including LiFeO_2 and LiCoO_2 . In addition, dimpled cathodes are being designed and fabricated to optimize electrical conduction and gas flow through the cathode.

We are also working with CMT to develop improved solid oxide fuel cells (SOFCs). The efficiency of a SOFC is dependent on transport of oxygen ions. If SOFCs are to be a viable power system for transportation, they must be able to operate at low temperatures. Therefore, a study of oxygen diffusion in a candidate electrode material, $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$ for $x = 0.1$ and 0.2 , was completed. Oxygen diffused via a vacancy mechanism, but too slowly at 500°C to allow for use of this material as an electrode in a low-temperature SOFC. Additionally, fuel cells must be robust to survive the rigors of a transportation system. Electrolytes will most likely be in the form of films, but each material's strength and fracture toughness are generally low. A program was started to improve strength (σ_f) and fracture toughness (K_{IC}) of a prototypical electrolyte, $\text{Ce}_{0.9}\text{Ge}_{0.1}\text{O}_2$. Initial results indicate that mechanical property improvements are possible. With addition of 10 wt.% Al_2O_3 , σ_f was doubled to 128 ± 11 MPa and K_{IC} was increased 25% to 1.60 ± 0.14 MPam^{1/2}.

Capacitor Development

A new DOE capacitor program, in collaboration with ANL's Materials Science Division, was initiated in January. The program goal is to reduce the size and weight of power electronic devices designed for electric and hybrid vehicles. Progress has been made on power electronic components such as MOS-controlled thyristors and insulated gate bipolar transistors. Additional effort on high-power capacitors is needed for load leveling and filter functions. As a subsystem component, the size, cost, and reliability of the high-power capacitor needs to be addressed for electric and hybrid vehicles.

ANL has extensive expertise in fabrication of ferroelectric thin films by metal organic chemical vapor deposition (MOCVD) and in characterization of their microstructures and properties. We are extending this capability to develop capacitors for power electronics. The MOCVD synthesis technique, which is widely used in semiconductor manufacturing, is

a method for depositing uniform layers over large surface areas. Most of our past efforts have focused on fabrication and electrical-property measurement of lead zirconate titanate films. Argonne is also collaborating with several companies that manufacture ferroelectric thin films for computer memory and decoupling capacitor applications. This technology is being adapted for development of high-power capacitors for the automotive industry.

High-Temperature Mechanical Properties

We are studying the steady-state deformation of a variety of important ceramics to determine deformation mechanisms, measure kinetics, and relate the kinetics to self-diffusion. Results can be used to optimize heat-treatment schedules and microstructures. Materials examined include dielectrics (BaTiO_3), mixed-conducting oxides ($\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$, LiFeO_2 , LiCoO_2 , and Li-doped NiO), in-situ-reinforced Si_3N_4 , and high-temperature superconductors ($\text{Tl}_2\text{Ba}_2\text{CaCu}_2\text{O}_x$).

In brief, BaTiO_3 was found to creep by grain-boundary sliding accommodated by diffusion. Activation energy was high and creep rate was nearly independent of oxygen partial pressure ($p\text{O}_2$). For deformation by diffusional flow, the creep rate of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ increased with increasing $p\text{O}_2$ and decreased with increasing Sr content. At 1400–1650°C, the Si_3N_4 deformed by pure diffusion at low stress and unaccommodated grain-boundary sliding at higher stress. LiCoO_2 was found to creep faster than LiFeO_2 , which crept faster than Li-doped NiO . These results suggest the relative dimensional stabilities during fuel cell operation. As for other high-temperature superconductors, the creep rate of $\text{Tl}_2\text{Ba}_2\text{CaCu}_2\text{O}_x$ increased substantially as $p\text{O}_2$ decreased.

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A. S. Wagh and D. Singh

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Thin Film Seeds for Melt Processing Textured Superconductors for Practical Applications

B. W. Veal, A. Paulikas, and U. Balachandran

Invention Report ANL-IN-94-130. DOE File No. S-83,001. U.S. Patent Application filed 04/15/96.

Method of Waste Stabilization Via Chemically Bonded Phosphate Ceramics

A. S. Wagh, D. Singh, and S.-Y. Jeong

Invention Report ANL-IN-95-140. DOE File No. S-85,477. U.S. Patent Application filed 03/18/96.

Phosphate Bonded Structural Products from High Volume Wastes

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Invention Report No. ANL-IN-95-005. DOE File No. S-84,056. U.S. Patent Application, 03/18/96.

Method of Producing Improved Microstructure and Properties for Ceramic Superconductors

J. P. Singh, R. A. Guttschow, J. T. Dusek, and R. B. Poeppel

Invention Report ANL-IN-91-072b. U.S. Patent No. 5,525,586 issued 6/11/96.

Method to Fabricate Long Length High-Tc Conductors with High Critical Current Density

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Invention Report ANL-IN-96-080, 07/96. U.S. Patent Application filed 03/31/97.

Oxygen Ion-Conducting Dense Ceramic

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Invention Report ANL-IN-92-095. U.S. Patent No. 5,580,497 issued 12/3/96.

Method of Waste Stabilization via Chemically-Bonded Phosphate Ceramics, Structural Materials Incorporating Potassium Phosphate Ceramics

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Programs of the Corrosion Section address specific concerns related to corrosion and the effects of various environments on mechanical behavior of materials used in several types of energy systems. The research is sponsored by various divisions of the Department of Energy (DOE) and the Nuclear Regulatory Commission. Research on aqueous, gaseous, and liquid-metal corrosion, thermodynamics and kinetics of gas/solid reactions, and high-temperature mechanical properties in three major program areas is summarized below.

Light Water Reactors

Fatigue and environmentally assisted cracking (EAC) of piping and pressure vessels in light water reactors (LWRs) are important concerns as extended reactor lifetimes are envisaged. Failure of reactor-core internal components after accumulation of relatively high fluence is also of interest. Nonsensitized austenitic stainless steels (SSs), e.g., control-blade sheaths and handles and instrument dry tubes of boiling water reactors (BWRs), that are subjected to relatively low or negligible stress levels become susceptible to intergranular failure by a degradation process commonly known as irradiation-assisted stress corrosion cracking (IASCC). Intergranular cracking has also occurred in safety-significant structural core components in BWRs, such as the top guide, shroud, and core plate; however, the relative contributions of neutron fluence, material composition, heat-treatment condition (sensitization), and fabrication variables (welding method and residual weld and fit-up stresses) to crack initiation and growth are not known. Research on EAC of LWR materials has focused on (a) fatigue of pressure vessel and piping steels, (b) crack growth in austenitic SSs, (c) IASCC of austenitic SSs, and (d) EAC in high-nickel alloys.

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Fatigue Testing of Carbon Steels and Low-Alloy Steels in Simulated Reactor Coolant Environments

Plain carbon and low-alloy steels and austenitic stainless steels are used extensively in light water reactor (LWR) steam supply systems as piping and pressure vessel materials. The environmental enhancement of fatigue crack growth rates in pressure vessel and piping steels in high-temperature water is well known. LWR coolant environments can also have significant effects on the fatigue lives of these steels. Under certain conditions of loading and environment, fatigue lives of these steels can be a factor of 70 lower in coolant environments than in air. Based on the existing fatigue S-N data, statistical models have been developed at ANL for estimating the effects of various material and loading conditions on fatigue lives of materials used in the construction of nuclear power plant components. Results of the statistical analysis have been used to estimate the probability of fatigue cracking in reactor components. Fatigue tests are being conducted in air and simulated LWR environments to obtain data under conditions where information is lacking in the existing S-N data base, and to determine the crack initiation and crack growth characteristics. Recent fatigue data, shown in the figure below, indicate that environmental effects on fatigue life in austenitic stainless steels are greater in low-dissolved oxygen (DO) than in high-DO water. These results are difficult to reconcile in terms of a slip-dissolution/oxidation mechanism that is commonly invoked to explain environmentally assisted decrease in fatigue life. It is possible that hydrogen-induced cracking is important in low-DO pressurized water reactor (PWR) environments. The existing fatigue data also indicate that even in air, the mean curve on which the current ASME Code design curve for austenitic stainless steels is based is nonconservative compared with the experimental data.

Fatigue lives of Type 316NG stainless steel tested at 288°C in air, simulated PWR water, and high-dissolved-oxygen water environments

Results of fatigue tests that examine the influence of reactor environment on formation and growth of short cracks in carbon and low-alloy steels indicate that the decrease in fatigue life of these steels in high-DO water is primarily caused by the effects of environment on the growth of short cracks. The next figure shows that relative to air, crack growth rates in high-DO water are nearly two orders of magnitude higher for cracks $< 100 \mu\text{m}$, and are one order of magnitude higher for cracks $> 100 \mu\text{m}$. Furthermore, in high-DO water, the surface cracks appear to grow entirely as Mode I tensile cracks normal to the stress axis whereas, in air or low-DO water, growth of surface cracks initially occurs

Crack growth rates plotted as a function of crack depth for low-alloy steel tested in air and water environments

as shear cracks $\approx 45^\circ$ to the stress axis. These results are consistent with slip-dissolution/oxidation model for enhanced crack growth rates in LWR environments.

Irradiation-Induced Stress Corrosion Cracking of Austenitic Stainless Steels in Simulated BWR Water

In recent years, failures of reactor-core internal components in both BWRs and PWRs have increased after accumulation of relatively high fluence ($>5 \times 10^{20} \text{ n}\cdot\text{cm}^{-2}$, $E > 1 \text{ MeV}$). The general pattern of the observed failures indicates that as nuclear plants age and neutron fluence increases, various apparently nonsensitized austenitic stainless steels become susceptible to intergranular failure. Some components (e.g., BWR core shroud, control-blade handle and sheath) are known to have cracked under minimal applied stress. Although most failed components can be replaced, some safety-significant structural components (e.g., BWR top guide, shroud, and core plate) would be very difficult or impractical to replace. Therefore, to understand this type of degradation, which is commonly known as IASCC, the structural integrity of these components after accumulation of high fluence has been a subject of concern and extensive research.

The primary effects of irradiation include alteration of microchemistry, microstructure, and mechanical properties of structural materials, which are usually fabricated from ASTM Type 304, 316, or 348 SSs. Irradiation produces defects and defect clusters in grain matrices and alters the dislocation network, dislocation loop, and dislocation channel structures, leading to radiation-induced hardening. Irradiation also leads to changes in the stability of second-phase precipitates and the local alloy chemistry near grain boundaries, precipitates, and defect clusters. Grain-boundary microchemistry that differs significantly from that of the bulk composition can be produced in association with not only radiation-induced segregation (RIS) but also thermally driven equilibrium and nonequilibrium segregation of alloying and impurity elements. Neutron irradiation also alters the water chemistry, especially near the grain-boundary surfaces in a crack-tip region.

For many years, irradiation-induced grain-boundary depletion of chromium has been considered as the primary metallurgical process that causes IASCC. One of the most important factors that has been considered by many investigators to support the chromium-depletion mechanism is the observation that the dependence of IGSCC of nonirradiated thermally sensitized material and of IASCC of BWR-irradiated solution-annealed material on water chemistry (i.e., oxidizing potential) is similar. Recently, attention has also focused on the effects of fabrication-related variables, i.e., grain-boundary segregation and depletion of alloying and impurity elements by thermal processes, the effect of final thermomechanical treatment, hardening by cold work, the use of recycled scrap metals, uptake of minor impurities during iron- and steelmaking, and contamination by minor impurities during welding of field components such as BWR core shrouds.

Our effort has focused on (a) slow-strain-rate-tensile testing of model stainless steel alloys irradiated in the Halden reactor, (b) characterization of deleterious impurities from contamination during welding of core internal components, (c) hardness and grain size distribution in weld heat affected zones (HAZs), and (d) analysis of fluorine and oxygen in the materials. Preliminary results obtained on base-metal components, such as neutron-absorber tubes and control-blade sheath irradiated in several BWRs, indicated that minor impurities such as fluorine and oxygen can be deleterious. Consistent with this, significant fluorine and oxygen contamination was observed in a BWR core shroud weld specimen and in nonirradiated weld specimens of Type 304 SS prepared by a shielded-metal-arc (SMA) process.

Analysis of Fluorine in BWR Components

To explore the role of fluorine in IASCC of stainless steel, the segregation behavior of fluorine to grain boundaries in BWR neutron-absorber tubes and a core shroud weld was analyzed with a scanning Auger microscope (SAM). Auger specimens were charged with hydrogen and fractured in situ in the ultrahigh vacuum of the SAM. Auger electron spectroscopy (AES) depth profiling appeared to be superior to other techniques for determining grain-boundary compositions of most nonmetallic impurities at low concentrations. Derivative AES signals were obtained with a focused electron beam of $\approx 1 \mu\text{m}$ diameter. The grain-boundary profile of a core shroud weld specimen was determined by an automated depth-profiling technique, i.e., repeated argon ion sputtering and acquisition of an AES fluorine signal at 625 eV.

Initial results indicated that fluorine concentration is higher on grain boundaries than in grain matrices. Therefore, further analysis was conducted on specimens from neutron-absorber tubes and a core shroud weld. The next figure shows the relative intensities of the fluorine signal at 625 eV from ductile and intergranular fracture surfaces of the neutron-absorber tubes. The commercial-purity (CP) and high-purity (HP) Type 304 SS absorber tubes were irradiated to a fluence of $\approx 2.0 \times 10^{21} \text{ n}\cdot\text{cm}^{-2}$ in a BWR. Average fluorine concentrations were significantly higher on grain boundaries than in grain matrices, but absolute concentrations on grain boundaries could not be determined.

Fluorine 625-eV intensities from spots on ductile (grain matrix, denoted "D") and intergranular (grain boundary, denoted "I") fracture surfaces in (a) commercial-purity and (b) high-purity BWR neutron-absorber tubes

Auger electron spectroscopy analysis of grain-boundary fluorine distribution was also conducted on the Type 304L SS BWR core shroud weld. The HAZ of the weld specimen was fractured in-situ in the ultrahigh vacuum environment of the SAM, and one of the grain boundaries was selected and analyzed by a depth-profiling technique. The fluorine signal (at 625 eV) as a function of distance beneath the grain boundary is plotted in the figure below and shows that fluorine content is significantly higher on grain boundaries than in grain matrices, probably as a result of thermal segregation during cooling of the HAZ. Because of its very low solubility in stainless steels, fluorine, together with oxygen, will segregate by a thermal process to local sites such as matrix/precipitate interfaces, grain boundaries, and stacking faults; however, irradiation-induced segregation cannot be ruled out and further investigation will be needed to clarify this point.

Intensity of fluorine AES signal as function of sputter distance from grain boundary surface in Type 304L core-shroud weld HAZ

Secondary-Ion Mass Spectroscopy of Fluorine and Oxygen in Laboratory Welds

Most internal core components in LWRs are welded by an SMA or submerged arc process in which the welding electrode is coated with a flux that contains fluorine, e.g., CaF_2 . The detection limit of fluorine in SMA welds of Type 304 SS, by the wet-chemistry method, is <30 wppm when several grams of material were available for analysis, but only ≈ 120 wppm (≈ 360 appm) when the sample mass was <1 mg. Therefore, fluorine and

oxygen contamination in several specimens was analyzed by secondary-ion mass spectroscopy (SIMS). An SMA weld specimen that contained the fusion zone and the HAZ was polished, etched in a dilute oxalic acid solution, and ultrasonically cleaned in alcohol before SIMS analysis. Secondary ions of fluorine that were ejected from the weld fusion zone and the HAZ were analyzed by using cesium primary ions in one case and oxygen ions in another.

The figure on this page shows distribution maps of fluorine and oxygen secondary ions obtained with cesium primary ions. The figure provides direct evidence that local regions high in oxygen concentration are also high in fluorine concentration. Although the solubility of fluorine and oxygen in steels is very low or negligible, the arc in the weld fumes may promote dissolution of atomic fluorine and oxygen in the fusion zone and the HAZ. This observation is consistent with the results of AES analyses of BWR core shrouds and neutron-absorber tubes, which showed a high fluorine content in oxide or spinel precipitates. It also seems to be consistent with the observation, by other researchers, that an oxide film promotes fluorine-assisted IGSCC.

*Fluorine (top) and oxygen (bottom) ion maps of SMA weld of Type 304 .
SS determined by SIMS analysis*

In summary, the susceptibility of the HAZ of core shroud welds to SCC is usually attributed to thermal sensitization during welding, but we believe that certain other factors may also increase susceptibility, including (a) oxygen, fluorine, and sulfur contamination that occurs during welding; (b) maximum in hardness at a distance of $\approx 1\text{-}3$ mm from the fusion zone; (c) hardened surface layers that are a result of postwelding grinding;

and (d) thermal sensitization. These other factors may be more significant for materials in which thermal sensitization is unlikely to occur, e.g., Type 304L SS, but cracking has been observed. Additional results after irradiation of the laboratory-produced weld specimens and microstructural and microchemical analyses of BWR core shroud specimens will provide more definitive evidence of the above factors and a more quantitative understanding of the cracking mechanism.

Environmentally Assisted Cracking of Alloys 600 and 690 in LWR Coolant Environments

Alloys 600 and 690 are used for a variety of structural elements in reactor systems in addition to their use as steam generator tubing. Cracking has been observed in a number of components, e.g., instrument nozzles and heater thermal sleeves in the pressurizer, penetrations for control-rod-drive mechanisms in reactor vessel closure heads in the primary system of PWRs, and in shroud-support-access-hole covers in BWRs. Experience strongly suggests that materials that are susceptible to SCC are also susceptible to environmental degradation of fatigue life and fatigue-crack-growth properties. Information has been obtained on the effect of temperature, load ratio, and stress intensity on EAC of these alloys in simulated BWR and PWR water, and the crack growth rates (CGRs) of these materials have been compared with rates determined in air.

The graphs on the next page show the dependence of the CGRs of annealed Alloy 600 and annealed and thermally treated Alloy 690 specimens on ΔK (i.e., $K_{\max} - K_{\min}$) for experiments in air at 289°C and the combined data at three temperatures for each alloy. The coefficient-of-fit of the data to a simple power law is quite good and the results over a wide range of load ratios ($R = 0.2-0.95$) indicate that the CGRs are independent of temperature in the range of 35-289°C.

Numerous corrosion-fatigue experiments were conducted on compact-tension specimens of Alloys 600 and 690 in various heat-treatment conditions in HP water that contains <5 and 300 ppb dissolved oxygen (DO). Best-fit correlations for the CGRs in water versus ΔK were obtained for each specimen and for the combined data from several specimens of the same material with different heat treatments. Typical results for the two alloys in these environments are shown in the graphs on page 118.

Compositional variations and differing heat treatment conditions influence mechanical properties (e.g., yield and ultimate strengths), grain size, and carbide distribution in the alloys. Within our present data base, limited individual comparisons of CGRs under nominally the same loading and temperature conditions can be made for the two alloys in the same test (in water or in air), or among specimens of the same alloy but from different heats and with different heat-treatment conditions. Fewer direct comparisons are possible between CGRs in water and air. Also, normal variability in individual CGR measurements may either obscure or enhance any differences in the rates that may exist.

Dependence of CGRs of Alloys 600 and 690 on ΔK in air at 289°C and combined data for each alloy at 35, 130, and 289°C

Dependence of CGRs on ΔK at 289°C: Alloy 690 specimens in deoxygenated HP water (left), and Alloy 600 specimens in oxygenated HP water (right)

Therefore, the effect of HP water that contains <5 and ≈ 300 ppb DO on CGRs relative to that in air was determined from a ratio of the CGRs at 289°C in water to that in air at 289°C obtained from the best-fit correlations to the data. The results in HP water with <5 ppb DO (left graph on next page) indicate that the CGRs for Alloy 600 are higher in

water than in air at ΔK values $< 6 \text{ MPa}\cdot\text{m}^{1/2}$ and are lower in water than in air at higher values of ΔK . In contrast, the CGRs of Alloy 690 are only slightly higher in water than in air over the entire range of ΔK . The results in the right-hand figure below indicate that the rates for Alloy 600 (0.03% carbon) are higher in water with ≈ 300 ppb DO than in air at ΔK values $< 18 \text{ MPa}\cdot\text{m}^{1/2}$ and are lower in water than in air at higher values of ΔK . At a ΔK of $\approx 2 \text{ MPa}\cdot\text{m}^{1/2}$, the rate in water is higher by a factor of ≈ 10 than in air. At this ΔK , CGRs for Alloy 600 in water that contained < 5 ppb DO were higher by only a factor of ≈ 2 than in air. In a general way, the results show the interplay of alloy composition (Alloy 600 versus 690), environment (low and high levels of DO), and loading condition (ΔK) on CGRs relative to those in air. Differing heat treatments and temperature within the range $240\text{--}320^\circ\text{C}$ had a relatively minor effect on CGRs in our experiments.

Dependence on ΔK of ratio of CGRs of Alloy 690 in deoxygenated HP water and air (left) and ratio of CGRs of Alloy 600 in oxygenated HP water and air (right)

The morphology of corrosion-fatigue cracks in the Alloy 600 and 690 specimens was transgranular cracking in air and HP water because of the strong contribution of mechanical cyclic loading in tests at load ratios of 0.1-0.8 and because of the relatively small degree of crack extension that occurred in tests at load ratios > 0.8 .

The present results suggest that larger differences in the CGRs of the two materials in water and of the materials in water versus air may occur under constant load, i.e., at $R = 1.0$ and $\Delta K = 0$. Because the corrosion-fatigue correlations cannot be extrapolated to ΔK values $< 2 \text{ MPa}\cdot\text{m}^{1/2}$, experiments are being conducted under constant load on the alloys with various heat-treatment conditions in oxygenated water at 289°C to provide this information.

Fossil Energy

Studies of the high-temperature corrosion of metallic and ceramic materials are being conducted in simulated coal-gasification atmospheres and coal-combustion environments typical of conventional pulverized coal-fired boilers, fluidized-bed systems, and low- NO_x boilers and High Performance Power Systems (HIPPS) being developed by DOE under the Combustion 2000 program. This effort is directed at establishing mechanisms of corrosion, predicting the onset of breakaway corrosion, and evaluating the role of stress and deformation in corrosion processes. Sulfidation and chloridation resistance of structural

alloys (namely, model Fe-Cr, Fe-Cr-Ni, Fe-Cr-Al alloys and Fe-Al intermetallics with controlled additions of several refractory metals and rare earth elements) is being evaluated by thermogravimetric tests, and postexposure microstructure of corrosion scales is being characterized by several electron/optical microscopy techniques. Alloy additions are made by bulk alloying and as overlay coatings. Another major thrust of the program is to evaluate the effects of corrosive environments on the tensile and creep properties of metallic alloys and fracture toughness properties of monolithic and ceramic/ceramic composite materials. Further collaborative programs, supported by Fossil Energy and the Materials Division of Basic Energy Sciences in DOE, are in progress to examine fundamental aspects of scaling in structural materials exposed to oxidizing environments and to develop ultrahigh-strength Mo-Si intermetallic alloys.

Corrosion Behavior in Advanced Combustion Power Generation Systems

In support of the DOE Combustion 2000 program, calculations were performed to evaluate the chemistry of gaseous and condensed products that arise in combustion of coal. The chemistry calculations involve determination of the type and amount of combustion products over a wide temperature range (700-1700°C) at 1 atm pressure. In addition, nonequilibrium conditions due to kinetic factors are incorporated to assess their influence on the chemistry of condensed products in these environments. Experiments are being conducted to evaluate corrosion performance and establish the role of corrosion in the degradation of mechanical properties of candidate materials.

In coal-fired combustion systems, the presence of slag constituents determines the thermodynamic activity of various deposit constituents, and alkali-sulfate-induced corrosion is generally not dominant. The degradation mode of ceramic materials in coal-fired combustion systems is via reactions with coal ash. This type of degradation depends on material composition, slag chemistry (acidic or basic), and gas-phase environment (either oxidizing or reducing). The slags generally contain phases such as SiO_2 , Al_2O_3 , CaO , Fe_2O_3 , Na_2O , a combination of these phases, and other ash constituents, depending on the coal feedstock. X-ray diffraction (XRD) data for deposits, obtained during a Combustion and Environmental Research Facility (CERF) run with combustion of Pittsburgh coal, showed the dominant phases to be mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), ordered calcian albite ($[\text{Na,Ca}] \text{Al} [\text{Si,Al}]_3\text{O}_8$), and hematite (Fe_2O_3). The melting temperatures of these phases are $> 1400^\circ\text{C}$; reactions between these compounds and the ceramic materials occur primarily in the solid state, and little or no penetration of the ceramics by liquid phase from the coal combustion environment occurs. Similarly, XRD data for the deposits, obtained during a CERF run with combustion of Alaskan/Russian blend coal, showed the dominant phases to be anorthite ($\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), calcium aluminum silicate ($\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2$), and ordered sodium anorthite ($(\text{Ca,Na}) (\text{Al,Si})_2 \text{Si}_2\text{O}_8$). These calcium-rich silicates also have melting temperatures $> 1400^\circ\text{C}$, and reactions between the coal ash deposit and the ceramics occur predominantly in the solid state. The experimental results also indicate that Na_2O activity in the deposits (with combustion of coals that contain Fe_2O_3 and CaO) is sufficiently low to form liquid sodium silicates of various types in coal-fired systems.

Experimental work requires procurement of materials such as highly alloyed metallic alloys, monolithic ceramic materials, metal-matrix ceramic composites and ceramic-matrix

ceramic composites, together with laboratory testing of these materials in air and in simulated combustion atmospheres that include coal ash deposits. In addition, the materials are also tested by exposing samples of several ceramic materials in the CERF at the Federal Energy Technology Center and the North Dakota combustor test facility. The CERF is a state-of-the-art, 0.5 million Btu/h pilot-scale combustion facility designed to achieve similarity with full-scale utility and industrial boilers. The ultimate goal of the program is to assess the materials from the standpoint of corrosion performance, mechanical integrity, microstructural stability, and long-term durability for application in heat transfer surfaces in HIPPS.

Oxidation of ceramic materials, such as monolithic and composite SiC and SiC-dispersed Al_2O_3 , involves reaction of carbide phases to form silicon-rich oxides. The rate of oxidation is generally influenced by the porosity of the material, the amount of free silicon and/or aluminum in the material, and exposure temperature. Earlier work showed a low rate of oxidation for these materials when exposed in dry air at 1200°C . One exception was SiC(p) in Al_2O_3 material, which exhibited a large weight gain in the early stage of exposure due to oxidation of free aluminum in the surface region of the sample. After ≈ 100 h of exposure, the oxidation rate slowed but was still higher than in other materials, indicating oxidation of free aluminum in the interior of the material dictated by the diffusion of oxygen. The results also established that these ceramic materials undergo passive oxidation at 1000 - 1400°C in a dry-air environment. Water vapor contents up to 30 vol.% in air had minimal effect on the oxidation rate of these materials at 1200°C . In laboratory slag tests, specimens showed significant surface cracking but the slag itself did not seem to penetrate the specimens, as indicated by EDX analysis of specimen cross sections. Because Na_2O activity in the slag is relatively low, the deposit did not melt, but significant bubbling of the slag, probably due to evolution of CO and CO_2 , was noted. The experimental results also indicated that Na_2O activities in the deposits (with combustion of coals that contain Fe_2O_3 and CaO) were sufficiently low to form liquid sodium silicates of various types in coal-fired combustion systems.

To examine the role of exposure environment on mechanical properties, several specimens of the ceramic materials were preexposed for 200 h at 1200°C to dry air, with and without water vapor, to three coal slags and to simulated combustion gas with and without water vapor. After exposure, the specimens were mechanically tested in vacuum at 1200°C . During these tests, the specimen surfaces exposed to the slag environments were on the support side of the fixture, ensuring a tensile mode of loading for the surfaces exposed to corrosive environments. The next figure shows the flexural strength of the four materials after 200 h of exposure to three slags with and without water vapor. The results show that the strength of the alpha SiC and SiC in silicon-matrix materials with almost no porosity differed negligibly after exposure to environments with and without water vapor. This finding also indicates that the flexural strength of the material with a free silicon content of 47% is inherently lower and that oxidation/corrosion is not of concern, based on these short exposure tests. Siliconized SiC (with 8% free Si and 2% porosity) and SiC(p) in alumina (with 10% Al and 1% porosity) showed a substantial increase in flexural strength in the presence of water vapor when compared with strengths of specimens exposed to an

Effect of 200-h exposure in various slag-containing environments at 1200°C on flexural strength of several ceramic materials

environment without water vapor. Additional experiments are underway to establish performance envelopes in terms of temperature, exposure time, and mechanical properties for candidate materials for application in coal-fired systems.

Materials Performance in Coal Conversion Systems

In coal-gasification and integrated gasification combined-cycle (IGCC) environments, the gas chemistry is such that oxygen partial pressures are relatively low and sulfur partial pressures are moderate to high, depending on sulfur content of the coal feedstock. In such environments, structural materials are subjected to sulfidation attack and catastrophic corrosion, especially at elevated temperatures. Because the major objective in the development of corrosion-resistant alloys is to form a slow-growing scale that is well bonded to the substrate alloy, research is underway on alumina-forming alloys to modify the surface regions of chromia-forming alloys for enhanced nucleation-growth of the oxide scale in the early stages of alloy exposure. Surfaces have been modified by several techniques to alter the chemical composition of the surface/subsurface regions and influence scaling during early stages of exposures at elevated temperatures. Coatings have been developed via pack diffusion, low-pressure plasma spray, sputter deposition, and electrospray deposition (ESD) techniques and were examined for corrosion resistance in sulfur-containing atmospheres. Tests were conducted to evaluate the sulfur resistance of coatings, determine the influence of coating technique on corrosion, and compare the corrosion rates of coated and noncoated structural alloys in simulated gasification atmospheres. The results showed substantial improvement for pack diffusion coatings and an ESD Cr carbide/Al coating in these reducing environments. Experimental results on alumina-forming Fe-based alloys showed that a threshold Al concentration of >12 wt.% is needed to form a protective scale in oxygen/sulfur mixed gas environments.

Several specimens with Al-enriched surfaces, applied on Type 316 stainless steel and Alloy 800 substrate alloys by the ESD process, were tested in simulated gasification environments that contained H₂S with or without HCl. In the first experiment, performance of the ESD coatings (which included Fe₃Al with differing bond coats of refractory metals and/or noble metals) was compared with that of uncoated austenitic alloys. All of the Fe aluminide coatings were resistant to sulfidation and chloride attack.

whereas the base alloys were susceptible to general corrosion and pitting attack, especially in the HCl-containing environment. The weight change data and extensive microscopic analyses of tested specimens showed that the bond coats themselves do not significantly influence the corrosion process. Further, the corrosion behavior of FeAl-coated alloys were compared with the performance of several commercial high-Cr alloys after exposure in simulated gasification environments with and without HCl (see figure below). Corrosion performance of the FeAl-coated specimen is comparable to or better than those of most other materials tested. Further, the cost of materials such as HR 160 and Alloy 556, which exhibit corrosion rates similar to FeAl layered specimen, will be a factor of 5 to 10 times higher than that of the FeAl-coated specimen.

Weight change data for several commercial alloys and FeAl-coated Type 316 stainless steel after exposure in O/S mixed gas of composition $p_{O_2} = 1.2 \times 10^{-23}$ and $p_{S_2} = 5.2 \times 10^{-10}$ atm, typical of gasification atmospheres, at 650°C: without HCl (left) and with $p_{HCl} = 2.1 \times 10^{-3}$ atm (right)

Specimens with Al-enriched surfaces, applied on Type 316 stainless steel and Alloy 800 substrate alloys by the ESD process, were also tested in simulated combustion environments that contained SO_2 with or without HCl. In the absence of HCl, the uncoated alloys developed scales of (Fe, Cr) oxide or Fe oxide and tended to crack and spall. The aluminide-coated alloys showed a small weight gain due to the development of a thin, adherent alumina scale. In the presence of HCl, both the uncoated and coated alloys showed substantial weight loss at 650°C. The attack was most notable in the Fe_3Al -coated alloy, less notable in the uncoated alloys, and least notable in the FeAl-coated alloy. The primary cause for the increased corrosion of Fe_3Al -coated alloys seems to be a low concentration of Al in the coating that resulted from dilution of the deposit layer with substrate constituents. Even though Fe_3Al contained ≈ 14 wt.% Al, the coated alloy contained < 8 wt.% Al after fabrication. The results also indicate that a threshold Al concentration of > 12 wt.% is necessary to improve the corrosion resistance of structural alloys. Further, the corrosion behavior of Fe-Al coated alloys was compared with the performance of several commercial high-Cr alloys after exposure in simulated combustion environments with and without HCl (see next figure). The corrosion performance of the FeAl-coated specimen is comparable to or better than those of most other materials tested.

Weight change data for several commercial alloys and FeAl-coated Type 316 stainless steel after exposure in O/S mixed gas of composition $p_{O_2} = 6.7 \times 10^{-3}$ and $p_{S_2} = 1.5 \times 10^{-5}$ atm, typical of combustion atmospheres, at 650°C: without HCl (left) and with $p_{HCl} = 1.7 \times 10^{-3}$ atm (right)

In the absence of HCl, all of the alloys except HR 160 exhibited negligible weight gains and the scales were predominantly Cr_2O_3 . HR 160 is the only alloy among those tested that contains ≈ 38 wt.% Ni, but whether the higher Ni content of this alloy had contributed to higher oxidation in the alloy is unknown. However, the oxidation rate is still negligibly small. In the presence of HCl in the environment, all the alloys except HR 160 exhibited substantial weight loss during the 900 h exposure. HR 160 contains 2.75 wt.% Si, which upon oxidation at the chromia/alloy interface, may act as a second barrier against cation migration outward and Cl penetration inward, resulting in negligible corrosive attack. MA 253, which contains 1.8 wt.% Si, exhibited weight loss for ≈ 300 h, beyond which the loss was negligible. Other chromia-forming alloys such as Type 310 stainless steel, Alloy 556, and Alloy 800 continued to lose weight during the 900 h exposure. The FeAl-coated Type 316 stainless steel, even though forming alumina scale, was found susceptible to corrosion in the HCl-containing combustion atmosphere and the rate was slightly higher than that of Alloy 556.

Fusion Energy

Structural materials in a fusion reactor will be cooled by either a liquid-metal or aqueous coolant while undergoing displacement damage and transmutation reactions that produce hydrogen and helium atoms within the materials due to 14-MeV-neutron irradiation. Austenitic stainless steels, ferritic steels, and vanadium-base and niobium-base alloys are likely candidates for the first wall and divertor in the International Thermonuclear Experimental Reactor (ITER) and in subsequent power-producing fusion reactors. Current research is directed toward the acquisition of material-property data for design and construction of ITER and subsequent reactors. Studies include evaluations of the tensile, creep, fracture-toughness, and swelling properties of vanadium-base alloys after fast-neutron irradiation in the Experimental Breeder Reactor-II, High Flux Isotope Reactor, Advanced Test Reactor, and Fast Flux Test Facility at 380 to 600°C, and development of electrically insulating coatings on vanadium-base alloys in liquid lithium. Substantial research effort is underway to evaluate the influence of interstitial elements such as oxygen and hydrogen on the morphological changes in the materials and on the

mechanical properties of different heats of vanadium-base alloys. Work on aqueous corrosion of vanadium- and niobium-base alloys in deoxygenated water has been completed.

Mechanical Properties of Irradiated V-Cr-Ti Alloys

Vanadium-base alloys are attractive for structural materials in fusion reactors because of their good mechanical, corrosion, and irradiation performance. Recent attention to vanadium alloys has focused on low-temperature irradiation performance of V-4Cr-4Ti, especially tensile and fracture behavior after irradiation at $<450^{\circ}\text{C}$. From several irradiation experiments at $80\text{--}430^{\circ}\text{C}$, some heats of V-4Cr-4Ti exhibited low uniform elongation as a result of complete loss of work-hardening capability. Significant susceptibility to loss of work-hardening capability (LWHC) has been not observed at irradiation temperatures $\geq 500^{\circ}\text{C}$, however. Initial assessment indicates that the LWHC phenomenon in V-4Cr-4Ti is strongly dependent not only on heat but also on irradiation variables at $<430^{\circ}\text{C}$. This is important in understanding the phenomenon because susceptibility to LWHC at low temperatures under fusion-relevant conditions is considered to be a major factor in governing the minimum operating temperature of a fusion reactor. Therefore, a systematic evaluation of the phenomenon was conducted on a wider variety of alloys, namely, V-Ti, V-Cr-Ti, and V-Ti-Si alloys irradiated at $<430^{\circ}\text{C}$ in several conventional and helium-charging irradiation experiments in the FFTF-MOTA, HFIR, and EBR-II.

Tensile specimens with a gauge length of 7.62 mm and a gauge width of 1.52 mm were machined from $\approx 1.0\text{-mm}$ -thick sheets that had been produced by rolling a $\approx 3.8\text{-mm}$ thick plate at 25 or 400°C . Specimens from the V-Cr-Ti alloys were annealed at $950\text{--}1125^{\circ}\text{C}$ for 1 h in an ion-pumped vacuum system, whereas specimens from V-Ti and V-Ti-Si alloys were annealed at $1000\text{--}1050^{\circ}\text{C}$ for 1 h. Following irradiation, retrieved specimens were cleaned ultrasonically in alcohol and tested without the customary degassing treatment at 400°C (used to expel hydrogen). Tensile properties were measured at the irradiation temperature in flowing argon at a strain rate of 0.0011 s^{-1} .

Uniform plastic strains of the alloy specimens irradiated at $200\text{--}600^{\circ}\text{C}$ as a function of irradiation/test temperature are summarized in the next figure. Uniform elongation of specimens from three heats of V-4Cr-4Ti exhibited considerable variation in the temperature range of $380\text{--}430^{\circ}\text{C}$. Understanding the cause of the large heat-to-heat variation of work-hardening capability at $<430^{\circ}\text{C}$ is therefore of major importance. Based on the observation that work-hardening behavior is sensitive to not only heat but also subtle irradiation variations, it is likely that one or more minor impurities are involved. One heat of V-3Ti-1Si exhibited excellent resistance to loss of work-hardening behavior when irradiated at $390\text{--}430^{\circ}\text{C}$. This heat also retained a very low ductile-brittle transition temperature (DBTT $< -190^{\circ}\text{C}$) after conventional irradiation at $420\text{--}600^{\circ}\text{C}$ to $14\text{--}33\text{ dpa}$. The plot below provides an estimate of the approximate threshold temperature above which uniform elongation of these alloys is higher than a threshold level, e.g., 2%. The threshold temperature is considered to be an important consideration in governing the minimum operating temperature of a fusion reactor.

Uniform plastic strain of V-Ti, V-Cr-Ti, and V-Ti-Si alloys as function of irradiation temperature (same as test temperature)

Uniform plastic strain of V-Cr-Ti alloys as function of irradiation temperature (same as test temperature)

A similar plot for V-Cr-Ti alloys is shown in the graph immediately above. The threshold temperature required to meet a minimum uniform elongation of 2% ($T_{2\%}$) appears to be anywhere between ≈ 320 and $\approx 470^\circ\text{C}$, depending on alloy type, heat, and basis of extrapolation. One heat of V-15Cr-5Ti (BL-24) showed high uniform elongation after irradiation at $\approx 420^\circ\text{C}$ in the FFTF-MOTA.

In summary, the work-hardening capability and uniform tensile elongation of V-Ti, V-Cr-Ti, and V-Ti-Si alloys during irradiation at 380 – 430°C appear to vary strongly from alloy to alloy and heat to heat. A strong heat-to-heat variation has been observed in three heats of V-4Cr-4Ti. Work-hardening capability of V-4Cr-4Ti alloys appears also to be influenced significantly by variations in irradiation conditions. Differences in susceptibility to LWHC from one heat to another correspond to $\approx 100^\circ\text{C}$ or more in minimum allowable operating temperature (e.g., 450 versus 350°C) for a fusion reactor.

Effect of Oxygen on Mechanical Properties of V-Cr-Ti Alloys

One concern in the use of vanadium-base alloys at elevated temperatures is their relatively high affinity for interstitial impurities, i.e., oxygen, nitrogen, hydrogen and carbon. These impurities are known to increase hardness and strength with an accompanying decrease in ductility and formability. In particular, oxygen may degrade alloy properties during service through solution in the matrix or formation of oxide particles in the matrix.

along the grain boundaries, and/or on the surface of the alloys. Also, the presence of titanium in vanadium-base alloys makes the problems associated with oxygen more of a concern and more complicated. This is because titanium is a stronger oxide former than either vanadium or chromium and is known to trap (or bind) oxygen in solution in refractory metals and alloys and also to act as a scavenger of oxygen. Therefore, a good understanding of the oxidation behavior and oxygen effect on the mechanical properties of the alloys intended for elevated-temperature use is of significant importance. The present program involves kinetics of oxidation of vanadium-base alloys with a wide range of chromium and titanium contents, effect of oxygen partial pressure in the exposure environment on the oxidation kinetics and microstructure of the alloys, and correlation of oxygen content and oxygen diffusion on the mechanical properties of the alloys.

To evaluate the effect of oxide scale formation and oxygen penetration into the substrate alloy, tensile behavior of the alloy was examined as a function of oxygen ingress and oxide scale formation. Specimens were exposed to air for 24-2060 h at 500°C and then tensile-tested in air at either room temperature or 500°C at a strain rate of $1.8 \times 10^{-4} \text{ s}^{-1}$. Stress/strain behavior of the alloy was virtually unaffected by 24 h of exposure in air at 500°C. As exposure time increased to 250 h, alloy strength increased, with some loss in tensile ductility. In the exposure range of 250-1000 h, the alloy had essentially the same ultimate tensile strength but showed substantial reduction in tensile ductility. Further exposure of the alloy to air at 500°C caused loss of strength and tensile ductility, as evidenced by the stress/strain curve for the specimen that was preoxidized for 2060 h. The figure below illustrates variations in maximum engineering stress and uniform and total elongation as a function of preoxidation time in air at 500°C for tests conducted at room temperature and 500°C. The information will be used to develop correlations among alloy chemistry, exposure conditions, and mechanical properties of alloys with emphasis on predictions of long-term performance of these alloys in a fusion reactor application.

Maximum tensile stress (left) and uniform and total elongation (right) as function of preexposure time in air for specimens of V-5Cr-5Ti alloy tested in air

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Irradiation Performance

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The principal objective of the activities in the Irradiation Performance Section (IPS) is to determine and assess the behavior of neutron-irradiated materials throughout the life cycle of the material from its in-reactor performance through its likely behavior during its ultimate interment in a repository. This environment includes neutron damage and chemical, metallurgical, and mechanical processes that occur during operation in fission or fusion reactors. Subsequent environments could include long-term storage in either water or air with degradation mechanisms attendant to these environments, together with the consequent concerns for maximum containment of the radioactive species. The Section's activities have changed from their former focus on fuel development to the characterization of a potpourri of irradiated materials for a variety of projects. The most significant change has been the increased effort on fuel, cladding, and structural materials for light water reactors (LWRs). The Section operates the Alpha-Gamma Hot Cell Facility (AGHCF) and the Irradiated Materials Laboratory (IML), in which it conducts postirradiation examinations and investigations. The capabilities of these facilities are rapidly becoming unique in the Department of Energy complex as other similar facilities are being shut down for budgetary reasons.

Fission Reactor Materials

The Section's activities in the fission reactor area are focused principally on LWRs but also include work on test reactor fuels and advanced naval reactor materials. The capabilities of the Section's AGHCF and associated Electron Beam Laboratory (EBL) are well suited for research in these areas. The principal current and upcoming activities in fission-reactor research are described below.

LWR Fuel under Accident Conditions

The U.S. Nuclear Regulatory Commission (NRC) has recognized the need to take a close look at its regulations that govern the allowable behavior of LWR fuel rods under design-basis accidents, in particular, the loss-of-coolant accident (LOCA). For the LOCA, the NRC criteria include those for cladding embrittlement (10CFR50.46), evaluation models (Appendix-K features for cladding oxidation, cladding swelling, incidence of rupture, and flow blockage), and best-estimate LOCA analyses (Regulatory Guide 1.157). In light of the significant changes made over the years in Zircaloy cladding materials, and the now-recognized deleterious effects brought about by the high-burnup levels, greater than 50,000 MWd/MT, now being achieved, the NRC has placed a program at ANL to investigate whether the current criteria, which were developed in the 1970s on nonirradiated Zircaloys of that time, are still applicable to the current cladding materials at the high burnup.

The IPS, in conjunction with the Corrosion Section and the Mechanics of Materials Section, has begun a comprehensive program to investigate the behavior of high-burnup fuel rod segments under LOCA conditions and to determine the mechanical properties of high-burnup cladding that are needed to analyze rod performance under LOCA and other transient conditions. This program, begun in FY 1997, is expected to continue for four years.

A key element of this program is determination of the oxidation kinetics of the high-burnup cladding. It is believed that the reaction kinetics at the higher burnups accelerate because of physical changes in the oxide layer. Hence, high burnup cladding undergoing a LOCA event may easily surpass the acceptance criterion of a maximum of 17% equivalent oxidation of the original cladding thickness. The oxidation studies will be followed by tests of fuel rod segments under simulated LOCA conditions (thermal ramp, flowing steam, and thermal quench) to determine the fragmentation character of the cladding and to assess whether oxidation and fragmentation behaviors are within acceptable limits. If necessary, sufficient tests will be conducted to assist the NRC in establishing new criteria for high-burnup fuel rods.

LWR Fuel Minimization

The Department of Energy has been actively planning a new program, to begin in FY 1998, to increase the burnup of LWR fuels to $\approx 100,000$ MWd/MT. The objective of this program is to minimize the amount of spent nuclear fuel that DOE must place in the Yucca Mountain repository. This past year has seen the IPS actively involved in the planning of this national program. When the program commences, our initial role will be to perform detailed characterizations of current LWR high-burnup fuel rods with the objective of understanding the fuel and cladding degradation mechanisms that now limit a fuel rod's lifetime. This information will be used in the program to develop new cladding alloys and fuel rod designs that will achieve the 100,000 MWd/MT burnup goal reliability and with assurance that all safety criteria can be met. This program is now perceived to be a seven-year effort.

In anticipation of the new program, the Section requested and received Laboratory discretionary funding in FY 1997 to evaluate the need for a capability in the AGHCF for measuring the thermal conductivity and melting point of high-burnup UO_2 fuel. Input from fuel performance analysts at ANL and other laboratories indicate the need for the thermal conductivity data and for a capability in the U.S.; none exists now. The apparatus, however, may be very expensive, and procurement of this apparatus is not a certainty.

Spent Nuclear Fuel

The Chemical Technology Division (CMT) has long studied the leaching characteristics of LWR spent fuel in the environments in which they may reside in the Yucca Mountain repository. Because of the stringent quality assurance requirements for these studies, only well-characterized fuel materials ("approved testing materials," or ATMs) can be used. Beginning in late FY 1997, the Section will take over the responsibility for characterizing ATMs for CMT and other participants in the national program. The first material to be characterized will be BWR fuel produced by the General Electric Co. that has achieved a burnup of $\approx 70,000$ MWd/MT. The characterization will examine fission-product

distributions across the fuel pellets, the nature of the pellet rim (where significant changes occur with burnup), and the nature of the fuel/cladding interface (where chemical bonding occurs and where volatile fission-products tend to collect). Knowledge of fission-product behavior during irradiation is the first step in understanding the leaching characteristics of these same fission products in a repository.

Characterization of EBR-II Stainless Steel

In 1996, the IPS and ANL's Engineering Division (ANL-W) made a proposal to the Power Reactor and Nuclear Fuels Development Corporation (PNC) of Japan to evaluate the properties of irradiated stainless steel structural materials from EBR-II, which have seen neutronic and thermal conditions similar to those seen by stainless steels in LWRs. PNC agreed to a collaboration, and a four-year contract was recently signed. Under the agreement, the microstructure and microchemistry and tensile properties of the materials will be studied at ANL with the participation of assignees from PNC.

The materials to be studied are the Type 316 stainless steel hex cans from nonfuel reflector subassemblies, which operated at temperatures of 370-390°C. These temperatures match the high end of the temperature range of interest for current or advanced LWRs. Because of its long residence time in the reactor (≈ 10 years), the steel has accumulated estimated radiation damage of 42 to 71 displacements per atom in a flux spectrum similar to that in an LWR. Thus, this material is of significant value in aiding the assessment of long-residence-time stainless steel components in LWRs.

Aluminum-Clad Spent Nuclear Fuel

The long-term storage and ultimate disposition of aluminum-clad fuel from production and domestic and foreign test reactors is the responsibility of DOE's Savannah River Site (SRS). Aluminum-clad fuels include UAl alloys and UAl_x, U₃O₈, and U₃Si₂ dispersions in an aluminum matrix. Since 1996, the IPS has assisted SRS in defining acceptance criteria for fuel that will be put into long-term storage at SRS and in assessing the potential problems that may develop either during storage or during a disposal process. The issues are the current corroded state of the cladding after years in wet storage, an acceptable cladding corrosion rate in the dry environment of the long-term storage, possible fission-product-driven fuel degradation during storage, fission-product release rates from degraded fuel, and fission-product release during possible high-temperature treatment.

In the past year, nonirradiated corrosion samples from SRS were evaluated here by optical and scanning electron microscopy and compared with similar evaluations of irradiated aluminum-clad fuels in the AGHCF inventory. Differences were noted and attributed to the likely "over-test" environment imposed on the SRS samples in the laboratory. In two long-term furnace tests, of one and four months duration, segments of irradiated aluminum-clad fuel plates were heated in air at temperatures expected in storage. The aluminum oxide layer increased somewhat in thickness, but no fission products were released, even though the edges of the fuel meat were exposed. Tests are now planned at higher temperatures for a four-month duration. Other furnace tests will investigate fission-product release when the fuel is melted, a possible step in the ultimate treatment process. This information would be used for designing the equipment to be used in the process.

Research and Test Reactor Fuel

The IPS continues to support the development of high-density, reduced-enrichment fuels for research and test reactors around the world. In the past, a U_3Si_2 dispersion was developed to satisfy the needs of most of the research and test reactors. However, the need to achieve higher flux levels has prompted the investigation of other, higher-density compounds.

The capabilities of the AGHCF, principally scanning and Auger electron microscopy, continue to be used to characterize fission-gas bubble morphology, chemical changes in the fuel, and fuel/matrix interactions that occur during irradiation. In the coming year, a series of samples irradiated in the Advanced Test Reactor in Idaho by ANL-West's Engineering Division will be examined here.

Nuclear Waste

Ceramicrete

"Ceramicrete" is a family of chemically bonded phosphate ceramics developed by the ET Ceramics Section for the low-temperature stabilization and solidification of mixed wastes and plutonium-bearing wastes. Because of the presence of radioactive nuclides in mixed waste and the hazards in handling plutonium, the Ceramics Section and the IPS have joined forces to use the nuclear facility capabilities of the IPS for conducting research on Ceramicrete with actual waste materials. To date, this collaboration has achieved the treatment of ^{99}Tc -bearing waste and has undertaken the process development for stabilizing Pu-containing ash from the incineration process at Rocky Flats. When the latter is completed during 1997, the process will be transferred to the ANL-W facilities where a near-full-scale demonstration of the process will be conducted by using actual Rocky Flats ash residues currently stored in Idaho. The IPS contribution to this work includes making Ceramicrete compacts with radioactive materials, based on the process developed in the Ceramics Section with nonradioactive surrogates, and then characterizing the product by scanning electron microscopy to evaluate microencapsulation and the formation of chemical compounds that chemically stabilize the radioactive species.

Because of the expanding number of potential uses for Ceramicrete in stabilizing radioactive wastes, IPS expects that its involvement in process development and product characterization will continue for some time.

Waste Characterization

The Idaho National Engineering and Environmental Laboratory (INEEL) is funding the IPS to perform analyses of the head-space gases in typical remotely handled transuranic waste (RHTRU) drums that are generated in the course of our work in the AGHCF. The drums contain either combustible or noncombustible waste materials and small quantities of transuranics, mainly plutonium. The generation of hydrogen by alpha-radiolysis in these drums is a concern during both shipping and long-term storage. The goal of this project is to characterize the head-space gas generation in typical, heterogeneous hot-cell waste

drums as a function of time to assess whether gas generation in such waste drums will be a potential problem. This project will begin in FY 1997 and end in FY 1998.

Other areas of potential cooperative activities with INEEL have also arisen. These include the development of nondestructive quantitative assay methods for waste drums. Methods developed at INEEL will be tested here with actual waste packages containing the variety of radioactive waste and spent nuclear fuel in the AGHCF inventory. This inventory represents a readily accessible and valuable resource for conducting such developmental activities at ANL-E.

Decontamination of Surface-Active Components

The ANL Chemistry Division has developed a very promising family of chemical decontamination agents based on diphosphonic acid and various reductants. The success of the decontamination agents was first demonstrated in the decontamination of the main coolant piping during the decommissioning of the Yankee-Rowe nuclear reactor, and they show promise for many surface decontamination needs in the DOE complex. The advantages of these agents are that they are environmentally friendly, the process temperature and pressure are low (typically 60°C and ambient pressure), and they react predominantly with surface oxides and not the base metallic material.

In cooperation with the Decontamination and Decommissioning Project of ANL's Technology Development Division, we have begun using these agents for preresearch decontamination of the AGHCF's slave manipulators, which become very contaminated (up to 2 R at contact) during use. This is part of a plan to seek ways to reduce radiation exposures to the technicians performing hands-on maintenance of the manipulators. These agents have been found to reduce the surface radiation levels of the manipulators by ~40% over that achieved with our previous methods, and there was no damage to the aluminum manipulator material.

This developmental activity is now expanding into decontamination investigations on very corroded surfaces of component parts from commercial light water reactors, including pieces of the lower head from the TMI-II reactor. The examination capabilities of the AGHCF, including scanning and Auger microscopy of the cleaned surfaces, are being gainfully used in this activity. It is expected that this project will continue for at least one year as new potential uses for these decontamination agents are identified and researched.

Fusion Reactors

Structural Materials

The IPS continues to play a lead role in ANL's fusion reactor structural materials program. The objective of the program is to develop low-activation vanadium alloys for the fusion reactor's first-wall and blanket structures. To study the effects of radiation damage on vanadium alloy materials, the IPS designed and built an irradiation experiment, ATR-A1, for the U.S./Japan collaborative program. The irradiation, in the Advanced Test Reactor in Idaho, was successfully completed in 1996, and disassembly of the irradiation capsule is

underway in the IPS hot cells. Pressurized vanadium creep specimens were developed by the IPS, the first such attempt in the U.S., and were included in the ATR-A1 experiment.

Utilizing our expertise, an IPS staff member was on hand to assist with the disassembly of the U.S./Russia Fusion-1 capsule after irradiation in the BOR-60 reactor in the Research Institute of Atomic Reactors in Russia. This collaboration was successful and may lead to future joint U.S./Russia experiments in the BOR-60 reactor.

To study the effects on vanadium alloys of environmental exposure in a tokamak, four collaborative experiments were conducted with General Atomics in their DIII-D tokamak. These experiments successfully demonstrated that impurities in the plasma chamber would not significantly degrade the tensile and fracture properties of vanadium alloys even after prolonged exposure. These data support the development of an advanced divertor for the DIII-D using vanadium alloys.

Ancillary Activities

Irradiation Behavior of BeO

The Irradiation Performance Section is performing the postirradiation examination of a group of BeO specimens that were irradiated in the Advanced Test Reactor by the Knolls Atomic Power Laboratory (KAPL). Of interest are radiation damage, as evidenced by macro- and microstructural changes and lattice parameter changes, and release and/or retention of the helium and tritium that were formed. The IPS is also involved in the data interpretation and analysis with KAPL.

Component Failure Analysis

The IPS performs failure analysis on radioactively contaminated components, or pieces, removed from service in reactors operated by Commonwealth Edison Co. (ComEd). These analyses, which consist only of direct metallurgical examinations and tests, support ComEd's in-house analytical efforts to determine the causes of component failures. The IPS provides ComEd with quick response over a range of services for radioactive materials unavailable elsewhere in the area.

Lithium-Bearing Capsule Disassembly

Through its work on the irradiation and disassembly of capsules containing lithium metal as a heat transfer medium in the Fusion Program, the IPS has developed a near unique capability in the DOE complex for disassembling Li-containing irradiation capsules for other programs. The IPS will begin soon to disassemble six capsules containing beryllium metal samples for the Karlsruhe Institute in Germany. The effort could expand to a total of 20 capsules in FY 1998. The retrieved samples will be sent to the Pacific Northwest National Laboratory for examination.

Examination Support

The Section is continuing its collaboration with the Corrosion Section in the NRC's Irradiation-Assisted Stress Corrosion Cracking Program for LWR stainless steel components. Our efforts provide the investigators with the necessary fractographic (SEM) and microchemical (Auger microprobe) data for evaluating the phenomenon.

Future Directions

The future direction of the Irradiation Performance Section continues to be in applying our expertise to in-reactor materials experiments and to characterization of irradiated materials. It is recognized, however, that these capabilities continue to face a dwindling nuclear market and that the AGHCF is an aging facility even as it becomes rare in the nuclear industry. Maintaining the Section's core competency in working with radioactive materials for both DOE and NRC materials programs is our goal. Sharing these capabilities with industry to complement their own facilities is a recognized path forward.

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Mechanics of Materials

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Members of the Mechanics of Materials Section conduct research in the areas of light water reactor (LWR) technology, (b) fusion reactor design and analysis, and (c) fabrication and analysis of nuclear fuels for the Reduced Enrichment Research and Test Reactor (RERTR).

Current work in support of LWR technology centers primarily on a major multiyear research program sponsored by the U.S. Nuclear Regulatory Commission entitled "Steam Generator Tube Integrity Program." This multidisciplinary program involves members of several sections in the Energy Technology Division and includes activities on non-destructive examination (NDE), testing and analysis of tube integrity, and the acquisition of service-degraded tubes from retired steam generators. The NDE activity is described in the research summary for the Sensors, Instrumentation and NDE Section, and the latter two activities are described here. Research related to nuclear technology has also been conducted in cooperation with ANL's Chemical Technology Division on metallic waste forms for spent nuclear fuels.

Four research activities are being conducted in the Mechanics of Materials Section in support of fusion reactor technology. These are (a) stress analysis and the development of design criteria for the International Thermonuclear Engineering Reactor (ITER), (b) analysis and modeling of plasma/first wall interactions in fusion reactors, (c) development of materials data bases for candidate fusion reactor structural materials, and (d) critical assessment of the data base and the development of models and correlations for the design analysis of Be- and Li-based tritium-breeding ceramics.

In addition, two research activities are being conducted in support of the RERTR. The first of these is fabrication of experimental low-enriched U fuel plates and irradiation targets for the production of ⁹⁹Tc. The second RERTR activity is modeling of the irradiation behavior of developmental fuel element designs for the RERTR.

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LWR Technology

Steam Generator Tube Integrity Program

Steam generator tubes account for more than 50% of the primary pressure boundary surface of PWRs and have experienced in-service corrosive and mechanical degradation of various forms since the beginning of PWR commercial operation. Environmentally induced degradation through intergranular stress corrosion cracking (IGSCC) and intergranular attack (IGA) has become a serious issue with respect to tube integrity and serviceability in recent years. This degradation commonly occurs in creviced regions at tube support plate and tube sheet locations or under sludge piles, although IGSCC has also been observed in the free span of tubes. Because of its variable and often complex morphology, this cracking can be difficult to accurately detect and size by conventional inspection techniques, and the burst pressure and leak-rate behaviors of degraded tubes are not readily predictable. In view of the changing nature of observed tube degradation, and in order to provide a more modern and flexible approach to steam generator inspection, maintenance, and repair, the NRC is developing a new performance-based rule and regulatory guide for steam generators. This research program has recently been initiated to address the new and complex forms of degradation observed in recent years and to support the new NRC rule-making activities.

The Steam Generator Tube Integrity Program is divided into four technical tasks, namely (a) Assessment of Inspection Reliability, (b) Research on Inservice Inspection Technology, (c) Research on Degradation Modes and Integrity, and (d) Tube Removals from Steam Generators. The first two of these tasks deal with nondestructive examination concerns and are being performed primarily by members of ET's Sensors, Instrumentation and NDE Section. The work being performed in these tasks is described in the research summary for that Section.

The objective of Task 3 ("Research on Degradation Modes and Integrity") is to evaluate and experimentally validate models to predict potential degradation modes, progression rates, leak/rupture behavior, failure pressures, and leak rates for steam generator tubes under normal operating and accident conditions. Existing models and predictive methodologies have often been generated on the basis of limited data and/or data and correlations in which flaw morphology and tube conditions are not necessarily representative of current-day degradation modes. The work performed under this task will update and validate these models and correlations and, as required, produce new correlations for flaw types of current interest. Specifically included is the evaluation of the continued serviceability of tubes repaired by sleeving, weld deposition, and Ni plating and the development of criteria for the qualification and acceptance of tube repair methods.

One of the first activities completed under the present task was the experimental evaluation of the integrity of steam generator tubes under hypothetical low-probability severe accident conditions, where relatively little prior experimental data were available. A high-temperature test facility for conducting burst tests on steam generator tubes was constructed, and more than 85 failure tests on unflawed and flawed (both axial and circumferential flaws) tubes were conducted. These included tests with pressure and temperature histories that have been calculated for the postulated severe accidents. Temperatures as high as 915°C (1679°F) and internal pressures to 33.1 MPa (4800 psi) were reached in these tests, as compared with normal steam generator tube operating

conditions of 300-325°C (570-620°F) and a pressure differential of 8.2-9.6 MPa (1200-1400 psi). Examples of tubes that failed under three different test conditions are shown in the figure below. Semiempirical models for predicting failure of flawed and unflawed tubes have been developed. Predictions of the models have compared fairly well with experimental observations, and the NRC has used these models to evaluate steam generator tube integrity in their analyses of severe accidents.

An experimental autoclave facility has been designed and constructed to produce SCC in steam generator tubes under accelerated conditions. These tubes will be used for subsequent NDE characterization and pressure and leak-rate testing. In the autoclave facility, six Alloy 600 steam generator tube specimens are contained in a Hastelloy C-276 autoclave and exposed at the outer surface to highly corrosive solutions (up to 60% NaOH plus CuO and Cu) at 300°C to produce accelerated SCC. The specimens are internally pressurized with high-purity water to produce a hoop stress in the wall equal to $\approx 60\%$ of their yield strength. As a simpler alternative to autoclave exposures, experimental procedures are also being developed for the production of SCC in tubes at ambient temperature and pressure by exposure to sodium tetrathionate and similar solutions.

In addition to the above facilities, a pressure and leak-rate test facility has been designed and constructed to experimentally determine the failure pressures and leak rates in flawed tubes under normal operating and design-basis accident conditions. This facility

Post-test appearance of initially unflawed steam generator tubes failed under three different sets of test conditions in simulated severe accident internal pressurization tests: (a) 228 min at constant 800°C and 12.4 Mpa, (b) ramped to 800°C and held for 33 min at 16.2 Mpa, and (c) ramped to 700°C and held for 38 min at 31.0 MPa

Schematic diagram of pressure and leak-rate test facility for the steam generator tube integrity program

(shown schematically above) will test tubes containing initially stable flaws to the point of instability by utilizing superheated water at temperatures to 343°C (650°F), pressures to 21 MPa (3000 psi), and flow rates to 760 L/min (200 gal/min). The facility utilizes a downstream back-pressure regulator valve operated under closed-loop feedback computer control to regulate the pressure differential across the tube wall and prevent nonprototypical two-phase flow on the primary side of the tube.

The objective of Task 4 ("Tube Removals from Steam Generators") is to obtain tubes and tube sections from retired steam generators for use in the other research tasks of this program. These samples of service-degraded tubes are needed for NDE characterization and analysis in the first two tasks of this program and for integrity studies and destructive examinations in the third task. An agreement has been reached with Duke Power Company of Charlotte, NC, for the removal of tube sheet samples, tube support plate samples, and individual tubes from the retired steam generators recently replaced at the McGuire Unit 1 Nuclear Station. The tube defects of specific interest in this sampling operation include circumferential cracks at the top of the tube sheet, cracks (primarily axial) at the tube support plates, and axial and circumferential cracks in the free span. These samples were removed during the summer of 1997.

Metallic Waste Forms for Spent Nuclear Fuels

An electrometallurgical treatment process has been developed by ANL to convert a various types of spent nuclear fuels into stable storage and waste forms for repository disposal. The process separates radioactive fission products from the long-lived actinides and generates a lower waste volume with significantly less long-term toxicity than that of spent nuclear fuels. The process waste forms include a mineral-based form that will contain fission products removed from an electrolyte salt, and a metal-based form that contains

metallic fission products, the fuel cladding, and process materials. Stainless steel-zirconium alloys were selected as the preferred candidate metal waste form material. ET is providing technical support to ANL's Chemical Technology Division in developing and evaluating these alloys.

Corrosion rates were measured for reference stainless steel-zirconium base waste form alloys in simulated repository groundwater. Corrosion rates of the waste form alloys were approximately the same as those for commercial-grade Types 304 and 316 stainless steel and Incoloy 825. Zirconium additions to the alloys appear to be beneficial in reducing the corrosion rates, while the noble-metal additions (Pd, Ru, Ag, Mo) appear to slightly increase corrosion rates. The Rockwell hardness was R_A 68-69 under a 60-kg load. Typical hardness values for annealed Type 316 stainless steel and hardened copper are R_A 58 and R_A 39, respectively. The uniaxial tensile fracture strength was 234-345 Mpa (34-50 ksi), which is approximately 30% higher than that of annealed oxygen-free copper, and 43% lower than that of annealed Type 316 stainless steel, although the total elongation was less than 1%. The Charpy impact fracture energy was 0.7-1.6 J/cm². This impact energy is much lower than that of commercial stainless steels, for which values range from 100 to 200 J/cm². However, the lower elongation and impact energy of the waste form alloys would not become a concern because the metal waste form itself is not a structural component. Initial characterization of the reference alloys are essentially completed, and two nominal reference alloy compositions, Type 316 stainless steel-15%Zr and Zr-8% Type 316 stainless steel, were selected on the basis of alloying temperature, amount of nonwaste addition, corrosion resistance, mechanical strength, and microstructural properties. Future work will include investigations of long-term aging effects, waste form qualification, and quality assurance.

Fusion Reactor Design and Analysis

ITER Stress Analysis and Design Criteria

The International Thermonuclear Engineering Reactor (ITER) is currently nearing the end of the Engineering Design Analysis phase, during which a detailed design of a reference shielding blanket (water-cooled Type 316 stainless steel) and a backup design of a breeding blanket (water-cooled solid breeder) are being completed by the four home teams, together with the Joint Central Team (JCT) at Garching, Germany. The ET division is extensively involved in two aspects of the design analysis effort. The first is a task on ITER structural design, whose purpose is to develop structural design criteria for ITER, and the second involves stress analysis in support of ITER blanket design.

During 1994 and 1995, three drafts of an interim structural design criteria were prepared by the European Community (EC) in collaboration with an international committee comprising members from each of the four parties and the ITER JCT team at San Diego. Since 1995 and continuing to the present, the U.S. (i.e., the ET Division) has been given the primary responsibility for developing new design rules to handle irradiation embrittlement. Draft 5 of the interim structural design criteria was released in June 1996, and a draft 6, including design rules for welded joints and bolted joints was scheduled for release in July 1997.

A preliminary analysis of the ITER back-up design of a water-cooled first wall/breeding blanket module was conducted to investigate the thermomechanical and heat transfer efficiencies of the various interfaces between the beryllium blocks, solid breeder, and first wall. An incremental iterative solution between the thermal and the stress analyses is planned for the future.

Analysis of Plasma/Material Interactions in Fusion Reactors

Loss of plasma confinement in magnetic fusion reactors and the resulting damage to plasma-facing components (PFCs) and structural materials are the most serious concerns for safe, successful, and reliable fusion reactor operation. Plasma instabilities can take various forms, such as hard disruptions, which include both thermal and current quench, edge-localized modes (ELMs), and vertical displacement events (VDEs). The intense energy deposition ($10\text{--}200\text{ MJ/m}^2$) in these events over a short period ($0.1\text{--}300\text{ ms}$) may result in severe surface and bulk effects. Surface damage includes high erosion losses due to surface vaporization, spallation, and melt-layer erosion. Bulk damage effects include large temperature increases in structural materials and at the interface between surface coatings and structural materials. These large temperature increases will cause high thermal stresses, possible melting, and material fatigue and failure. Other bulk effects of some plasma instabilities, particularly those of longer duration such as VDEs and those with deeper deposited energy (such as runaway electrons), can cause high heat flux levels at the coolant channels; this may cause burnout of these tubes and result in significant down-times for repair and maintenance. In addition to these effects, the transport and redeposition of the eroded surface materials from vaporization, melt-layer splashing, and macroscopic particle emission to various locations on plasma-facing and nearby components are of major concern for safety, plasma contamination, and successful and prolonged plasma operations following plasma instability events. A procedure to clean up the redeposited debris of the eroded materials should be as important as the procedure to repair the incurred damage on PFCs.

The initial stage of energy deposited during plasma instabilities is known to cause sudden formation of a vapor cloud above the exposed area. This vapor cloud, if well confined, may significantly reduce the net energy flux to the original disruption location, thus substantially reducing vaporization losses. Detailed physics of plasma/solid/liquid/vapor interactions in a strong and oblique magnetic field have been developed and evaluated in a comprehensive self-consistent manner. Such detailed treatment of the magnetohydrodynamics and photon radiation transport in the vapor-cloud region, for example, is very important in determining the net erosion thickness from surface vaporization.

The extent of the resulting damage to plasma-facing materials (PFMs), structural materials, and coolant channels depends mainly on the total plasma energy deposited, deposition time, and thickness and the type of armor material. During the short disruption events ($<1\text{ ms}$), the initially evaporated material may significantly reduce further PFM vaporization erosion. The stability and confinement of this vapor plasma during the disruption is therefore a very important issue in evaluating the overall lifetime of PFCs. Models to study and evaluate hydrodynamic instabilities that cause the loss of vapor plasma during the disruption have been developed. During longer plasma instabilities, however, such as VDEs ($\geq 100\text{ ms}$) or runaway electrons, no significant self-shielding is expected and

therefore serious erosion, melting, and structural damage can occur. In addition, hydrodynamic instabilities and other forces will further erode melt layers of metallic PFMs. The amount of melt-layer loss will depend on many parameters, such as duration of melt layer in the liquid phase, melt-layer thickness, and magnitude and direction of various existing forces. Longer plasma instabilities will also allow enough time for the deposited plasma energy to be conducted from the surface to the structural material and finally to the coolant channels where it can cause burnout.

Because heat loads and disruption conditions in future power reactors are not achievable in current tokamak machines, laboratory experiments are used to study and simulate disruption effects. Modeling of these experiments and their relevancy to reactor conditions during a disruption are performed. Because erosion losses of the more relevant simulation experiments to tokamak conditions are very small, data on plasma/vapor interaction during simulated disruptions have been analyzed and found to be in good agreement with model predictions. Generally, however, plasma instabilities must be avoided or sharply reduced by one to two orders of magnitude below current specifications. In addition, the effects of resulting debris from the eroded and splashed materials on plasma contamination and on subsequent reactor operations must be studied and evaluated in detail.

Data Bases for Candidate Fusion Reactor Structural Materials

Stress analysis and design criteria require well-developed data bases for the candidate structural materials. The Advanced Materials Section of DOE's Office of Fusion Energy is actively supporting mechanical property research into austenitic and ferritic/martensitic steels, V alloys, and, to a lesser extent, monolithic and composite SiC. The Mechanics of Materials Section has the lead U.S. role as interface between the design community and the materials community. This role requires assessing the data bases and presenting them in a usable, relevant form to the ITER design team, as well as to the design teams for advanced reactor concepts (e.g., Starlite). Responsibility has also been assumed for other materials (e.g., Cu and Cu alloys) that may be used in heat-sink, magnetic, and solid-breeder/Be components. Contributions to date include the writing of chapters for the ITER Material Properties Handbook on the effects of irradiation on Type 316L stainless steel and on the properties for unirradiated and irradiated vanadium alloys (e.g., V-4Cr-4Ti), and property summaries for the Starlite assessments of ferritic/martensitic steel and V alloys. In addition, the extensive tensile data bases for Type 316L stainless steel and V alloys have been critically reviewed and corrected. In the case of Type 316L stainless steel, the major correction has been in the area of uniform elongation, which required input, cooperation, and acceptance from Japanese and European colleagues. For V alloys, more than 200 force-deflection curves were reanalyzed by a consistent standardized methodology, resulting in significant revisions of both the uniform and total elongations for these alloys.

Models/Properties for Beryllium and Lithium-Based Tritium-Breeding Ceramics

This task has been expanded over the past several years from modeling tritium transport/retention in Li-bearing ceramics to critically assessing the data base and developing models/correlations to be used in design analysis for the thermal, mechanical, chemical compatibility, tritium, and He performance of Be- and Li-based ceramics. This

work has been guided by the needs of the ITER program. Development and validation of the TIARA code for the prediction of tritium inventory in, and tritiated gas species released from, Li_2O was completed and documented in June 1995. Since that time, semiempirical models have been developed and tested for analyzing the tritium transport in ternary breeder ceramics (e.g., Li_2ZrO_3 and Li_4SiO_4). Also completed and documented in November 1993 under this task was the lithium-based ceramic and Be data base, with input from the Russian Federation, the European Community, the United States, and Japan. This ongoing task is coordinated by the Mechanics of Materials Section. Funding for both of these areas has been minimal in fiscal year 1997 because of budget cuts and redirection of effort within the DOE Office of Fusion Energy. The present FY 1998 budget indicates significant increases in these areas.

Because of the successful integration of data base and design-analysis needs in the solid breeder data base work, this task has been expanded to include liquid coolants/breeders (e.g., Li and Li-Pb). Along with the modeling and data base work, design calculations are routinely performed for the ITER design team in the areas of thermal performance, compatibility, and tritium retention/release for ITER conceptual breeding-blanket designs. With the worldwide decline in funding for breeding-blanket materials R&D for 1996-97, the primary contribution to ITER has been in the design calculations that use the models and data bases developed as part of this program. Again, increases in these efforts are anticipated for FY 1998, depending on the final budget and allocation of funds within DOE.

Nuclear Fuel Development and Evaluation

Fabrication Activities in Support of the Development of Reduced Enrichment Fuel for Research and Test Reactors

The Reduced Enrichment Research and Test Reactor (RERTR) program was established in 1978 at ANL by the Department of Energy as part of the federal government's nuclear nonproliferation policy. The primary objective of the program is to develop the technology needed to use low-enrichment uranium (LEU) instead of high-enrichment uranium (HEU) in research and test reactors, and to do so without significant penalties in experiment performance, economic, or safety aspects of the reactors. Nearly all of the civilian use of HEU is in research and test reactors. The near-term RERTR program goal to eliminate the use of a significant amount of HEU in civil nuclear programs was reached after the successful development of the high-density fuel, U_3Si_2 , which has been approved by the Nuclear Regulatory Committee for use in licensed nonpower reactors with uranium loading densities of up to 4.8 g/cm^3 . This loading is sufficient to convert nearly 90% of the research reactors that use HEU of U.S. origin, and, through the efforts of the program, many research reactors have been successfully converted to LEU. The current focus of the program has been on the two areas that require most of the remaining HEU: the 10% of the research reactors that require a loading greater than 4.8 g U/cm^3 , and the production of the medical isotope ^{99}Tc .

In March 1996, DOE provided both the funding and the guidance needed by the program to develop advanced LEU fuels. The Mechanics of Materials section has provided crucial support to the RERTR program through the fabrication of the primary irradiation targets and other related experiments. The highly skilled fuel fabrication team has more than 15 years of experience in producing highly loaded powder-metallurgy fuel plates.

A complementary effort between ANL-East and ANL-West has begun to develop a state-of-the-art fabrication facility. Various fuels with uranium loadings of 4.8 to 8.0 g U/cm³ are currently being fabricated. Multiple irradiations of assemblies consisting of 64 "micro"-plates are scheduled for 1997-98. Irradiation of the first microplates was expected to begin in the Advanced Test Reactor (ATR) in Idaho during August 1997. In addition, technical support has been supplied to ANL's Materials Science Division for fundamental studies on the behavior of LEU fuel candidates under ion and/or neutron irradiation.

Significant progress has been achieved during this period on the production of ⁹⁹Tc from fission targets utilizing LEU in place of HEU. The heart of the irradiation targets is the 0.127-mm (0.005-in.)-thick U foils. Both pure U and an alloy of U have been successfully rolled into foils. Four targets were shipped to the Indonesian National Atomic Energy Agency (BATAN) facilities for irradiation in July 1996. After irradiation, the U foils showed no signs of bonding to the cladding and were successfully removed from the cladding and delivered to the chemistry dissolution hot cell facility. As a result of this experiment, refinements were made to the design and an additional eight targets were shipped to BATAN in March 1997. These targets have been irradiated and are currently undergoing postirradiation examination. Also, in support of the ongoing dissolution studies, various uranium foils, fuel plates, and metallography assistance were provided to the Chemical Technology Division.

Modeling Irradiation Behavior of Fuel Element Designs for the RERTR

A new fuel developmental program initiated this fiscal year is aimed at development of high-density, low-enriched uranium alloy fuels for the RERTR. The modeling subtask is fully integrated with the experimental program. An experimental fuel matrix has been developed, and fuel fabrication activities are currently underway. Irradiation in the ATR will begin soon.

Detailed technical documents titled "DART Model for Irradiation-Induced Swelling of Uranium Silicide Dispersion Fuel Elements Including Aluminum-Fuel Interaction," by J. Rest and G. L. Hofman (January 1997), and "On Mechanisms of Irradiation-Induced Swelling of Uranium Alloy Fuels," by J. Rest (February 1997) were prepared and transmitted to the Institute of Inorganic Materials in Moscow, Russian Federation, as part of an ongoing U.S.-Russian collaboration on developing an improved DART model for assessing the irradiation behavior of advanced alloy low-enriched dispersion fuels. Technical discussions on these documents and related issues were held during ANL-Russian meetings in Moscow.

Work is underway to describe fission-product-induced swelling of uranium-molybdenum alloy dispersion fuel elements. Existing irradiation data consist of relatively high-temperature irradiations (500-800°C) to very low burnups (<1 at.%). The uranium-molybdenum alloy fuels test capsules scheduled to be irradiated in the ATR as part of the advanced alloy test matrix are expected to see temperatures of <300°C. At these relatively low temperatures, irradiation-enhanced diffusion will be the dominant mechanism of gas-atom diffusion. On the other hand, at 800°C, thermal diffusion will be the dominant mechanism of gas-atom diffusion. Thus, at present, data are not available to validate derived

models for fission-gas-induced swelling mechanisms for advanced uranium alloys irradiated under typical research and test reactor operating conditions.

Models for both irradiation-enhanced and thermal gas-atom diffusion in uranium-molybdenum fuel were derived on the basis of assumptions stated in the following two paragraphs.

The thermal gas-atom diffusion coefficient in uranium-molybdenum alloy fuels, D_g^{UMo} , can be obtained from the gas-atom diffusion coefficient measured in pure U, D_g^U , by scaling D_g^U by the ratio of the U self-diffusion coefficient in uranium-molybdenum to the U self-diffusion coefficient in pure uranium. This approach to deriving the thermal gas-atom diffusion coefficient in uranium-molybdenum is reasonable if the mechanism of diffusion is similar in the two materials (e.g., a vacancy diffusion mechanism). The U self-diffusion coefficient in uranium-molybdenum as a function of the wt.% of molybdenum was obtained from the work of Y. Adda and J. Philibert on diffusion of uranium with some transition metals.

Irradiation-enhanced gas-atom diffusion coefficients can be extracted on the basis of studies of structural changes taking place in uranium-molybdenum alloys under the action of neutron irradiation. Again, this assumption is reasonable if the irradiation-induced gas-atom diffusion mechanism is similar to the irradiation-induced phase-mixing mechanism. In addition, if point-defect behavior in the uranium-molybdenum alloy is dominated by the presence of sinks, then the gas-atom diffusion coefficient will be independent of temperature and linearly dependent on fission rate. This dependence has been observed in other types of nuclear fuels, such as the oxides, mixed oxides, carbides, and nitrides, and is predicted by the irradiation-induced phase mixing. In addition, the value of the gas-atom diffusion coefficient obtained in this manner lies within the scatter of the measured diffusion coefficients in oxides, mixed oxides, carbides, and nitrides.

The figure on the next page shows the derived thermal and irradiation-induced gas-atom diffusion coefficients in uranium-molybdenum alloys. The irradiation-induced diffusion coefficient is shown for three values of the fission rate. As is clear from the previous figure, the crossover from thermal to irradiation-enhanced diffusion depends on fission rate as well as on temperature. For example, at a rate of 1×10^{15} fissions $\text{cm}^{-3} \text{ s}^{-1}$, thermal diffusion will dominate over irradiation-enhanced diffusion at temperatures of $>600^\circ\text{C}$.

The diffusion coefficients were implemented into the uranium-molybdenum version of the DART code, DART-UM, and the model was applied to the analysis of swelling and fission gas bubble morphology in irradiated uranium-molybdenum at relatively high temperatures (773-1073 K). The results of this analysis are shown on page 161, where DART-calculated bubble-size distributions within the grains, on the grain faces, and on the grain edges are compared with an electron micrograph of a shadowed replica of a U-14 wt.% Mo specimen that was irradiated to 0.22% burnup at 800°C . As shown, the DART-calculated distributions are in qualitative agreement with the observations. However, as seen in the previous figure, fission gas behavior at 800°C is dominated by a thermal diffusion mechanism. On the other hand, fission-gas-induced swelling in uranium-molybdenum stabilized in the gamma phase at temperatures of interest for the RERTR Program (473-573 K) is driven by

*Derived thermal and irradiation-induced gas-atom diffusion
coefficients in uranium-molybdenum alloys*

irradiation-enhanced-diffusion processes. Preliminary DART calculations for the irradiation behavior of uranium-molybdenum dispersion fuel in the ATR indicate that fuel loaded to 25% fuel volume fraction will complete the irradiation without fuel/particle interaction. However, the calculations also indicate that loadings of 50% will not survive irradiation to full burnup without fuel/particle interconnection and resultant gross swelling. Thus, it is of interest to obtain data in this temperature regime in order to validate the derived diffusivities. A literature search aimed at this objective is currently underway.

A generalized model for radiation-induced amorphization and crystallization of U_3Si and U_3Si_2 and recrystallization of UO_2 was developed by J. Rest (J. Nucl. Mater., 240 (1997) 205-214). Calculation of the structure of nuclear fuels is an important component in assessing the stability of these materials under irradiation. U_3Si and U_3Si_2 aluminum-dispersion fuels (U_3Si_2 was developed at ANL under the RERTR Program) are commonly used in research and test reactors throughout the world. New low-enriched, high-density uranium alloy research and test reactor fuels, aimed at providing more efficient proliferation resistant fuels, are currently under development at ANL. In addition, the potential extension of existing LWR fuel (UO_2) to higher burnup is contingent on issues such as irradiation-induced fuel restructuring, or fuel recrystallization, commonly referred to as the rim effect. The rim effect is irradiation-induced grain subdivision that leads to enhanced fission-gas-bubble growth and increased fuel porosity. The generalized model has been applied to ion-irradiation and in-reactor experiments on U_3Si and U_3Si_2 and provides an interpretation for the amorphization curve (the dose required to amorphize the material as a function of temperature), for the ion-radiation-induced nanoscale polycrystallization of these materials at temperatures above the critical temperature for amorphization, as well as for the role of small crystallites in retarding amorphization. This same model is utilized to provide an alternative mechanism for the evolution of recrystallization nuclei for a model of

*Electron micrograph of shadowed replica of U-14 wt.% Mo specimen
irradiated to 0.22% burnup at 800°C, and DART-calculated
bubble-size distributions for the irradiation*

irradiation-induced recrystallization of UO_2 , wherein the stored energy in the UO_2 is concentrated in a network of sinklike nuclei that diminish with dose due to interaction with radiation-produced defects. The sinklike nuclei are identified as cellular dislocation structures that evolve relatively early in the irradiation period.

This physical picture of the so-called "rim effect" proposed as the basis of a theory of recrystallization of high-burnup UO_2 nuclear fuel (J. Rest and G. L. Hofman, Fundamental

Aspects of Inert Gases in Solids, eds. S. E. Donnelly, and J. H. Evans (Plenum, New York, 1991, p. 443); and J. Rest and G. L. Hofman, J. Nucl. Mater., 210 (1994, 187-202) has been experimentally verified (K. Nogita and K. Une, Nucl. Instr. and Meth. B 91 (1994, 301). The complicated kinetics involved in the formation of a cellular dislocation network are approximated by the formation and growth of subgrains due to the interaction of shock waves produced by fission-induced damage to the UO_2 .

Support for Other Programs

Work has been performed for the Electromechanics and Superconductivity Applications Section in the area of design and analysis for flywheel containment systems. At design flywheel energies, the particles at the outer radius of a flywheel have very high velocities (about 600-700 m/s). In the event of flywheel failure, containment of the flywheel fragments is a major safety issue. Analytical models have been developed and calculations have been performed for containment safety analyses of test facilities. Also, materials and design concepts have been evaluated with regard to their potential for absorbing flywheel-fragment energy. Although the application for the current flywheel project is the conversion and storage of electrical energy for stationary utility units, an LDRD project is being pursued for developing containment concepts for the automotive industry. The Partnership for a New Generation of Vehicles has identified flywheel containment as the major issue in the area of flywheel applications for automobiles, buses, and heavy transportation vehicles.

Recent Work by the Mechanics of Materials Section

Publications

Materials for Breeding Blankets

R. F. Mattas and M. C. Billone

Presented at 7th International Conference on Fusion Reactor Materials (ICFRM-7), Obninsk, Russia, Sept. 25-29, 1996; in J. Nucl. Mater. 233-237, Part A (1996) 72-81.

Interim Fatigue Design Curves for Carbon, Low-Alloy, and Austenitic Stainless Steels in LWR Environments

S. Majumdar, O. K. Chopra, and W. J. Shack

Proceedings 20th Water Reactor Safety Information Mtg., NUREG/CP-0126, Vol. 3, pp. 223-240, March 1993.

Results of Examinations of Pressure Vessel Samples and Instrument Nozzles from the TMI-2 Lower Head

G. E. Korth (EG&G, INEL), D. R. Diercks, and L. A. Neimark

Nuclear Engineering and Design 167 (1997) 267-285.

Fracture Behavior of Advanced Ceramic Hot-Gas Filters

J. P. Singh, S. Majumdar, M. Sutaria, and W. Bielke

Proceedings 10th Ann. Conference on Fossil Energy Materials, Knoxville, TN, May 14-16, 1996, CONF-9605167, ORNL/FMP-96/1, pp. 193-204 (1996).

Effects of Thermal Aging on Fracture Toughness and Charpy-Impact Strength of Stainless Steel Pipe Welds

D. J. Gavenda, W. F. Michaud, T. M. Galvin, W. F. Burke, and O. K. Chopra

NUREG/CR-6428, ANL-95/47, May 1996.

Density Decrease in Vanadium-Base Alloys Irradiated in the Dynamic Helium Charging Experiment

H. M. Chung, T. M. Galvin, and D. L. Smith

Fusion Reactor Materials Progress Report for Period Ending Dec. 31, 1995 (DOE/ER-0313/19), pp. 83-87 (1996).

Application of a mechanistic model for radiation-induced amorphization and crystallization of uranium silicide to recrystallization of UO₂

J. Rest

Invited speaker at International Workshop on Interfacial Effects in Quantum Engineering Systems, Mito, Japan, Aug. 21-23, 1996.

Calculating Irradiation-induced Changes in Thermal Conductivity of U₃Si₂ Aluminum Dispersion Fuels

J. Rest

Presented at ET Division Review Meeting, March 12, 1996.

DART Model for Thermal Conductivity of U_3Si_2 Aluminum Dispersion Fuel

J. Rest, J. L. Snelgrove, and G. L. Hofman

Presented at 18th International Meeting on Reduced Enrichment for Research and Test Reactors, Paris, France, Sept. 18-21, 1995.

Study of In-Reactor Creep of Vanadium Alloy in the HFIR RB-12J Experiment

R. V. Strain, C. F. Konicek, and H. Tsai

Fusion Reactor Materials Progress Report for Period Ending June 30, 1996
(DOE/ER-0313/20), pp. 103-104.

Engineering Options for the U.S. Fusion Demo

M. S. Tillack (U. of California, San Diego), M. C. Billone, L. El-Guebaly (U. of Wisconsin, Madison), D. K. Sze, L. M. Waganer (McDonnell Douglas Aerospace, St. Louis), C. Wong (General Atomics, San Diego), and the ARIES Team

Proceedings 16th IEE/NPSS Symposium on Fusion Engr. (SOFE-16),
Champaign, IL, Sept. 30-Oct. 1, 1995, Vol. 2, pp. 1202-1205

Modeling and Simulation of Melt-Layer Erosion During a Plasma Disruption

A. Hassanein; V. Belan, I. Konkashbaev, L. Nikandrov, V. Safronov, and A. Zhitlukhin
(Troitsk Inst. for Innovation & Fus. Res.); V. Litunovsky (Efremov Scientific Res. Inst.)

Proceedings 12th International Conference on Plasma Surface Interactions in
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J. of Nuclear Materials.

ITER Breeding Blanket Design

Y. Gohar (ITER Joint Central Team, Garching, Germany), M. Billone, (ITER Joint
Central Team, Garching, Germany), A. Cardella (ITER Joint Central Team, Garching,
Germany), W. Danner (NET Team, Garching, Germany), K. Ioki (ITER Joint Central
Team, Garching, Germany), T. Kuroda (Japan Atomic Energy Res. Inst., Naka), D.
Lousteau (NET Team, Garching, Germany), P. Lorenzetto (NET Team, Garching,
Germany), S. Majumdar, R. Mattas, K. Mohri (ITER Joint Central Team, Garching,
Germany), R. Raffray (ITER Joint Central Team, Garching, Germany), Y. Strebkov (R&D
Inst. of Power Engr., Moscow), H. Takatsu (Japan Atomic Energy Res. Inst., Naka), and
E. Zolti (ITER Joint Central Team, Garching, Germany)

Proceedings 16th IEE/NPSS Symposium on Fusion Engr. (SOFE-16), Champaign,
IL, Sept. 30-Oct. 1, 1995, Vol. 1, pp. 410-417.

Lifetime Evaluation of Plasma-Facing Materials During a Tokamak Disruption

A. Hassanein and I. Konkashbaev (Troitsk Inst. for Innovation, Russian Federation)

Seventh International Conference on Fusion Reactor Materials (ICFRM-7),
Obninsk, Russia, Sept. 25-29, 1995; in J. Nucl. Mater. 233-237 (1996) 713-717.

Nuclear Power Plant Generic Aging Lessons Learned (GALL): Vols. 1 & 2

K. E. Kasza, D. R. Diercks, J. W. Holland, S. U. Choi, J. L. Binder, W. J. Shack, O. K.
Chopra, D. C. Ma, and A. Erdemir; J. L. Edson, L. C. Meyer, E. W. Roberts (INEL)
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Implications of Radiation-Induced Reductions in Ductility to the Design of Austenitic Stainless Steel Structures

G. E. Lucas (Univ. of CA), M. C. Billone, J. E. Pawel (ORNL), and M. L. Hamilton (PNL)
Presented at 7th International Conference on Fusion Reactor Materials, Obninsk, Russia, Sept. 25-29, 1995; in J. Nucl. Mater. 233-237, Part A (1996) 207-212.

The Application of a Mechanistic Model for Radiation-Induced Amorphization and Crystallization of U_3Si and U_3Si_2 to Recrystallization in UO_2

J. Rest

To be published in J. Nucl. Mater.

The DART Dispersion Analysis Research Tool: A Mechanistic Model for Predicting Fission-Product-Induced Swelling of Aluminum Dispersion Fuels (User's Guide for Mainframe, Workstation, and Personal Computer Applications)

J. Rest

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Steam Generator Tube Integrity Program: Semiannual Report August 1995-March 1996

D. R. Diercks, S. Bakhtiari, O. K. Chopra, K. E. Kasza, D. S. Kupperman, S. Majumdar, J. Y. Park, and W. J. Shack

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D. R. Diercks, W. J. Shack, and J. Muscara (USNRC)

Proceedings 24th Water Reactor Safety Information Mtg., NUREG-CP-1057, Vol. 2, Feb. 1997, pp. 297-312.

Predictions of Structural Integrity of Steam Generator Tubes under Normal Operating, Accident, and Severe Accident Conditions

S. Majumdar

Invited paper in Proceedings 24th Water Reactor Safety Information Mtg., Bethesda, MD, Oct. 21-23, 1996; NUREG/CP-0157, Vol. 2, pp. 389-413 (1997).

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Steam Generator Tube Integrity Program

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Thermal and Tritium Transport in Li_2O and Li_2ZrO_3

M. C. Billone
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Fracture Behavior of Advanced Ceramic Hot Gas Filters: Final Report

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Thermomechanical Analysis of the ITER Breeding Blanket

S. Majumdar, H. Gruhn, Y. Gohar (Max-Planck Institut fur Plasmaphysik), and M. Giegerich (Max-Planck Institut fur Plasmaphysik)
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S. Majumdar
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y-High-Density Low-Enriched-Uranium Fuels

G. L. Hofman, C. L. Trybus, and T. Wienczek

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A Generalized Model for Radiation-Induced Amorphization and Crystallization of U_3Si and U_3Si_2 and Recrystallization of UO_2

J. Rest

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Steam Generator Tube Integrity Program: Annual Report, August 1995-September 1996

D. R. Diercks, S. Bakhtiari, K. E. Kasza, D. S. Kupperman, S. Majumdar, J. Y. Park, and W. J. Shack

NUREG/CR-6511, ANL-97/3, Vol. 2

Effect of Helium on Tensile Properties of Vanadium Alloys

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DART Model for Irradiation-Induced Swelling of Dispersion Fuel Elements Including Aluminum-Fuel Interaction

J. Rest and G. L. Hofman

To be presented at the 1997 Winter Mtg., ANS, Albuquerque, Nov. 16-20, 1997.

Dynamic Behavior of Plasma-Facing Materials during Plasma Instabilities in Tokamak Reactors

A. Hassanein and I. Konkashbaev (Troitsk Inst. for Innovation and Fusion Research)

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Data Required to Correctly Evaluate ITER Plasma-Facing Component Lifetime during Disruptions

A. Hassanein

INVITED presentation at Troitsk Institute for Innovation and Fusion Research, Russia, June 7, 1996.

Various Damage Effects of PFC Due to Plasma Instabilities

A. Hassanein

INVITED presentation to Troitsk Inst. for Innovation and Fusion Research, Russia, Nov. 19, 1996.

Modeling and Simulation of Plasma/Material Interactions in Fusion Devices

A. Hassanein

INVITED presentation at Sandia National Laboratories, Albuquerque, Nov. 7, 1996.

Plasma/Material Interactions during Disruptions in Tokamak Devices

A. Hassanein

INVITED presentation at U. of California, San Diego, Feb. 3, 1997.

Patent

Method for Fabricating Mo Production Targets Using Low Enriched Uranium, Mo
Production Targets Comprising Low Enriched Uranium

T. C. Wiencek, J. E. Matos (EP), and G. L. Hofman (FE-AW)

Invention Report ANL-IN-93-048, 12/93. DOE File No. S-84,216. U.S. Patent No.
5,615,238 issued 03/25/97.

Tribology

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The Tribology Section conducts research and development on low-friction and wear-resistant surface coatings and treatments applied to ceramics, polymers, and high-temperature metal substrates for use under high loads and high temperatures in aggressive chemical atmospheres. These coatings are primarily intended to protect engine-component surfaces undergoing sliding and rolling contact in advanced transportation systems, including diesels, automotive gas turbines, and other new-generation engine systems being developed under the sponsorship of DOE's Office of Transportation Technologies, and by U.S. industry. The overall goal of the Tribology Section is to promote industrial competitiveness by developing new tribological technologies and by solving problems associated with friction, wear, and lubrication. The Section has established and maintained close contact with transportation-related industries to determine their critical tribological needs and to facilitate the transfer of technologies developed at ANL.

The section's research activities include efforts on the development of surface modification processes, and evaluation of tribological properties. A significant effort focuses on the assessment of the friction and wear properties of regenerator core seals for advanced, high-temperature gas turbines under development for the Partnership for a New Generation of Vehicles program. Another program concentrates on development of corrosion- and wear-resistant surface treatments to protect polymer components exposed to alternative fuels. A third program is centered on development of boric-acid-based solid lubricants for aluminum metal-forming operations. We also anticipate starting a new effort to investigate degradation of top-of-rail lubricants and their impact on the environment. Other programs are aimed at the development and tribological assessment of ultra-smooth polycrystalline diamond and amorphous diamondlike carbon (DLC) coatings for transportation, metal-forming and cutting, and textile applications. Efforts are also underway to develop DLC coatings for biomedical applications.

Coating Process Development

The coating process development projects are aimed at exploring the factors that control the tribological properties of surfaces that use surface-modifying treatments such as ion-beam-assisted deposition (IBAD), ion beam deposition (IBD), microwave and plasma-assisted chemical vapor deposition, and plasma-sputter deposition. The major emphasis is on

¹Serves two sections.

developing low-friction, highly wear-resistant surfaces that can endure extreme tribological conditions due to high loads, speeds, and temperatures. A variety of low-friction, wear-resistant coatings were developed and deposited on metals, ceramics, and polymers intended for utilization in components for low-heat-rejection diesels and other high-temperature advanced engines and engine power trains. Specific examples include the development and testing of diamondlike carbon coatings on polymers and ceramic parts under conditions of high loads, speeds and temperatures; development of smooth, nanocrystalline diamond films on ceramics for machining and sliding wear applications; development of naturally occurring lubricious-oxide layers on superalloys for gas turbine applications at temperatures to 1000°C; and development of wear-resistant boride layers on steels and other alloys. More recently, we have made significant advances in developing DLC films, produced by plasma-assisted CVD, that exhibit extremely low friction coefficients (<0.001) under dry sliding conditions.

Our coating processing research focuses on two broad deposition techniques: development of low-friction and low-wear surfaces by a variety of surface-modification methods, including physical-vapor-deposition techniques (IBAD, plasma sputtering, and thermal evaporation), and development of ultrahard, low-friction diamond, diamondlike, and other similar coatings by plasma-assisted CVD.

Physical Vapor Deposition Coatings

When the Tribology section was started 10 years ago, its primary focus was on generic, long-term research on advanced surface modification processes to improve the friction and wear performance of materials used in energy-intensive sectors such as transportation. The coating research originally focused on physical vapor deposition (PVD) processes with major emphasis on IBAD (see figure on next page). This facility consists of a large, ultrahigh-vacuum chamber with two individually controlled electron-beam evaporation sources and simultaneous or sequential ion sources that produce ions with energies from a few hundred eV up to 1500 eV. Materials that can be evaporated include Ag, Al, B, Ba, Ca, Cr, Cu, Fe, Mg, Mo, Nb, Ni, Pt, Sn, Ti, and Zn, either singly or in combination. Various types of specimen holders enable rotational motion, ensuring uniformity in the coating. Parts as large as engine pistons can be coated. In addition, the Section has a three-target sputtering apparatus for deposition of multilayer coatings comprising Si, Au, B, C, Al, Ti, Cr, Fe, Ni, Cu, Mo, Ag, W, Pt, or Al_2O_3 . An ultrahigh-vacuum apparatus for depositing Fullerene compounds as tribological coatings has recently been built, as has an apparatus for exposing test specimens to fuels and solvents.

Previous research activities using the IBAD facility included efforts to deposit adherent silver coatings on oxide, carbide, and nitride ceramics to improve the friction and wear at elevated temperatures. The results obtained were very impressive; reductions in the wear rate ranged from factors of 10 to 3500, depending on load, temperature, velocity, and substrate. Under prior efforts, we also developed and tested soft metallic coatings on ceramics and piston ring/liner assemblies under conditions that simulate actual engine operation. Tests with synthetic liquid lubricants show a synergistic effect on both wear and friction, with improvements in both properties when a liquid lubricant is used in conjunction with a silver-coated ceramic. With the simultaneous use of silver and synthetic oil, friction coefficients were reduced by factors of 3 to 10 from that with oil alone, depending on test temperature (e.g., from 0.15 to 0.05 at room temperature, and from 0.13 to 0.015 at 300°C). Furthermore, in most cases, wear was reduced to unmeasurable levels even during tests at high temperatures and loads.

*Photo and diagram of ion-beam-assisted
deposition of thin films*

Other studies performed in the IBAD chamber demonstrated that the deposition of boron, boron oxide, Re, Ce, and Ni coatings on Ni-base superalloys dramatically reduces wear and provides low coefficients of friction (<0.3) at high temperatures. This study aims to develop new surface alloys that will result in the formation of lubricious oxides through the selective and/or collective oxidation of elemental species at high temperatures. The low-friction mechanisms of these coatings are due mainly to the formation of a soft oxide layer in open air. Some oxides can form a low-melting-point eutectic that acts as a grease at high temperatures. This research effort helped establish a program with a gas turbine engine manufacturer to develop high-temperature seal materials.

The IBAD facility was also used to develop coatings that prevented absorption of fuel (and the resulting degradation of engine efficiency) by thick thermal barrier coatings (TTBCs) used in advanced diesel engines. Using IBAD processing, we found that a thin, dense metallic

coating (Ag or an Nb/Cr alloy) could be deposited on porous zirconia TTBCs, with the coating filling or covering large open pores that intersect the surface.

More recently, the IBAD chamber was used to develop processes to deposit DLC coatings using IBD technology. In this process, a thin amorphous carbon film is deposited by introducing a carbonaceous gas (e.g., methane) into the ion source. The resulting films are quite hard, and, due to their amorphous nature, are quite smooth. They have excellent tribological properties, exhibiting friction coefficients as low as 0.05 under dry sliding, and have minimal wear (to themselves and to the mating component). As discussed below, the DLC activities are now being transitioned to a plasma-assisted CVD system for improved scale-up to large manufacturing capabilities.

Currently, the IBAD system is used for more routine nontribological applications where specialty coatings with excellent adhesion are needed. Many of these applications include coatings for fuel cell and battery applications, as well as unique capacitor and high- T_c superconductor applications.

Chemical Vapor Deposition Coatings

In addition to the IBAD PVD facilities, the section also has facilities for plasma-assisted chemical vapor deposition (PACVD). One facility consists of a microwave PACVD system that was procured for R&D on polycrystalline diamond (PCD) films. While the microwave plasma deposition source allows production of diamond, DLC, and similar ultrahard coatings on ceramics and other high-temperature materials, the main thrust of the R&D on this system is production of smooth, adherent diamond films that serve as lubricious, wear-resistant coatings without causing severe wear of mating components. Initial efforts were directed at forming smooth, thick diamond films at high deposition rates of up to 1 $\mu\text{m}/\text{h}$. A multistep microwave-plasma-deposition process was recently developed to deposit relatively smooth diamond films on rough-surfaced films. Gas-phase chemistry and deposition parameters were effectively controlled in order to improve adhesion and tribological properties of diamond films deposited on pure elements (e.g., Si) and advanced ceramics (such as SiC and Si_3N_4). Preliminary evaluation of these coatings indicates that low friction coefficients (0.2-0.3) can be achieved with coatings and that wear of underlying substrates can virtually be eliminated. Unlike DLC coatings, diamond coatings appear to have much better temperature durability; tests at up to 400°C revealed no problems. Recently, the section has teamed with sections in the Chemistry Division at ANL that have developed microwave PACVD processes by using Fullerene-rich Ar gases as precursors for the formation of PCD films. The Fullerene-based films have a much finer microstructure than conventional PCD films grown from CH_4/H_2 plasmas, and consequently have better tribological properties.

The second PACVD facility is actually a plasma-sputtering deposition system that was designed as a PVD system, but is now being used in a PACVD mode (see figure below) using the RF-etch capability. This system consists of a 24-in.-diameter vacuum vessel set up for diode sputter deposition (RF-diode, RF-magnetron, or DC-magnetron) from three different target locations. This system is being utilized because PACVD can much more readily be scaled up in size to handle large quantities of parts than can IBAD or microwave-assisted CVD where the technology is not as mature. Recent R&D efforts with this system have shown that very high quality DLC films can be achieved and that the tribological properties can be effectively tuned and optimized for particular conditions by controlling the gas chemistry.

*Plasma-assisted CVD
system used to de-
posit diamondlike
carbon coatings*

Tribological Testing and Surface Characterization

The Tribology Section has developed a number of specialized benchtop systems to evaluate and characterize the friction and wear properties of materials and components under a range of operating conditions. These facilities include three high-temperature pin-on-disk tribometers, a reciprocating wear-test rig for dry and lubricated tests at temperatures up to 900°C, a pin-on-disk machine for dry and lubricated testing at room temperature, a high-resolution surface profilometer, and an adhesion-force tester. Recently, the section acquired a system for testing coatings on shaft seals at pressures up to 300 psi, and is currently fabricating a high-speed reciprocating pin-on-flat test rig for lubricated wear tests at temperatures up to 300°C and rates up to 15 Hz. Computerized control and digitized data acquisition systems are used extensively with these machines for better data management and accuracy.

A high-temperature three-pin-on-flat rig is being used extensively to evaluate the friction and wear of ceramic coatings and monolithic ceramics sliding against honeycombed ceramic structures. Temperatures up to 1000°C can be obtained, and loads in excess of 60 lb at rotational speeds in excess of 100 rpm are feasible. The system handles samples up to 6 in. in diameter and is fully instrumented to monitor friction and wear continuously during the tests, which last up to 200 h. Another pin-on-disc rig is being used extensively to evaluate the friction and wear behavior of carbon-based materials (graphites, polymers, and carbon/carbon composites) for high-temperature seal applications. This rig has temperature

capabilities of up to 650°C at rotational speeds to 2000 rpm and loads to 1000 N. Operation of the system is computer-controlled with friction and wear monitored and recorded continuously.

The third pin-on-disc rig is a room-temperature setup that operates at up to 500 rpm and loads up to 10-15 N. This system is being used for routine friction screening and lifetime testing of DLC films. A fourth pin-on-disc rig is a low-load system (10 N maximum) that can operate at up to 600°C at rotational speeds to 200 rpm. The rig is mounted in a glovebox and thus can be used to evaluate friction and wear properties under controlled environments.

The reciprocating pin-on-flat rig has been routinely used at temperatures up to 900°C and reciprocating rates of 1 Hz. The load can be varied from 1 N to greater than 3500 N if needed. Currently, the system being used to evaluate the friction and wear behavior of coatings for gear applications at elevated temperatures. A second reciprocating wear tester is currently being developed to operate at higher reciprocating rates (15 Hz) at temperatures to 300°C to evaluate friction and wear under lubricated conditions.

The shaft-seal tester is designed to evaluate the lifetime performance of prototype shaft seals (with and without coatings). This system accepts seals of a specific design and monitors the leakage of a working fluid (either nitrogen or water) at pressures up to 300 psi. Modifications are planned to incorporate frictional torque measurements.

Additional facilities to characterize the mechanical properties of surfaces include a low-load microhardness tester, coating adhesion measurements, surface profilometers, optical and scanning electron microscopy, and more recently, a high-temperature extensometer to monitor thermal bowing in laminated materials arising from differences in the thermal expansion properties.

Together, these rigs and characterization capabilities are used to evaluate the friction and wear properties of materials and components under a wide range of conditions that simulate tribological conditions in advanced systems. The characterization capabilities are used extensively to evaluate and identify the physical mechanisms that contribute to friction and wear of materials and components and subsequently to develop strategies for improving the tribological performance.

R&D Activities

The facilities described above are used in support of a number of R&D activities including efforts on development of advanced seal materials for automotive gas turbines, development of boric-acid-based solid lubricants for metal-forming operations, evaluation of low-cost polymers for alcohol-based fuels, development of DLC films, evaluation of Fullerene-based PCD films, and evaluation of top-of-rail lubricants. Highlights of these activities are presented below.

Gas Turbine Seal Activities

One of the major activities in the section involves the development of advanced regenerator core seal materials for gas turbine engines being developed in support of the Partnership for a New Generation of Vehicles (PNGV). The primary goal of the PNGV is the development of a high-efficiency (up to 80 mpg) six-passenger vehicle; a gas-turbine hybrid

power unit is one of three power units under consideration. To achieve the required thermal efficiencies, the gas turbines under consideration will not only operate at elevated temperatures ($>2500^{\circ}\text{F}$), requiring the use of advanced ceramics, but will also utilize small compact heat exchangers to recover some of the energy in the exhaust stream. One design concept calls for the use of a rotating honeycombed heat exchanger (called a regenerator) to transfer exhaust heat to incoming compressed air prior to combustion. This effort focuses on development of new seal compositions and fabrication processes to manufacture durable regenerator core seal materials that can survive elevated operating temperatures. Our research is in the development and testing of materials for the "hot" cross-arm seal, which is exposed to temperatures above 1800°F , and for peripheral rim seals exposed to temperatures above 1200°F .

Current cross-arm seal technology uses a plasma-spraying process to deposit ceramic coatings on nickel-based substrates. The baseline coatings are not expected to survive at the higher operating temperatures, nor are they expected to be sufficiently durable or cost-effective for automotive applications. The R&D performed in the Tribology section seeks to develop new coating compositions that are more durable than existing technology, as well as lower in cost. The results thus far have identified a number of plasma-spray coating compositions that exhibit lower friction coefficients than the baseline NiO/CaF_2 coatings currently in use. Some of these compositions contain Ni, which in NiO form is carcinogenic and thus highly undesirable for use as seal that will undergo some finite amount of wear. However, other seal compositions that do not contain Ni (or NiO) were identified, most notably coatings containing MgO , ZnO , SnO_2 , ZrO_2 , BaOTiO_2 , SrF_2 , and CaF_2 , which exhibited friction coefficients lower than the baseline case. This effort is now starting to focus on the development of monolithic ceramic seals in an effort to reduce manufacturing costs. The monolithic effort is centered on ZrO_2 as a baseline component with additions of selected lubricious compounds (e.g., BaOTiO_2) to reduce friction and wear.

For peripheral seals, the current technology utilizes a stabilized graphite for operation to temperatures above 900°F . However, production costs for automotive applications are too high, and questions remain about its performance at higher temperatures. Activities in this effort include assessing the friction and wear performance of a number of different carbon-based materials (including high-temperature polymers, carbon/carbon composites, and commercial brake material). Tests were performed in a high-temperature pin-on-disc rig at temperatures of 400 to 1150°F for extended periods (up to 200 h). Nearly 60 different carbon-based materials have been assessed to date, and of these, several have been identified as having tribological properties that equal or excel those of the baseline material.

A third R&D effort related to gas turbines was to assess the tribological performance of different commercial and experimental gear-coating treatments. The regenerator core is rotated by a ring-and-pinion gear arrangement that must operate at elevated temperatures in the absence of liquid lubricants. The baseline treatment, a plasma-sprayed WC coating, had adequate performance, but its cost is too high for automotive applications. Our activities in this effort focused on assessing the friction and wear behavior of a number of commercial and experimental surface treatments in a reciprocating pin-on-flat test rig to simulate the sliding contact experienced during gear movement. The coatings examined included several ion-nitriding treatments, a boron-carbide treatment, several electroless nickel treatments, and a commercial and experimental DLC treatment. The results of the tests, which lasted up to 20 h, indicated that the DLC coatings provided the best performance of the different coatings and outperformed the WC baseline treatment in terms of friction and wear. This activity also

looked at different high-temperature greases. Results of these tests identified a number of commercial high-temperature greases that outperformed the baseline greases. In tests combining greases with coatings, the results suggest that the DLC coatings with an application of a high-temperature grease performed the best.

Boric-Acid-Based Metal-Forming Lubricants

Efforts continue in examining the tribological properties of boric-acid-based compounds as solid lubricants. Initial efforts, which led to IR-100 and STLE Al Sontag Tribology awards, demonstrated that such compounds exhibit extremely low friction coefficients under humid conditions. Recent efforts have proven successful in licensing this technology for grease additives. Recently, using DOE/BES funding, we initiated an effort to develop boric-acid compounds for use as a solid lubricant in Al metal-forming operations. The emphasis of this research is basic in nature and focuses on determining the nature of the interfacial bonding between a boric acid film and an underlying Al substrate. The basis of this research is to identify the proper surface treatments that must be applied to Al metals and alloys to ensure that the boric-acid-based coatings (which exhibit extremely low dry friction coefficients) remain intact during metal-forming operations.

Corrosion and Wear Performance of Polymers

This activity is supported through a CRADA with a U.S. automotive manufacturer to address the corrosion and wear performance of a number of low-cost polymers that the company wants to incorporate into advanced fuel-handling components for alcohol-based fuels. A number of low-cost polymers have been identified and are in current use in fuel-handling components that run on conventional gasoline. However, when exposed to alcohol-based fuels (e.g., M85 and E85, or 85% methanol/15% gasoline, and 85% ethanol/15% gasoline), these polymers swell extensively and no longer function properly. The research therefore seeks to determine if coatings could be used to eliminate swelling of the baseline polymers in the presence of M85 and E85. The results (which are subject to CRADA agreements) are providing information on the swelling (corrosion) and wear performance of a number of polymers that received a range of different coating treatments, and are identifying failure/degradation mechanisms.

Diamondlike Carbon Coatings

These activities are supported under a number of separate efforts, including the gas-turbine program and several CRADAs looking at DLCs for fuel injection and other diesel components. Originally, these activities utilized the IBAD technology described above, but recently, we have started to focus on PACVD processes because they offer great potential for coating components at automotive rates and costs. Recent results using PACVD processes have demonstrated that friction coefficients as low as 0.001 can be achieved under dry sliding conditions in a dry nitrogen environment. The results further demonstrate that this low friction value is achieved at a specific CVD gas composition.

Not only does friction coefficient depend strongly on the gas composition used during deposition, but also on the test environment. Under normal lab air, a minimum friction coefficient of approximately 0.05 is achieved, while with dry N₂, friction coefficients as low as 0.001 are observed.

To date, these coatings have been applied to a number of engine components, including fuel injector plungers, turbocharger rotors, camshafts, swashplates, sliders, thrust washers, and others. Other applications are also being investigated for gas turbines, compressors, and commercial seals.

Nanocrystalline Diamond Coatings

As mentioned above, the Tribology section is collaborating with the Chemistry Division in evaluating the tribological properties of their Fullerene-based PCD coatings. These efforts were originally supported with laboratory-directed funds and are now supported with DOE CRADA funds (ER-LTR). The emphasis of these activities is on assessing the friction and wear properties of Fullerene-based PCDS in benchtop tests and more recently in seal wear test rigs. In the latter effort, PCD films are being applied to prototype seals by the Chemistry Division, and the Tribology section is evaluating the wear life of these coatings under dry N₂ and water environments.

Top-of-Rail Lubrication

This work focuses on assessing the environmental and tribological performance of a new railroad lubrication concept being proposed by an industrial partner. This concept utilizes a biodegradable lubricant applied to the top of the rail to minimize frictional losses between rail-car wheels and the rail. The lubricant additives are properly formulated and applied so that they function as a lubricant for a limited period of time. After the train passes over the lubricated sections, the lubricant degrades and no longer functions. This concept is being developed as a means to reduce energy consumption and improve track wear and rail safety. While field trials have shown that this concept works, the Tribology Section will perform laboratory studies to identify the by-products of the lubricant, the degradation mechanism(s) and how wear processes influence them, how environmental factors affect the performance and degradation process, and environmental concerns (if any).

Program Direction

The Tribology Section plans to continue its close alliance with the transportation sector. There are a number of systems where the technologies and expertise being developed in our Section can be successfully applied. As part of this effort, Section members are in close contact with DOE-OTT working on "detail" to support development of their programs. Such effort in the past has proved beneficial in establishing new programs within our Section, and we anticipate that this effort will continue in the future.

Recent Work by the Tribology Section

Publications

Physical and Tribological Properties of Diamond Films Grown in Argon-Carbon Plasmas

C. Zuiker, A. R. Krauss, D. M. Gruen, X. Pan, J. C. Li, R. Csencsits, A. Erdemir, C. Bindal, and G. R. Fenske

Thin Solid Films 270 (1995) 154-159.

High-Temperature Durability and Tribological Performance of Diamond and Diamondlike Carbon Films

A. Erdemir, G. R. Fenske, and P. Wilbur (Colorado State U.)

INVITED paper presented at NATO Advanced Research Workshop, Portimao, Algarve, Portugal, May 29-June 5, 1996; in Protective Coatings and Thin Films: Synthesis, Characterization and Applications, eds. Y. Pauleau and P. B. Barna, pp. 169-184 (1997).

Tribology of Naturally Occurring Boric Acid Films on Boron Carbide

A. Erdemir, C. Bindal, and C. Zuiker, E. Savrun (Quest Integrated, Inc.)

Presented at ICMCTF 96 Conference, San Diego, CA, April 22-26, 1996; in Surface & Coatings Technology 86-87 (1996) 507-510.

The Effects of Beam Energy and Substrate Temperature on the Tribological Properties of Hard-Carbon Films on Aluminum

R. Wei and P. J. Wilbur (Colorado State U.), A. Erdemir, and F. M. Kustas (Martin Marietta Corp., Denver)

Surface and Coatings Technology, 51 (1992) 139-145.

Louis Pasteur Remembered

R. H. Lee

Presented at I/M-95 Conf., Chicago, July 11, 1995.

Metallography at the Met Lab - the First Fifty Years

R. H. Lee

Proc. of Intl. Metallographic Soc. Mtg., Albuquerque, July 23-26, 1995; Microstructural Science, Vol. 23, pp. 243-247 (1996).

Application of a Mechanistic Model for Radiation-Induced Amorphization and Crystallization of Uranium Silicide to Recrystallization of UO_2

J. Rest

Invited paper presented at Intl. Workshop on Interfacial Effects in Quantum Engineering Systems, Mito, Japan, Aug. 21-23, 1996.

Friction and Wear Properties of Smooth Diamond Films Grown in Fullerene+Argon Plasmas

A. Erdemir, C. Bindal, G. R. Fenske, C. Zuiker, A. R. Krauss, and D. M. Gruen

Presented at 6th European Conf. on Diamond, Diamondlike, and Related Materials, Barcelona, Spain, Sept. 10-15, 1995; Diamond and Related Materials 5 (1996) 923-931.

Solubility of Hydrogen in V-4Cr-4Ti and Lithium

J.-H. Park, G. M. Dragel, R. A. Erck, D. L. Smith, and R. E. Buxbaum

Fusion Reactor Materials Progress Report for the Period Ending Dec. 31, 1995
(DOE/ER-0313/19), pp. 59-62 (1996).

Tensile Properties of V-(4-5)Cr-(4-5)Ti Alloys

H. M. Chung, L. Nowicki, D. Busch, and D. L. Smith

Fusion Reactor Materials Progress Report for Period Ending Dec. 31, 1995
(DOE/ER-0313/19), pp. 17-21 (1996).

Solubility of Hydrogen in V-4Cr-4Ti and Lithium

J.-H. Park, G. M. Dragel, R. A. Erck, and D. L. Smith; and R. E. Buxbaum (Michigan State U.)

Fusion Reactor Materials Progress Report for Period Ending Dec. 31, 1995
(DOE/ER-0313/19), pp. 59-62 (1996).

Coatings and Alternatives for SEM Microscopy

R. H. Lee

Microscope, Vol. 43, No. 4, 179-185 (1995).

Microscopic Evaluation of Synthetic Diamond Films

R. H. Lee

Microstructural Science, Vol. 22, "Metallographic Techniques and the Characterization of Composites, Stainless Steels, and Other Engineering Materials," eds. M. T. Shehata, T. R. Leduc, I. LeMay, and M. R. Louthan, Jr., pp. 17-27.

Pin-pull Adhesion Measurements of Copper Films on Ion-bombarded Alumina

R. A. Erck

Thin Solid Films 253 (1994) 362-366.

Tribological Characterization of Smooth Diamond Films Grown in Ar-C₆₀ and Ar-CH₄ Plasmas

A. Erdemir, C. Bindal, G. R. Fenske, C. Zuiker, R. Csencsits, A. R. Krauss, and D. M. Gruen
Diamond Films and Technology, Vol. 6, No. 1 (1996) 31-47.

Formation of Ultralow Friction Surface Films on Boron Carbide

A. Erdemir, C. Bindal, and G. R. Fenske

Appl. Phys. Lett. 68 (12), 18 March 1996.

Development of Lubricious Coatings for Advanced Turbine Engine Applications

A. Erdemir and G. R. Fenske, and J. H. Munson (Allison Engine Co., Indianapolis)

Proc. 29th Intl. Symp. on Automotive Technology and Automation, Florence, Italy, June 3-7, 1996, pp. 769-780.

Effect of Niobium Interlayer on High-Temperature Sliding Friction and Wear of Silver Films on Alumina

A. Erdemir and R. A. Erck

Presented at Intl. Conf. on Metallurgical Coatings and Thin Films, ICMCTF-94, San Diego, April 25-29, 1994; Tribology Letters 2 (1996), 23-36.

Ultralow Friction Behavior of Borided Steel Surfaces after Flash Annealing

C. Bindal and A. Erdemir

Appl. Phys. Lett. 68 (7), pp. 923-925 (Feb. 12, 1996).

High-Temperature Tribological Characteristics of Silver and Gold Coatings on Ceramics Prepared by Ion-Beam-Assisted Deposition

A. Erdemir, R. A. Erck, G. R. Fenske, and F. A. Nichols

Proc. China Intl. Symp. for Youth Tribologists, Lanzhou, China, Aug. 10-14, 1992, pp. 35-49.

Tribological Performance of Diamond and Diamondlike Carbon Films at Elevated Temperatures

A. Erdemir and G. R. Fenske

Presented at 1996 Ann. Mtg. of Society of Tribologists and Lubrication Engineering, Cincinnati, May 19-23, 1996; Tribology Trans., Vol. 39 (1996), 4, 787-794.

Solid/Liquid Lubrication of Ceramics at Elevated Temperatures

A. Erdemir, R. A. Erck, and G. R. Fenske, H. Hong (The Lubrizol Corp.)

Wear, Vol. 203-204 (1997), pp. 588-595.

Tribological Properties of Hard Carbon Films on Zirconia Ceramics

A. Erdemir, C. Bindal, and G. R. Fenske, P. Wilbur (Colorado State U., Fort Collins)

Presented at 1995 ASME/STLE Tribology Conf., Kissimmee, FL, Oct. 8-11, 1995; Tribology Trans., Vol. 39 (1996), 3, 735-744.

Materials and Microanalysis

R. H. Lee

Presentation for Science Careers for Women Conference, Argonne, May 3, 1996.

The Imaging Science: How a Materials Scientist Studies the Structure, Composition, and Properties of Materials

R. H. Lee

Laboratory Tour at '96 Science Careers in Search of Women Conference, Argonne, May 3, 1996.

Tribology of Smooth Diamond Films

A. Erdemir

Presentation to Energy Technology Division Review Committee, Argonne, March 11, 1996.

Tribological Characteristics of DLC Films and Duplex Plasma Nitriding/DLC Coating Treatments

E. I. Meletis (Louisiana State U., Baton Rouge), A. Erdemir, and G. R. Fenske

Surface and Coatings Technology 73 (1995) 39-45.

Energy and Wear Analysis in Lubricated Sliding Contact

A. Alp (Illinois Inst. of Technology), A. Erdemir, and S. Kumar (Illinois Inst. of Technology)

Wear 191 (1996) 261-264.

Solid/Liquid Lubrication of Ceramics at Elevated Temperatures

A. Erdemir, R. A. Erck, G. R. Fenske, and H. Hong (Lubrizol Corp.)

Presented at 11th Intl. Conf. on Wear of Materials, San Diego, April 21-24, 1997; in Wear 203-204 (1997) 588-595.

Preparation of Ultralow Friction Surface Films on Vanadium Diboride

A. Erdemir, M. Halter (Purdue Univ.), and G. R. Fenske

Presented at 11th Intl. Conf. on Wear of Materials, San Diego, April 20-23, 1997; Wear 205 (1997) 236-239.

Friction and Wear Mechanisms of Smooth Diamond Films During Sliding in Air and Dry Nitrogen

A. Erdemir, M. Halter (Purdue U.), G. R. Fenske, R. Csencsits, A. R. Krauss, and D. M. Gruen

Presented at 1997 STLE Ann. Mtg., Kansas City, MO, May 18-22, 1997.

Sliding Friction and Wear Mechanisms of Diamondlike Carbon Films on Ceramic Substrates

A. Erdemir, G. R. Fenske, and M. Halter (Purdue U.)

Paper to be presented at 1997 STLE/ASME Tribology Conf., London, Sept. 8-12, 1997.

Effect of Source Gas and Deposition Method on Friction and Wear Performance of Diamondlike Carbon Films

A. Erdemir, G. R. Fenske, J. Terry (U. of Wisconsin), and P. Wilbur (Colorado State U.)

Presented at Intl. Conf. on Metallurgical Coatings and Thin Films, San Diego, April 21-25, 1997.

Durability and Tribological Performance of Smooth Diamond Films Produced by Ar-CO₂ Microwave Plasmas and by Laser Polishing

A. Erdemir, M. Halter, G. R. Fenske, A. Krauss, D. M. Gruen, S. M. Pimenov (Russian Academy of Sciences), and V. I. Konov (Russian Academy of Sciences)

Presented at Intl. Conf. on Metallurgical Coatings and Thin Films, San Diego, April 21-25, 1997.

Formation and Self-lubricating Mechanisms of Boric Acid on Borided Steel Surfaces

A. Erdemir and C. Bindal (Sakarya U.)

Surface and Coatings Technology 76-77 (1995) 443-449.

Adhesion and Corrosion Resistance of TiN and TiC Coatings on Graphite

P. S. Maiya, R. A. Erck, and B. M. Moon (SGL Carbon Corp.)

Surface and Coatings Technology, 1997 (in press).

Characterization of Transfer Layers Forming on Surfaces Sliding Against Diamond-Like Carbon
A. Erdemir, C. Bindal (Colorado State U.), G. R. Fenske, C. Zuiker, and P. Wilbur (Colorado State U.)

Surface and Coatings Technology 86-87 (1996) 692-697.

Measurement of Hydrogen Solubility and Desorption Rate in V-4Cr-4Ti and Liquid Lithium-Calcium Alloys

J. H. Park, R. Erck, E. T. Park, S. Crossley, and F. Deleglise

Fusion Reactor Materials Semiannual Progress Report for the Period Ending Dec. 31, 1996 (DOE/ER-0313/21), pp. 45-51 (1997).

An Investigation of the Relationship between Graphitization and Frictional Behavior of DLC Coatings

Y. Liu (Louisiana State U.), A. Erdemir, and E. I. Meletis

Presented at ICMCTF '96 Conf., San Diego, April 22-26, 1996; Surface and Coatings Technology 86-87 (1996), 564-568.

Pitting of 3003 Aluminum

D. G. Ryding, D. Allen, R. J. Dortwegt, and R. H. Lee

Microstructural Science, Vol. 24, ASM Intl. (1996).

Influence of Environmental Parameters on the Frictional Behavior of DLC Coatings

Y. Liu (Louisiana State U.), A. Erdemir, and E. I. Meletis

Presented at Intl. Conf. on Metallurgical Coatings and Thin Films (ICMCTF) '97, San Diego, April 21-25, 1997.

A Study of the Wear Mechanism of Diamond-like Carbon Films

Y. Liu (Louisiana State U.), A. Erdemir, and E. I. Meletis (Louisiana State U.)

Presented at ICMCTF '96 Conf., San Diego, April 22-26, 1996; Surface and Coatings Technology 82 (1996) 48-56.

Appendix A: Regular ET Employees

Administration

Staff R. B. Poeppel, Division Director
 W. J. Shack, Associate Division Director
 R. A. Valentin, Associate Division Director
 A. C. Smith, Building Manager, 212
 P. E. Domagala, Computer Support
 R. N. Lanham, Building Manager, 300 Area
 B. H. Malak, Financial Administration
 C. A. Malefyt, Communication Coordinator
 R. R. Fabian, QA Representative
 D. L. Telfer, Staff Assistant

Salaried B. K. Baudino
 J. L. Toenies

Hourly E. A. Moravek^{TRP}

Engineering Technology

	Electromechanics and Superconductivity Applications	Analytical Thermal- Hydraulics	Thermal, Mechanical, and Chemical Processes
<i>Staff</i>	J. R. Hull Y. S. Cha T. M. Mulcahy R. C. Niemann L. R. Turner K. L. Uherka	R. A. Valentin (acting manager) F. C. Chang ⁺⁺ T. H. Chien H. M. Domanus R. C. Schmitt	M. W. Wambsganss S. U. Choi K. E. Kasza
<i>Technicians</i>	D. J. Evans A. Wantroba		R. K. Smith
<i>Secretary</i>	J. A. Stephens*	C. Vargas	J. A. Stephens*
	Sensors, Instrumentation, and NDE	Transportation of Hazardous Materials	
<i>Staff</i>	A. C. Raptis S. Bakhtiari H.-T. Chien S. L. Dieckman W. A. Ellingson N. Gopalsami J. A. Jendrzeczyk E. R. Koehl D. S. Kupperman W. P. Lawrence S.-H. Sheen J. G. Sun	Y. Y. Liu R. R. Fabian J. J. Oras, Jr. D. T. Raske ⁺⁺ V. L. Shah P. Turula	
<i>Salaried</i>	R. N. Lanham		
<i>Technicians</i>	J. W. Lucas		
<i>Secretary</i>	S. A. Moll	J. L. Fisher	

Materials Technology

	Ceramics	Corrosion	Mechanics of Materials
<i>Staff</i>	U. Balachandran S. E. Dorris B. L. Fisher ⁺⁺ K. C. Goretta M. T. Lanagan B. Ma ^{**} P. S. Maiya J. J. Picciolo J. L. Routbort D. Singh J. P. Singh A. Wagh	T. F. Kassner O. K. Chopra H. M. Chung A. B. Hull K. Natesan J.-H. Park W. K. Soppet	D. R. Diercks M. C. Billone A. M. Hassanein S. Majumdar J. Y. Park J. Rest T. C. Wiencek
<i>Salaried</i>			C. F. Konicek J. E. Franklin
<i>Technicians</i>	J. E. Emerson R. L. McDaniel	R. W. Clark T. M. Galvin L. J. Nowicki D. R. Perkins D. L. Rink F. E. Soppet, Sr. ⁺⁺ J. C. Tezak ⁺⁺	L. Knoblich G. J. Talaber ⁺⁺ J. A. Zic ⁺⁺
<i>Secretary</i>	S. M. Hagamann*	S. M. Hagamann*	D. K. Moores*
	Tribology	Irradiation Performance	
<i>Staff</i>	G. R. Fenske D. E. Busch R. A. Erck A. Erdemir	L. A. Neimark A. B. Cohen R. V. Strain H. C. Tsai	
<i>Salaried</i>		W. Kettman	
<i>Technicians</i>	R. H. Lee	L. F. Essenmacher D. O. Pushis	
<i>Secretary</i>	D. K. Moores*	P. K. Frankovich	

*Assigned to more than one section. **Postdoctoral appointment. +Part-time. ++Term appointment.
TRP = Temporary Resource Pool.

Appendix B: Honors, Awards, and Professional Activities

Honors and Awards

S. Bakhtiari, 1996 Exceptional Performance Award, Recipient of Argonne's Exceptional Performance Award, 1996.

U. Balachandran, Recipient of the University of Chicago Distinguished Performance Award, July 25, 1996.

U. Balachandran, Certificate of recognition and appreciation for outstanding service as organizer for symposium titled High Temperature Superconductors--Synthesis, Processing and Large Scale Applications, Anaheim, CA, Feb. 4-8, 1996.

U. Balachandran, Recipient of plaque in recognition and appreciation for exemplary service to TMS Electronic, Magnetic and Photonic Materials Division (EMPMD) as Superconducting Materials Chair.

U. Balachandran, Certificate in recognition as Symp. Organizer for "Impact of Recent Advances in Synthesis and Processing of Ceramic Superconductors" Symp., 99th Annual Meeting, American Ceramic Society, Cincinnati, May 4-7, 1997.

U. Balachandran, Certificate of recognition and appreciation for outstanding service as organizer for Symposium on High-Temperature Superconductors, TMS Annual Meeting, Orlando, Feb. 9-13, 1997.

U. Balachandran, J. T. Dusek, P. S. Maiya, and R. L. Mieville, Recipient of Laboratory Director's Award for FY 1996.

H. M. Domanus and R. C. Schmitt, Deployment of the Casting Process Simulator (CaPS) Software, Recipients of Federal Laboratory Consortium Award of Merit for Excellence in Technology Transfer for 1997, April 15, 1997.

A. Erdemir, Certificate of Appreciation for contribution of time, dedicated effort and experience in the planning & carrying out of an excellent Ann. Mtg. of Soc. of Tribologists and Lubrication Engineers (STLE), Kansas City, MO, May 18-22, 1997.

A. Erdemir, Certificate of Appreciation for Excellent Education Program in Cincinnati, OH, May 19-23, 1996, presented by the Society of Tribologists and Lubrication Engineers.

S. Gopalsami, S. Bakhtiari, and A. C. Raptis, Millimeter Wave Fabric Inspection System, Recipients of Pacesetter Award, April 18, 1997.

N. Gopalsami, S. Bakhtiari, A. C. Raptis, and K. Razazian, Winners of 1996 R&D 100 Award, sponsored by R&D Magazine for entry entitled "Millimeter Wave Fabric Inspection System," June 1996.

J. R. Hull, Named One of Industry Week Magazine's "R&D Stars to Watch," 1996.

R. H. Lee, Second Place and Honorable Mention for Artistic Microscopy in IMS's 1996 Metallographic Contest.

R. H. Lee, Microanalysis of Murals, Third Place Winner in 1996 Ceramographic Exhibit, ACerS 98th Annual Meeting, Indianapolis, April 14-17, 1996.

R. H. Lee, Photomicrograph award, Second Place, Class 8 Artistic B&W, HT-9 eutectic photographed in SEM. IMS/ASM 1996 contest, Pittsburgh, July 22-24, 1996.

R. H. Lee, Photomicrograph award, Honorable Mention, Class 7 Artistic Color, Fullerene crystals precipitated from solution. IMS/ASM 1996 contest, Pittsburgh, July 22-24, 1996.

R. H. Lee, Photomicrograph award, Third Place, Microanalysis class, American Ceramic Society, Indianapolis, April 1996.

R. H. Lee, Synthetic Diamond Crystal Formation on Silicon, Photomicrograph selected for 1997 Buehler Microstructural Calendar.

K. Natesan, Elected Fellow of National Association of Corrosion Engineers (NACE), Dec. 1996.

R. B. Poeppel, J. E. Emerson, S. A. Johnson, and U. Balachandran, An Efficient New Process to Produce Commercial Quantities of Pure High-Temperature Superconductors, Recipients of Federal Laboratory Consortium Award for Excellence in Technology Transfer for 1996, May 7, 1996.

B. Shelton, Pollution Prevention Appreciation Award for Participation in "ANL-E WMin/PP Initiatives in Restoration of a Hot Foundry/Machine Shop," July 1996.

J. P. Singh, Awarded certificate of appreciation for serving as Program Chair for Engineering Ceramics Division of American Ceramic Society, 1996.

J. P. Singh, Recipient of Award for Recognition in Argonne's Exceptional Performance Program, 1996.

Tjuan Smith, Recipient of Student Research Award, DOE, Baton Rouge, LA, March 1997.

H. C. Tsai, H. M. Chung, A. Hins, R. V. Strain, and L. Nowicki, Pacesetter Award for extraordinary effort in design, fabrication, and assembly of an important fusion materials experiment irradiated in EBR-II, Aug. 18, 1995.

A. S. Wagh and D. Singh, Winners of 1996 R&D 100 Award, for entry titled "CERAMICRETE Binder," June 25, 1996.

A. S. Wagh and D. Singh, CERAMICRETE Binder, Recipients of Pacesetter Award, April 18, 1997.

Other Recognition

R. C. Niemann, Registered Professional Engineer, State of Illinois.

R. B. Poeppel, Registered Professional Engineer, State of Illinois.

Professional Society Activities

S. Ahuja, Member, American Ceramic Society.

S. Ahuja, Member, American Foundrymen's Society Design & Process Modeling Committee.

S. Ahuja, Reviewer of paper, American Ceramic Society

S. Ahuja, Reviewer of paper for Metallurgical and Materials Transactions.

S. Ahuja, Reviewer of paper for Experiments in Fluids.

S. Ahuja, Reviewer of paper for AFS Transactions.

U. Balachandran, Organizer of symposium titled High-Temperature Superconductors--Synthesis, Processing and Large Scale Applications, Anaheim, CA, Feb. 4-8, 1996, during Annual Meeting.

U. Balachandran, Member, Steering Committee of American Ceramic Society, 98th Annual Meeting and Exposition, Indianapolis, April 14-17, 1996.

U. Balachandran, Member, Electronics Division Business Meeting of American Ceramic Society, 98th Annual Meeting and Exposition, Indianapolis, April 14-17, 1996.

U. Balachandran, Member, Program Advisory Committee of American Ceramic Society, 98th Annual Meeting and Exposition, Indianapolis, April 14-17, 1996.

U. Balachandran, Session Chair, "Superconductor I" of American Ceramic Society, 98th Annual Meeting and Exposition, Indianapolis, April 14-17, 1996.

U. Balachandran, Co-Organizer, Symp. on High Temperature Superconductors, TMS Annual Meeting, Orlando, Feb. 9-13, 1997.

U. Balachandran, Co-Organizer, Symposium on High-Temperature Superconductors, TMS Ann. Meeting, San Antonio, Feb. 15-19, 1998.

U. Balachandran, Session Chair, American Ceramic Society Meeting, Cincinnati, May 4-7, 1997.

U. Balachandran, Session Chair, TMS Annual Meeting, Orlando, Feb. 9-13, 1997.

F. C. Chang, Session Developer and Session Chairman of Fluid-Structure Interactions Committee of 1995 ASME Pressure Vessels and Piping Conference, Honolulu, July 23-27, 1995.

F. C. Chang, Member, American Society of Mechanical Engineers, 1995 to present.

F.C. Chang, Editor, Conference Proceedings "Fluid-Structure Interaction - 1996, PVP Vol. 337, 1996 ASME Pressure Vessel & Piping Conference, Montreal, July 21-26, 1996.

S. S. Chen, Member, Materials and Structures Technical Group Operating Board of ASME, 1996-1998.

S. S. Chen, Fellow of American Society of Mechanical Engineers, 1984.

S. S. Chen, Division Chair (1995-96) and Executive Committee member (1990-1996) of PVPD, ASME.

W. A. Ellingson, Fellow of American Society of Mechanical Engineers, 1970 to present.

W. A. Ellingson, Member, Joint Army, Navy, NASA, Air Force (JANNAF) Subcommittee on Nondestructive Evaluation, 1990 to present.

W. A. Ellingson, Senior Member, Rapid Prototyping Association of Society of Manufacturing Engineers.

W. A. Ellingson, Fellow of American Ceramic Society Society, Dec. 1993.

W. A. Ellingson, Chairman, Inspection Requirements, Task Group on Graphite and Ceramic Pressure Equipment (ASME Boiler & Pressure Vessel Code Committee)

W. A. Ellingson, Chairman, Nondestructive Testing & Evaluation, "Composite Materials," U.S. Dept. of Defense MIL Handbook 17.

W. A. Ellingson, INVITED Member, ASME B89 Working Group 4.23 on X-ray Computed Tomographic Imaging Metrology, Feb. 1997.

A. Erdemir, Member, STLE Annual Meeting Program Committee, 1992 to present.

A. Erdemir, Member Society of Tribologists and Lubrication Engineers, 1988 to present.

A. Erdemir, Member, American Society for Metals and Materials-International, 1983 to present.

A. Erdemir, Member, American Society of Mechanical Engineers, 1992 to present.

A. Erdemir, Member, Sigma Xi Honor Society, 1988 to present.

A. Erdemir, Chairman, Solid Lubricants Technical Committee, Society of Tribologists and Lubrication Engineers, 1995-1996.

A. Erdemir, Associate Member, Research Committee on Tribology, American Society of Mechanical Engineering, 1995 to present.

A. Erdemir, Member, Organizing Comm. of International Conference on Metallurgical Coatings and Thin Films, 1992 to present.

A. Erdemir, Member, The Minerals, Metals, and Materials Society, 1983 to present.

A. Erdemir, Member, Amer. Vacuum Society, 1985 to present.

A. Erdemir, Committee Chairman, Solid Lubricants, Society of Tribologists and Lubrication Engineers, 1995-1996.

A. Erdemir, Session Co-Chair, Friction and Wear of Ceramics and Glasses II, 11th International Conference on Wear of Materials, San Diego, April 20-23, 1997.

A. Erdemir, Member, Executive Program Comm., Symp. E, Tribological Coatings/Surface Modifications, 1997 International Conference on Metallurgical Coatings and Thin Films, San Diego, April 21-25, 1997.

A. M. Hassanein, Chair of PSI and PFMs session at U.S.-Japan Workshop on High Heat Flux Components and Plasma Surface Interactions for Next Fusion Devices, Colorado Springs, CO, Jan. 9-11, 1996.

J. R. Hull, Fellow of American Society of Mechanical Engineers.

D. S. Kupperman, Member, American Physical Society, 1970 to present.

D. S. Kupperman, Member, American Society of Mechanical Engineers, 1975 to present.

D. S. Kupperman, Fellow of American Society for Nondestructive Testing.

R. H. Lee, President, State Microscopical Society of Illinois (SMSI), 1996 to present.

R. H. Lee, President, Argonne Toastmasters Club, 1994-1996.

K. Natesan, Member, International Advisory Committee of Latin American Congress on Corrosion, Cancun, Mexico, Aug. 30-Sept. 3, 1998.

R. C. Niemann, Member, Cryogenic Society of America, Vice Chairman of Midwest Chapter.

R. C. Niemann, Fellow of American Society of Mechanical Engineers.

R. B. Poeppel, Member, American Ceramic Society, 1982 to present.

R. B. Poeppel, Member, Sigma Xi.

R. B. Poeppel, Member, National Institute of Ceramic Engineers (NICE), 1983 to present.

J. L. Routbort, Fellow of American Ceramic Society, 1989.

J. P. Singh, Chair-Elect, Engineering Ceramics Division Officers, 21st Annual Cocoa Beach Conference & Exposition on Composites, Advanced Ceramics, Materials and Structures, Cocoa Beach, FL, Jan. 12-16, 1997.

J. P. Singh, Organizer of Ceramic Matrix Composite Symposium at 1996 Annual Meeting of American Ceramic Society, Indianapolis, April 14-17, 1996.

J. P. Singh, Program Chair, 21st Annual Cocoa Beach Conf. & Exposition on Composites, Advanced Ceramics, Materials and Structures, Cocoa Beach, FL, Jan. 12-16, 1997.

L. R. Turner, Senior Member, IEEE.

L. R. Turner, Member, American Physical Society's Publicity Committee of the Forum on Physics and Society, 1996 to present.

P. Turula, Member, American Society of Mechanical Engineers, Boiler Code Section III, Subcommittee on Nuclear Packaging (NUPACK) and working group on design for Nuclear Packaging, 1990 to present.

P. Turula, Member, INMM/ANSI-N14 Standards Committee for Packaging and Transportation of Radioactive Materials, 1987 to present.

A. S. Wagh, Session Chair, International Symposium on Waste Management in Ceramic and Nuclear Industry, American Ceramic Society Annual Meeting, Cincinnati, May 4-7, 1997.

M. W. Wambsganss, Fellow of American Society of Mechanical Engineers, 1994.

Other Committee Participation

U. Balachandran, Examiner of Ph.D. thesis of candidate at U. of Woolongong, Australia.

U. Balachandran, Novel Synthesis Route for High Surface Area Metal Oxide Pillared Montmorillonite, Invited member of Board of Adjudicators, U. of Kerala, India, to evaluate Ph.D. thesis.

U. Balachandran, Board of Directors - EIS, Invited to serve on Board of Directors for Educational Institute for Superconductivity (EIS).

U. Balachandran, Member of Ph.D. Thesis Examination Committee, Illinois Inst. of Technology.

U. Balachandran, Assessor, Research Fellowship Scheme Application, Research Training and Careers Committee, Australian Research Council (ARC), Canberra.

U. Balachandran, Assessor, Large Research Grant Application, Australian Research Council (ARC), Research Grants Committee, Canberra.

U. Balachandran, Ph.D. Committee at Illinois Inst. of Tech., Chicago, June 28, 1996; Oct. 17, 1996.

U. Balachandran, Ph.D. Committee at Northwestern U., Evanston, Oct. 2, 1996.

U. Balachandran, M.S. Thesis Examination Committee at Illinois Inst. of Tech., Chicago, Nov. 26, 1996.

U. Balachandran, Ph.D. thesis examiner, U. of Wollongong, Australia, Feb. 13, 1997.

U. Balachandran, Ph.D. Committee at Northwestern U., April 23, 1997.

U. Balachandran, Ph.D. Committee at U. of Missouri-Rolla, Oct. 24, 1996.

U. Balachandran, Ph.D. Committee at Northwestern U., Oct. 31, 1996.

U. Balachandran, Member, Program Committee, International Cryogenic Materials Conference (ICMC), Portland, OR, July 28-Aug. 1, 1997.

U. Balachandran, Chair, Technical Session, International Cryogenic Materials Conference (ICMC), Portland, OR, July 28-Aug. 1, 1997.

U. Balachandran, Member, Technical Advisory Board for Third Pacific Rim International Conference on Advanced Matls. and Processing (PRICM), Honolulu, July 12-16, 1998.

Y. S. Cha, Member, Safety Committee of 300 Area.

F. C. Chang, Technical Program Representative of Fluid-Structure Interactions Comm. of 1996 ASME Pressure Vessels and Piping Conference, Montreal, July 21-26, 1996.

F. C. Chang, Seminar Chairman of ET Division, Argonne National Laboratory.

F.C. Chang, Point of contact for projects related to metal casting and electromagnetic casting in DOE Industrial Projects Locator for DOE's Office of Industrial Technologies (OIT).

F. C. Chang, Appointed Technical Program Rep. of PVP Fluid-Structure Interaction Committee of 1997 ASME International Mechanical Engr. Congress and Exposition (Dallas), Nov. 16-21, 1997.

F. C. Chang, Technical Program Rep. of Fluid-Structure Interactions Committee of 1996 ASME PVP Conference, July 21-26, 1996.

S. U. Choi, Member, International Energy Agency District Heating and Cooling Advanced Fluids Experts Group (1987-present).

S. U. Choi, Member, Fluid Applications and Systems Technical Committee, ASME, July 1995 to present.

S. U. Choi, Co-Chair of Energy Performance Working Group of the Pulp and Paper Comm., 1995 to present.

H. M. Chung, Member DOE Fusion Reactor Materials Vanadium Task Group, 1993 to present.

H. M. Chung, Session Chairman, International Conference on Material Sci. and Techn. for 21st Century, Dec. 1995.

H. M. Domanus, Member, Argonne Manufacturing Working Group.

H. M. Domanus, Member, Argonne Energy and Environmental Technology Working Group.

H. M. Domanus, ANL Representative for Metal Casting Industry of the Future.

S. E. Dorris, Member of Chemical Industry Working Group, charged with preparing document conveying Argonne's chemical and materials expertise to chemical industry at large.

W. A. Ellingson, Member, 13th Congressional District of Illinois, Science and Technology Advisory Committee, 1986 to present.

W. A. Ellingson, Charter Member, Rapid Prototyping Association of Soc. of Manufacturing Engineers, 1993 to present.

W. A. Ellingson, Member, National Committee, ASME - B89 Computed Tomography Measuring Machine Member List, 1997.

W. A. Ellingson, Ph.D. Thesis Committee at U. of Texas at Austin, Jan. 24, 1997.

W. A. Ellingson, M.S. Thesis Committee at Northwestern U., June 1997.

W. A. Ellingson, M.S. Thesis Committee at Florida State U., March 1997.

R. A. Erck, Secretary, Ceramics and Composites Section, Soc. of Tribologists and Lubrication Engineers, May 1997 to present.

R. A. Erck, Seminar Chairman, Energy Technology Division, 1995 to present.

A. Erdemir, U.S. National Coordinator for VAMAS-TWA-I (Versaille Project on Advanced Materials and Standards)-Wear Test Methods Technical Working Area-I, June 1992 to 1996.

A. Erdemir, Member, Advisory Committee of the Center for Advanced Tribology at Western Michigan U., 1994-1997.

A. Erdemir, Member, International Scientific Comm., 5th International Conference on Plasma Surface Engineering, Garmisch-Partenkirchen, Germany, 1996.

G. R. Fenske, Member, Transportation Working Group Subcommittee, June 1994 to present.

G. R. Fenske, Member, Argonne Materials Coordinating Center, June 1996 to present.

G. R. Fenske, Member, PNGV Gas Turbine Technical Team, 1994 to present.

G. R. Fenske, ANL Lab representative to C4 USAMP, April 1997 to present.

K. C. Goretta, External examiner for M.S. thesis of Keith L. Duncan, U. of West Indies, Kingston, Jamaica, May 1996.

K. C. Goretta, Member, Ph.D. Qualifier Committee, Florida State U., Tallahassee, March 17, 1997.

K. C. Goretta, Chair and Manuscript Review Coordinator, Poster Session, Applied Superconductivity Conference, Pittsburgh, Aug. 25-30, 1996.

K. C. Goretta, Science Fair Judge, Avery Coonley Grade School, Downers Grove, IL, Jan. 23, 1997.

K. C. Goretta, Chair and Paper Review Coordinator, Oral Session, International Cryogenic Matls. Conference (ICMC), Portland, OR, April-Aug. 1997.

A. M. Hassanein, Appointed U.S. task leader for International Thermonuclear Experimental Reactor (ITER) disruption efforts (requested by Textor [Germany], JET [England], JAERI [Japan], and TRINITI and Efremov, [Russia]), 1994 to present.

A. M. Hassanein, Advisor to International Atomic Energy Agency (IAEA) on modeling and data needs in plasma-surface interactions area, 1993-1996.

A. Hassanein, Chairman, Plasma-Surface Interaction Oral Session, 4th International Symposium on Fusion Technology, Tokyo, April 1997.

D. S. Kupperman, Member, ASME Task Group on Preparation of Code for Digitization of Industrial Radiographs.

D. S. Kupperman, Member International Advisory Comm. of 14th World Conference on Nondestructive Testing (1996).

D. S. Kupperman, Member, Technical Advisory Committee for the Strategic Environmental Research and Development Program (SERDP) on Passive Acoustic Emission for Detection and Location of Leaks in Underground Pipelines.

Y. Y. Liu, Leader, Working Group on Frequently Asked Questions in SARP Review, Packaging Safety Review Steering Comm., DOE Office of Facility Safety Analysis.

Y. Y. Liu, ET Division Representative to Argonne Risk Working Group.

- B. H. Malak, Charter member of FACET, 1986 to present.
- C. A. Malefyt, Technology Transfer Representative, ET Division, 1990 to present.
- C. A. Malefyt, ET Coordinator, ES&H Training (1991 to present).
- C. A. Malefyt, Authorized Derivative Classifier, 1993 to present.
- C. A. Malefyt, Alternate Records Coordinator, ET Division, 1992 to present.
- C. A. Malefyt, Coordinator for ET Division, ANL 50th Anniversary Open House, 1996.
- L. A. Neimark, Member, ANL ALARA Committee, 1991 to present.
- R. C. Niemann, Member, Argonne APS Liquid Nitrogen Distribution and Vent System Review Panel, Jan. 1997.
- R. C. Niemann, Member, Argonne Materials Coordinating Committee (HTS Materials), ongoing.
- R. B. Poeppel, Member, ANL Environment, Safety, and Health Advisory Committee (ESHAC), 1993 to present.
- R. B. Poeppel, Member, ANL Management Team for Negotiation of Technician Union Contract.
- J. L. Routbort, Member, Scientific Advisory Board, Ceramica Acta, 1989 to present.
- W. J. Shack, Member of U.S. Nuclear Regulatory Commission Advisory Committee on Reactor Safeguards, 1993 to present.
- D. Singh, Member, ASME Mixed Waste Committee on Waste Minimization and Processing, 1994 to present.
- D. Singh, Designated single point of contact for Energy Technology Division for the ANL-E Remedial Actions Project (ERAP), Feb. 1997.
- L. R. Turner, Wrote bylaws for International COMPUMAG Society. Adopted at ICS Board Mtg., Okayama, Japan, March 1996.
- P. Turula, Alternate member, ANL's Risk Working Group Steering Committee, 1995 to present.
- P. Turula, Co-chairman, ANL Hazardous and Radioactive Material Transportation coordinating council, 1995 to present.

Invited Teaching and Research at Other Institutions

S. Ahuja, Visiting Scholar, U. of Alabama, Casting Process Simulator (CaPS) software instruction, May 1993.

A. Hassanein, Associate Membership, Graduate Faculty, Department of Nuclear Engineering, North Carolina State U, 1997.

A. Hassanein, Adjunct Associate Professor, Department of Nuclear Engineering, College of Engineering, North Carolina State U, 1996-1997.

J. L. Routbort, Adjunct Professor, Department of Materials Science & Engineering, Illinois Institute of Technology, 1986 to present.

J. L. Routbort, Adjunct Professor, Department of Materials Science & Engineering, North Carolina State U, 1984-1997.

Invited Conference Participation

U. Balachandran, Co-organizer of Symposium "High Temperature Superconductors" at 1997 TMS Annual Meeting, Orlando, FL, Feb. 9-13, 1997.

U. Balachandran, Co-organizer of Symposium of HTSC Materials Processing '97, at THERMEC '97, International Conference on Materials Processing at High Temperatures, Wollongong, Australia, July 7-11, 1997.

U. Balachandran, Chairman for Session F: HTS Processing, 10th Annual HTS Workshop on Physics, Materials and Applications, Doubletree Hotel at Allen Center, Houston, March 12-16, 1996.

U. Balachandran, Session Co-chair, MVA, Bi-2223 Wires and Tapes II, 1996 Applied Superconductivity Conference, Pittsburgh, Aug. 25-30, 1996.

U. Balachandran, Co-organizer of "Impact of Recent Advances in Synthesis and Processing of Ceramic Superconductors" Symposium, 99th Annual Meeting, American Ceramic Society, Cincinnati, May 4-7, 1997.

H. M. Chung, Session Chairman, 2nd Workshop of International Energy Agency Working Group on Vanadium Alloy Development for Fusion, Petten, The Netherlands, May 20-22, 1996.

H. M. Chung, Paper selection and review committee, 12th International Symposium on Zirconium in Nuclear Industry, Toronto, June 15-18, 1998.

S. E. Dorris, Invited to present research results at Wire Development Group Meeting, Madison, WI, May 21, 1997.

W. A. Ellingson, Session Chairman, First U.S.-Japan Symposium on Advances in NDT, Kahuku, HI, June 24-28, 1996.

A. Erdemir, Symposium Chairman, Tribological Coatings and Surface Modifications, International Conference on Metallurgical Coatings and Thin Films, starting 1996.

A. Erdemir, Symposium Chairman, Tribological Coatings/Surface Modifications, International Conference on Metallurgical Coatings and Thin Films, San Diego, April 21-25, 1997.

A. Erdemir, Member, International Scientific Committee, 5th International Conference on Plasma Surface Engineering, Garmisch-Partenkirchen, Germany, Sept. 9-13, 1996.

A. Erdemir, Member, Advisory Board, Chamber of Metallurgical Engineers (CME), 9th International Metallurgy and Materials Congress, Istanbul, June 11-15, 1997.

A. Erdemir, Member, Annual Meeting Program Committee, 52nd Annual Meeting of Society of Tribologists and Lubrication Engineers (STLE '97), Kansas City, MO, May 18-22, 1997.

A. Erdemir, Session Chair, Solid Lubricants II: Carbon, Graphite, 52nd Annual Meeting of Society of Tribologists and Lubrication Engineers (STLE '97), Kansas City, MO, May 18-22, 1997.

A. Erdemir, Session Chair, Advanced Materials and Coatings I, 52nd Annual Meeting of Society of Tribologists and Lubrication Engineers (STLE '97), Kansas City, MO, May 18-22, 1997.

A. Erdemir, Session Co-Chair, Symposium E, Tribological Coatings/Surface Modifications, International Conference on Metallurgical Coatings and Thin Films, San Diego, April 21-25, 1997.

A. Erdemir, Invited Member, International Scientific Committee for the Asian-European International Conference on Plasma Surface Eng. (AEPSE '97), Seoul, Oct. 5-9, 1997.

A. Erdemir, Member, Advisory Committee, Sec. B, "Corrosion and Tribology of Advanced Ceramics," 9th International Conference on Modern Materials & Technologies (CIMTEC '98), Florence, Italy, June 14-19, 1998.

A. Erdemir, Session Chair, Surface Modification to Reduce Friction and Wear II, ASM International Materials Solutions Conference, Indianapolis, Sept. 15-18, 1997.

R. C. Niemann, Chairman (Invited) "Leads and Power Cables," Session LMB, Applied Superconductivity Conference, Aug. 1996.

R. C. Niemann, Member, Fermilab High- T_c Lead Proposal Review Panel, Sept. 1996.

R. C. Niemann, Invited Chairman, "Current Leads," Session GPH, Cryogenic Engineering Conference, Portland, OR, July 1997.

J.-H. Park, INVITED member of Advisory Committee for International Symposium on Value Addition Metallurgy at 1997 TMS Annual Meeting, Orlando, Feb. 9-13, 1997.

D. Singh, INVITED chair, session on "Ceramic Composites: Interface/Interphases Stability," 21st Annual Conference & Exposition, Cocoa Beach, FL, Jan. 12-16, 1997.

D. Singh, Chair, Chicago-Milwaukee Chapter, American Ceramic Society, July 1996 to July 1997.

L. R. Turner, INVITED speaker and session chairman, CEFC, Okayama, Japan, March 1996.

L. R. Turner, Invited speaker and session chairman, 3rd International Conference on Electromagnetic Field Problems & Applications, Wuhan, China, Oct. 1996.

L. R. Turner, Invited speaker and session chairman, Asian TEAM Workshop, Yichang City, China, Oct. 1996.

Editorial Activities

U. Balachandran, Reviewer for J. of Materials Research.

U. Balachandran, Reviewer of paper for J. Materials Research, Oct. 23, 1996.

U. Balachandran, Reviewer of paper for Materials Research Bulletin, Dec. 1996.

U. Balachandran, Reviewer of paper for Applied Superconductivity, Jan. 11, 1997.

U. Balachandran, Reviewer for J. of the Electrochemical Society, April 10, 1997.

U. Balachandran, Technical Editor, Advances in Cryogenic Engineering, Vol. 44.

U. Balachandran, Reviewer for J. of Applied Physics.

U. Balachandran, P. J. McGinn, and J. S. Abell, High Temperature Superconductors: Synthesis, Processing, and Large-Scale Applications, Co-Editor Proc. Symp. sponsored by EMPMD and SMD of TMS, Anaheim, CA, Feb. 4-8, 1996.

N. P. Bansal and J. P. Singh, Co-Editor of Advances in Ceramic-Matrix Composites III, Ceramic Transactions Vol. 74, 1996.

Y. S. Cha, Reviewer, J. of Applied Physics.

Y. S. Cha, Reviewer, DOE SBIR Phase I Grant Application.

Y. S. Cha, Reviewer, IEEE Transactions on Applied Superconductivity.

Y. S. Cha, Reviewer, ASME Transactions, J. of Energy Resource Technology.

Y. S. Cha, Reviewer, Physica C - Superconductivity.

S. S. Chen, Associate Editor, J. of Pressure Vessel Technology, ASME, 1996-1999.

S. S. Chen, Member, International Advisory Editorial Board of ACTA Mechanica Solida Sinica (1990 to present).

S. S. Chen, Member, Advisory Board of Japan Society of Mechanical Engineers International J. (1993-1996).

S. S. Chen, Invited to serve as Associate Editor of Applied Mechanics Reviews, ASME (1995-1998).

H. M. Chung, Member Editorial Board of J. of Materials Chemistry and Physics, 1992 to present.

R. A. Erck, Associate Editor of Lubrication Engineering, 1994 to present.

A. Erdemir, Associate Editor of Tribology Transactions, 1994 to present.

A. Erdemir, Reviewer for J. Appl. Phys., Appl. Phys. Lett., J. of Tribology, Tribology Trans., Lubrication Engr., Wear, Surface and Coatings Tech., Thin Solid Films, Tribology Lett.

A. Erdemir, Associate Editor, Society of Tribologists and Lubrication Engineers, 1995-1996.

A. M. Hassanein, Reviewer for J. of Nucl. Mater., Physics of Plasma, Fusion Techn., J. of Appl. Electromagnetics in Mater., and J. of Appl. Physics.

A. B. Hull, Co-editor and Member of Publishing Committee for Proc. 7th Intl. Conf. on Fusion Reactor Materials (ICFRM-7); Obninsk, Russia, Sept. 25-29, 1995; in J. Nucl. Mater. 233-237, 1996, Parts A and B.

P. N. Kumta, G. S. Rohrer, and U. Balachandran, Coeditors, "Role of Ceramics in Advanced Electrochemical Systems," Ceram. Trans. Vol. 65, published by American Ceramic Society, Westerville, OH, 1996.

D. S. Kupperman, Associate Editor, Applied Physics Ltrs., 1992 to present.

D. S. Kupperman, Associate Technical Editor of journal "Materials Evaluation," 1984 to present.

M. T. Lanagan, Reviewer, J. American Ceramic Society, July 1996.

M. T. Lanagan, Reviewer for Superconductor Science and Technology, Physica C, Applied Physics Letters, J. Materials Research, J. American Ceramic Society, J. Applied Physics, Applied Superconductivity.

M. T. Lanagan, Reviewer, Physica-C, Superconductivity, Oct. 1996.

R. C. Niemann, Peer Reviewer, "Cryogenics," Sept. 1996.

R. C. Niemann, Peer Reviewer, "Wiley Encyclopedia of Electrical and Electronics Engr.," May 1997.

R. B. Poeppel, Editor-in-Chief, Applied Superconductivity, Elsevier Science, Ltd., 1992 to present.

R. B. Poeppel, Author of Superconductivity Section, The World Book Encyclopedia, 1992 to present.

J. L. Routbort, Associate Editor, Applied Physics Letters, January 1990 to present.

L. R. Turner, Served on editorial board and reviewed papers for CEFC, Okayama, Japan, March 1996.

L. R. Turner, Served on editorial board and reviewed papers for COMPUMAG-Rio, May 1997.

L. R. Turner, Reviewed papers for 41st Magnetism and Magnetic Materials Conf., Atlanta, Nov. 1996.

L. R. Turner, Reviewed papers for 1996 Applied Superconductivity Conf., Nov. 1996.

L. R. Turner, Reviewed paper for J. of Applied Physics, Nov. 1996.

L. R. Turner, Reviewed paper for Fusion Engineering and Design, April 1997.

L. R. Turner, Reviewed paper for COMPEL, Sept. 1996.

T. Wiencek, Contributing author to quarterly report, Reduced Enrichment Research and Test Reactors (RERTR) Program, June 1996-May 1997.

ATIT P. 4
72%

ATF 2

ATH p. 5
72%

ATH p. 5

$J = 3$

$J = 2$

2.00 m/s
Time: 10.70 s

1 Ceramics p. 85

55.

of 87.

78%

↑
5 1/2
↓

Ceramics p 88 LEFT

S.S.,
full frame

Ceramics p. 88, P154T

S.S., full
frame

Corrosion p 115

90% full
frame

Fluorine Ion Maps

Oxygen Ion Maps

Fusion
Zone

Heat Affected Zone

1.0 mm

Electromechanics
p. —

87%

← 3 1/2" →

↑
5 1/4"
↓

Handwritten: $\frac{160}{70\%}$

Corrosion p. 16
(upper fig.)
80%

|---|
1 μ m

Electron micrograph of shadowed replica of U-14 wt. % Mo specimen irradiated to 0.22% burnup at 800°C, and DART-calculated bubble-size distributions for the irradiation.

80%

Handwritten notes:
C. J. ...
P. ...
(...)

SINCE P. - 90%

2 1/2"

← 4 3/4" →

← 5 7/16

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80 81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100

INCHES

← 3 3/16 →

COMPUTATIONAL EM MODELING

FEM-Based Simulation of Eddy Current Probe Response for NDE of Steam Generator Tubes

Distribution of current density

Differential bobbin coil inside Inconel 600 tube
embedded with a narrow axial notch

Coolant
Heat Exchanger

Test
Section

Refrigerant
After-condenser

Refrigerant
By-pass Cooler

Coolant
Pump

Refrigerant
Pump

Refrigerant
Evaporator /
Superheater

Fig 213 415 67 819 101 121

Fig. 2. Predicted Nusselt number vs. experimental Nusselt number from four test series summarized in Table 1 (438 data points)

TEMP 152

70,

↑

43/

↓

← 4" →

TC WP P. 53

83

↑
8"
↓

1-55/ "

TEMP 8.54

(90%)

57
1/4

Fig. 5. Endothermic fuel heat transfer apparatus

6 3/8"

TMP
P. —

65.7

Fig. 6. Enhanced thermal conductivity of oxide nanofluids system

Geometry of the RH72B Package

Numerical Model of the RH72B Package

9676

Thickener

9676

100-371
170
100

74

6570