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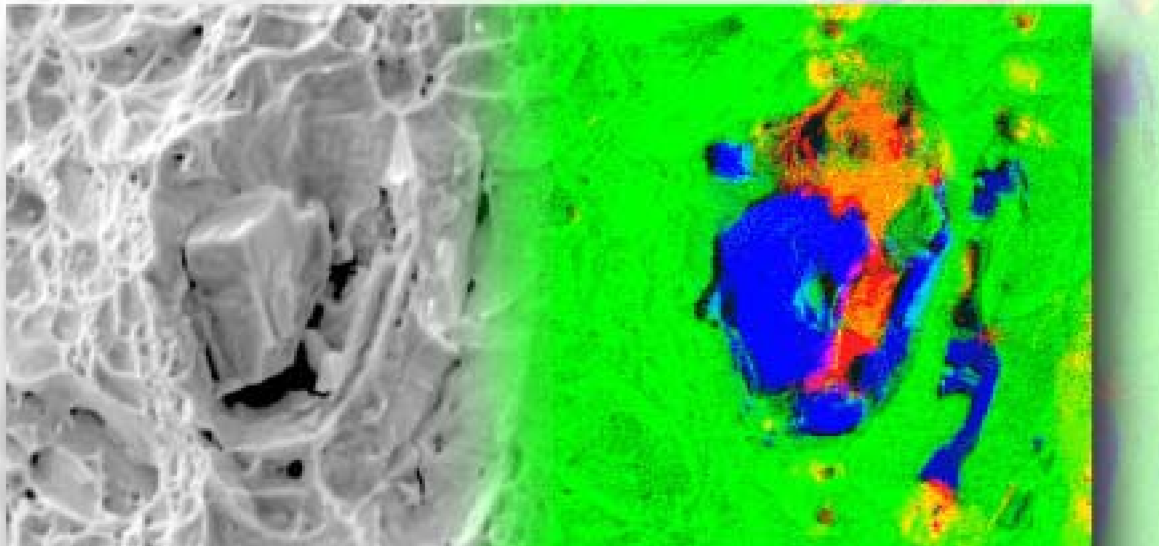
ORNL/TM-2002/57

High Temperature Materials Laboratory Annual Report

FY 2001 (October 1, 2000–September 30, 2001)



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HIGH TEMPERATURE MATERIALS LABORATORY

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High Temperature Materials Laboratory

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A. E. Pasto
B. J. Russell

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ACRONYMS AND ABBREVIATIONS

ACA	American Crystallographic Association
ACEM	aberration-corrected electron microscope
AEM	analytical electron microscope
AISI	American Iron and Steel Institute
AMCL	Advanced Materials Characterization Laboratory
ANSI	American National Standards Institute
ASTM	American Society for Testing and Material
BES	DOE Office of Basic Energy Sciences
BGA	ball grid array
CRADA	cooperative research and development agreement
CTE	coefficient of thermal expansion
CVI	chemical vapor infiltration
DOE	U.S. Department of Energy
DUC	Diffraction User Center
EB-PVD	electron-beam physical vapor deposition
EBSD	electron-backscattered diffraction
EDS	energy-dispersive spectrometer
EE/RE	DOE Office of Energy Efficiency and Renewable Energy
EFAS	electric-field-assisted sintering
ELSAM	Ernst Leitz Scanning Acoustic Microprobe
FEG	field-emission gun
FIB	focused ion beam
FSW	friction-stir weld
HFET	heterostructure field-effect transistor
HFIR	High Flux Isotope Reactor
HTML	High Temperature Materials Laboratory
HTXRD	high-temperature X-ray diffraction/diffractometer
ISO	International Standards Institute
IR	infrared
LAS	Lord, Aeck and Sargent, Inc.
LEK	Lord, Aeck and Sargent, Inc.
LDRD	Laboratory Directed Research and Development
MAUC	Materials Analysis User Center
MCAUC	Mechanical Characterization and Analysis User Center
MEA	membrane electrode assembly
MITUC	Machining, Inspection, and Tribology User Center
NDE	nondestructive evaluation
NRSF	Neutron Residual Stress Mapping Facility
ODF	orientation distribution function
OHVT	DOE Office of Heavy Vehicle Technologies
OIT	DOE Office of Industrial Technologies
ORNL	Oak Ridge National Laboratory
PAN	polyacrylonitrile
PTS	powder-texture stress
R&D	research and development
RSUC	Residual Stress User Center

RUS	resonant ultrasound spectroscopy
SAN	scanning auger nanoprobe
SEM	scanning electron microscope
SIU	Southern Illinois University
SNS	Spallation Neutron Source
SRS	Savannah River Site
STEM	scanning transmission electron microscope
TBC	thermal barrier coating
TEM	transmission electron microscopy
TGO	thermally grown oxide
TPUC	Thermophysical Properties User Center
UTK	University of Tennessee at Knoxville
UTSI	University of Tennessee Space Institute
WSRC	Westinghouse Savannah River Company
XRD	X-ray diffraction
YSZ	yttria-stabilized zirconia

ADVANCED MATERIALS CHARACTERIZATION AT THE HIGH TEMPERATURE MATERIALS LABORATORY

Arvid E. Pasto, Director

The Facility

The High Temperature Materials Laboratory (HTML) is a national user facility designed to support the development of advanced materials. It is sponsored by the U.S. Department of Energy (DOE) Office of Transportation Technologies, in the Office of Energy Efficiency and Renewable Energy. HTML provides researchers from U.S. industries, universities, and governmental agencies with access to a skilled staff and to a number of sophisticated, often one-of-a-kind devices for materials characterization. Located at Oak Ridge National Laboratory (ORNL), the 64,500-ft^2 building houses six "user centers," which are clusters of specialized equipment designed for specific types of properties measurements.

HTML was conceived and built in the mid-1980s in response to the oil embargoes of the 1970s. The concept was to build a facility that would allow direct work with American industry, academia, and government laboratories in providing advanced high-temperature materials such as structural ceramics for energy-efficient engines. HTML's scope of work has since expanded to include other, non-high-temperature materials of interest to transportation and other industries.

The User Centers

Materials Analysis User Center (MAUC)

MAUC researchers employ electron microscopy and surface chemical analysis to determine structure, surface chemistry, and microstructure to the atomic level. Advanced microscopy capabilities allow rapid, direct elemental analysis of grain boundaries in metals and ceramics. Auger spectroscopy is available for analyzing material surfaces.

Machining, Inspection, and Tribology User Center (MITUC)

MITUC employs instrumented grinders to investigate the grinding process as applied to hard materials such as ceramics and special alloys. These dynamometer-equipped machine tools provide unique capabilities for studying grinding parameters and their roles in controlling the topography and mechanical and wear properties of the resulting surfaces. Dimensional inspection capabilities include a multisensor coordinate measuring machine, instruments for measuring surface texture and topography, and instruments for determining the dimensional form of axially symmetric objects (e.g., circularity, cylindricity, and concentricity). In addition, MITUC contains numerous specialized instruments for measuring friction and wear, including fretting, rolling, and sliding.

Mechanical Characterization and Analysis User Center (MCAUC)

MCAUC researchers study fracture toughness, tensile strength, flexure strength, and tensile creep of advanced materials at temperatures to 1500°C in air or controlled atmospheres. Special instrumentation is available for studying fiber-matrix interactions in both metal and ceramic matrix composites.

Diffraction User Center (DUC)

DUC has both room-temperature and furnace-equipped X-ray and neutron diffractometers. The X-ray furnace is used to study material properties at temperatures up to 2700°C in vacuum and up to 1500°C in air. DUC users have access to the National Synchrotron Light Source at Brookhaven National Laboratory.

Residual Stress User Center (RSUC)

RSUC has two principal areas of expertise: X-ray diffraction and neutron diffraction. Its X-ray facility includes X-ray diffractometers to measure residual stress and texture in and near the surface of ceramics and alloys. Two systems provide highly flexible sample-tilt systems and either a divergent or a parallel beam. Users can also access the National Synchrotron Light Source, located at Brookhaven National Laboratory, through RSUC. HTML maintains a beamline at Brookhaven with structure and residual stress analysis capability. The neutron residual stress facility includes a special neutron spectrometer for rapid data collection, plus computer capabilities for data analysis. The spectrometer instrumentation is located at the High Flux Isotope Reactor (HFIR) at ORNL. This facility allows researchers to quickly measure and map the stress fields inside relatively large solid objects.

Thermophysical Properties User Center (TPUC)

TPUC researchers study thermal stability, expansion, and thermal conductivity of materials to 1400°C. A laser flash instrument measures thermal diffusivity to temperatures of 1900°C. The center also possesses a high-speed, high-sensitivity infrared camera for capturing thermal events digitally, allowing on-line or postoperation measurement of temperatures during rapid transient events.

The Programs

Within HTML are programs that function to help outside researchers use state-of-the-art characterization instrumentation to solve materials problems. In the HTML User Program, either nonproprietary or proprietary research can be performed. The former is provided free of charge if the user publishes the information produced, while the latter requires payment.

Nonproprietary research projects typically last from 1 to 3 weeks at HTML. The major proviso is that the results must be submitted for publication within 6 months after completion of the research.

For proprietary research, the user and HTML staff estimate the amount of HTML staff time required to complete the work. The user agrees to pay for this time at an hourly rate specified by DOE before research begins. These projects typically are more extensive than nonproprietary projects, and the user owns the research data.

Work is performed for other branches of DOE via direct funding or through cooperative research and development agreements (CRADAs), which typically consist of a cost-sharing arrangement between HTML and the outside organization but can also include 100% funds-in work. HTML can also characterize materials for another organization on a noncompetitive, full-cost-recovery basis under a Work for Others agreement.

Most, but not all, projects involve materials primarily related to the transportation industry. Ceramics, metal- and ceramic-matrix composites, lightweight materials such as aluminum and magnesium alloys, steels, and electronic materials have all been characterized at HTML.

HTML DIRECTOR'S REPORT

Dr. Arvid Pasto is the director of the HTML. Arvid holds a Ph.D. in Ceramics from the New York State College of Ceramics at Alfred University.

The HTML User Program continued to work with industrial, academic, and governmental users this year, accepting 92 new projects and developing 48 new user agreements. Table 1 presents the breakdown of these statistics. Figure 1 depicts the continued growth in user agreements and user projects. You will note that the total number of HTML proposals has now exceeded 1000. Also, the large number of new agreements bodes well for the future. At the end of the report, we present a list of proposals to the HTML and a list of agreements between HTML and universities and industries, broken down by state.

Program highlights this year included several outstanding user projects (some of which are highlighted in later sections), the annual meeting of the HTML Programs Senior Advisory Committee, and approval by ORNL for the construction of a building to house our new aberration-corrected electron microscope (ACEM) and several other sensitive electron and optical instruments.

The new building, to be called the Advanced Materials Characterization Laboratory (AMCL), will be located adjacent to the HTML, in what is now a parking lot (see Fig. 2). The building will be specially designed and constructed to provide an environment as free from vibration and electric and magnetic fields as possible. Construction is slated to begin in the spring of 2002 and to be completed by summer of 2003, when the ACEM is due to arrive.

Table 1. Statistics of HTML operations for FY 1999–FY 2001, showing user proposals and user agreements

FY	New Proposals				Cumulative Proposals			
	Total	Industrial	University	Other	Total	Industrial	University	Other
1999	91	39	49	3	906	387	497	22
2000	86	32	48	6	992	419	545	28
2001	92	33	49	10	1084	452	594	38
	New Agreements				**Cumulative Agreements**			
	Total	Industrial	University	Other	Total	Industrial	University	Other
1999	37	26	11	0	481	283	182	16
2000	50	38	8	4	531	321	190	20
2001	48	32	15	1	579	353	205	21

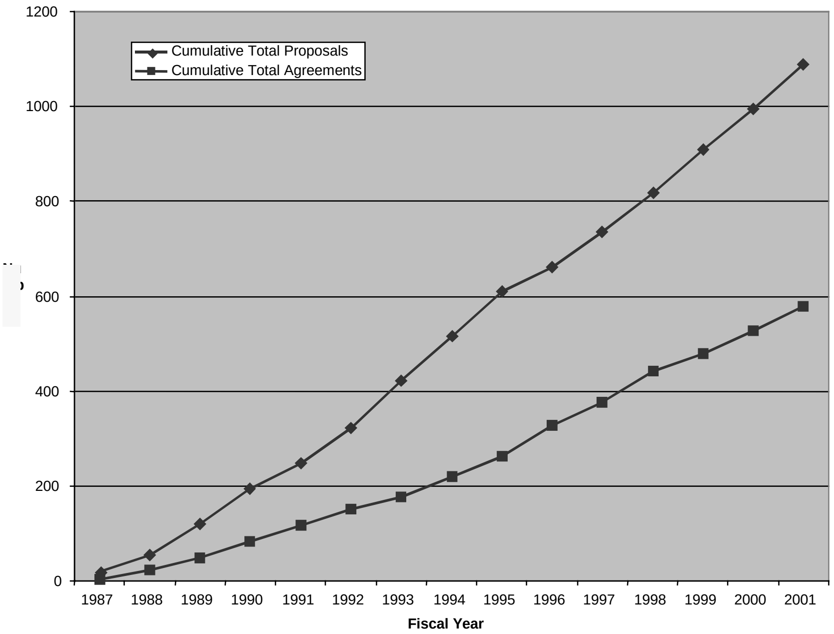


Fig. 1. Plot showing HTML User Program growth through user agreements and user proposals.

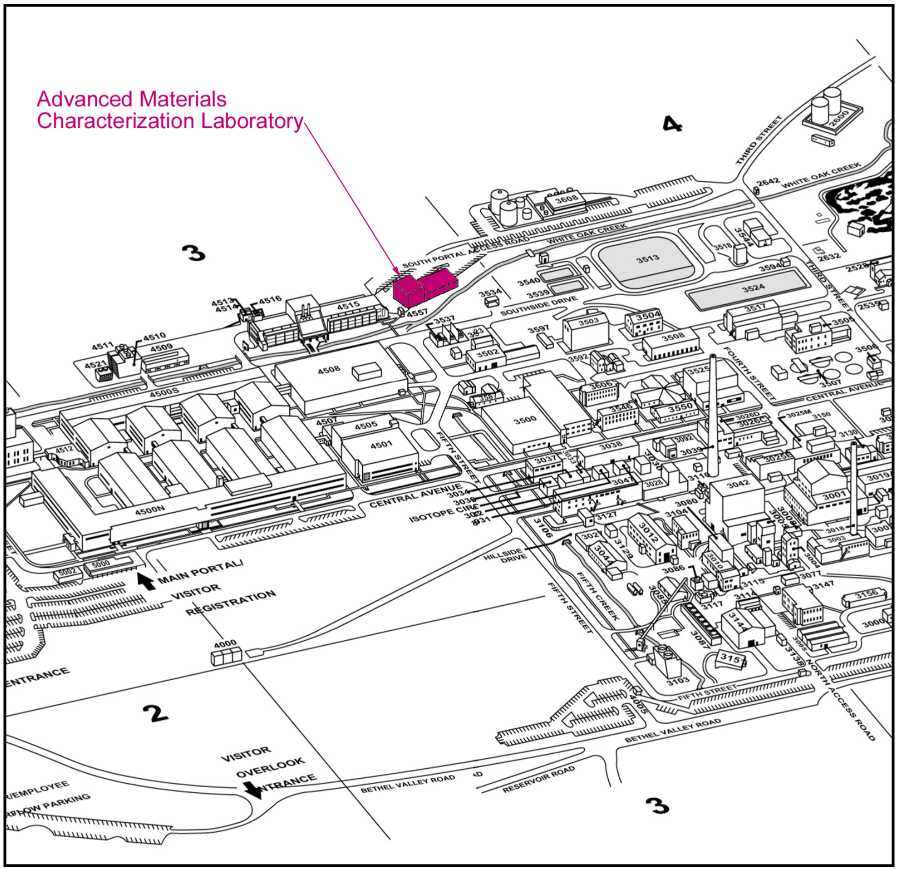


Fig. 2. Location of the new Advanced Materials Characterization Laboratory (AMCL).

1. MATERIALS ANALYSIS USER CENTER (MAUC)

Group Members

Larry Allard, User Center Leader
Carolyn Wells, Secretary

Doug Blom
Dorothy Coffey
Bernhard Frost, University of Tennessee, Electron Holography
David Joy, University of Tennessee, Distinguished Scientist
Harry Meyer
Karren More, Guest Researcher, Transmission Electron Microscopy
Ted Nolan
Larry Walker

The Materials Analysis User Center (MAUC) provides world-class facilities and a staff of technical experts for characterizing the structure and chemistry of advanced materials. We emphasize using these tools to relate microstructure to materials performance. The MAUC comprises a suite of laboratories that contain the latest generation electron microscopes and surface analysis instruments, all of which are available to visiting researchers. Research specialties include characterization of nanophase materials such as catalysts, fullerenes (carbon nanotubes), and nanoparticulates; structural ceramics; electron holography (e.g., for dopant profiling in semiconductors); and characterization of multilayer surface films.

MAUC Instruments

- Hitachi S4700 field-emission gun (FEG) scanning electron microscope (SEM) with energy-dispersive spectrometer (EDS)
- Hitachi S-800 FEG SEM with EDS
- Hitachi HF-2000 FEG analytical electron microscope (AEM)
- Hitachi HD-2000 FEG scanning transmission electron microscope (STEM)
- JEOL 8200 electron microprobe (Spring 2002)
- PHI 680 scanning auger nanoprobe (SAN)
- Hitachi FB-2000 focused ion beam (FIB) micromill
- Leica UCT ultramicrotome with cryosectioning capability

MAUC News

TelePresence Microscopy

All MAUC instruments are configured for computer-controlled operation and digital imaging. They are therefore accessible via internet connections that, with video conferencing, allow collaborative, live-time research sessions or “TelePresence Microscopy” sessions between ORNL staff and outside users. This facilitates the timely completion of user projects, with the additional benefit of minimizing travel costs for MAUC users.

Update on New Advanced Materials Characterization Laboratory

The architectural firm Lord, Aeck and Sargent, Inc. (LAS), of Atlanta, has progressed on the plans for the new Advanced Materials Characterization Laboratory (AMCL) and has recently delivered the "30% submittal" plan (Fig. 1-1). The building design to this 30% point has been developed in close collaboration with MAUC staff and ORNL engineers. A budget of $4.8M has been provided (General Plant Project funds), of which about $3.1M will be available for the construction costs. Initial estimates by LAS suggest that the budget will support a laboratory with eight instrument rooms, along with a "core support" section that will attach to the laboratory section via an air-lock arrangement. The core support section will have two floors, the lower of which will house two offices for laboratory technical staff, a sample preparation room, and a conference room/lobby that will accommodate "virtual" tours. The upper floor will house the heating, ventilation, and air-conditioning equipment; electrical distribution panels; and water chiller units for the instruments. The AMCL is designed to provide an ultimately quiet environment for the sensitive instruments. The specialized construction parameters are substantially more critical, and therefore more costly, than standard construction practices. Thus, an "office section" designed as part of the core support section has been added as an alternate plan, as it may not be able to be constructed within the available budget.

Fig. 1-1. Artist's rendering of the new Advanced Materials Characterization Laboratory.

Ultramicrotome Added to MAUC Specimen Preparation Instrument Suite

A new Leica UCT ultramicrotome with cryosectioning capability has recently been added the to the transmission electron microscopy (TEM) sample preparation equipment available to MAUC users. The ultramicrotome uses a diamond knife to slice thin sections of materials for TEM. Reproducible section thicknesses of 70 nm are routinely achieved, allowing for high-resolution imaging and microchemical analysis. The cryosectioning option allows for sectioning of materials that are too elastic at room temperature to be sectioned well. By cooling the material (typically a polymer) to a low temperature (between 0 and –40°C), often the sectioning of 70-nm-thick slices becomes possible. Ultramicrotomy is a standard technique in the biological sciences and for polymer-based materials; it has become more common recently for "materials science" samples that are difficult to prepare by traditional materials science techniques, either because of their porosity or because they have received ion-beam damage. Initial successes with this technique have been achieved in studies of the structure of membrane electrode assemblies (MEAs) in proton exchange membrane fuel cells. Figure 1-2 shows a cross section of an MEA, which exhibits a relatively large area of electron-transparent material, allowing direct characterization of the size and distribution of heavy metal catalyst species.

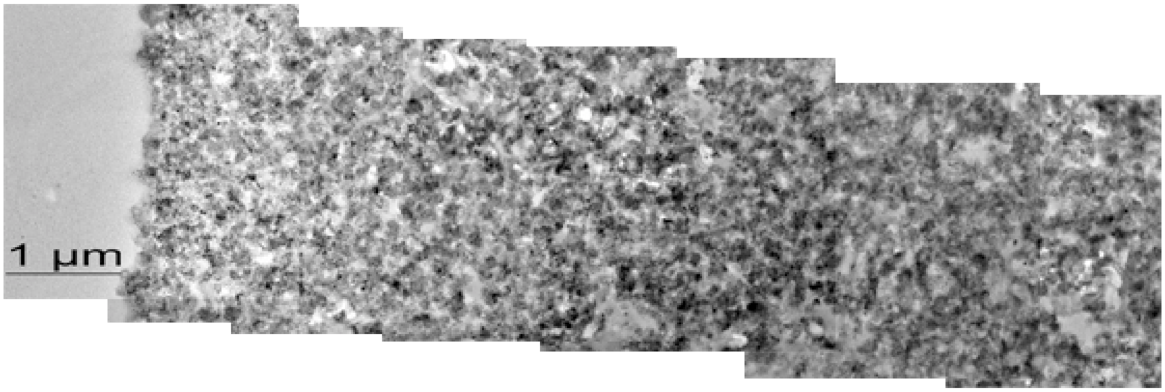


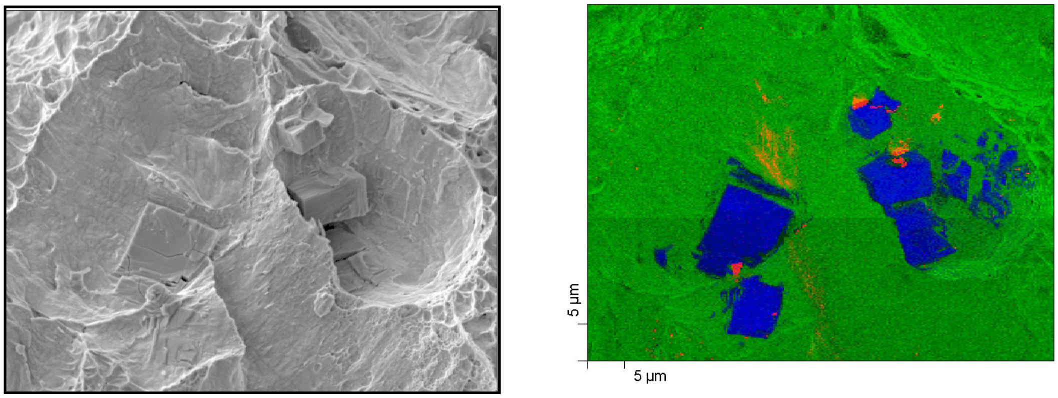
Fig. 1-2. Membrane electrode assembly sections prepared with the Leica UCT ultramicrotome.

Selected Highlights

Elemental Segregation and Fracture Toughness in Steels—Caterpillar Inc.

Industrial Collaborator: C. Hsieh
HTML: H. Meyer

The task performed for this user project was to investigate a series of low alloy steel samples with the PHI 680 SAN to identify the elements that segregate to interfaces and contribute to fracture initiation. The samples, prepared under a variety of process conditions, contained titanium nitride cubic inclusions (so called "cuboids") that are known to be located in "pits" that have been identified as fracture initiation sites [Fig. 1-3 (a)]. A dozen samples were fractured inside the SAN, and their surfaces were characterized by secondary electron imaging and Auger elemental mapping. Elemental maps of iron, titanium, and sulfur revealed the presence of sulfur at the interface between the steel matrix and the cuboids [Fig. 1-3 (b)]. The data clearly show that sulfur preferentially segregates to the TiN/steel interface and probably affects the ultimate fracture toughness. During this initial investigation, a direct correlation between the quantitative amount of S that was detected and fracture toughness could not be established. This project is ongoing; further work is planned in the near future.



(a) (b)

Fig. 1-3. (a) Pits in low alloy steel, known fracture-initiation sites. (b) Sulfur identified at the interface between TiN cuboids and the steel matrix within the pits.

Novel Characterization of Bulk Aluminum Nitride Crystal Growth—Kansas State University

*University Collaborators**:** J. Edgar and B. Liu*
HTML: L. Walker

The growth of bulk aluminum nitride crystals by vapor transport processes on silicon carbide substrates is being studied by Prof. Jim Edgar and graduate student, Bei Liu. A number of processing parameters are being studied to see how they ultimately affect the nucleation and growth of the AlN crystals. These novel materials are expected to have many applications in the semiconductor industry. To characterize the structure and morphology of the AlN crystals, and to determine their crystallographic orientations one to another and relative to the substrate, Ms. Liu used the Noran electron backscattered diffraction (EBSD) pattern capability presently on consignment on the Hitachi S-4700 SEM. Figure 1-4 illustrates the beautiful results: a secondary electron image of a number of AlN crystals on a SiC substrate. EBSD patterns collected from the SiC and the AlN single-crystal “islands” are shown, respectively, in the upper and lower inset pictures. These patterns clearly show an epitactical crystallographic relationship between the crystals and the substrate. In all cases, the crystals also were oriented similarly with respect to each other. This technique is optimal for samples such as these and allows a large number of measurements to be made in a relatively short time. The evaluation of the relationship between processing parameters and quality of AlN crystal growth is being expedited by these techniques.

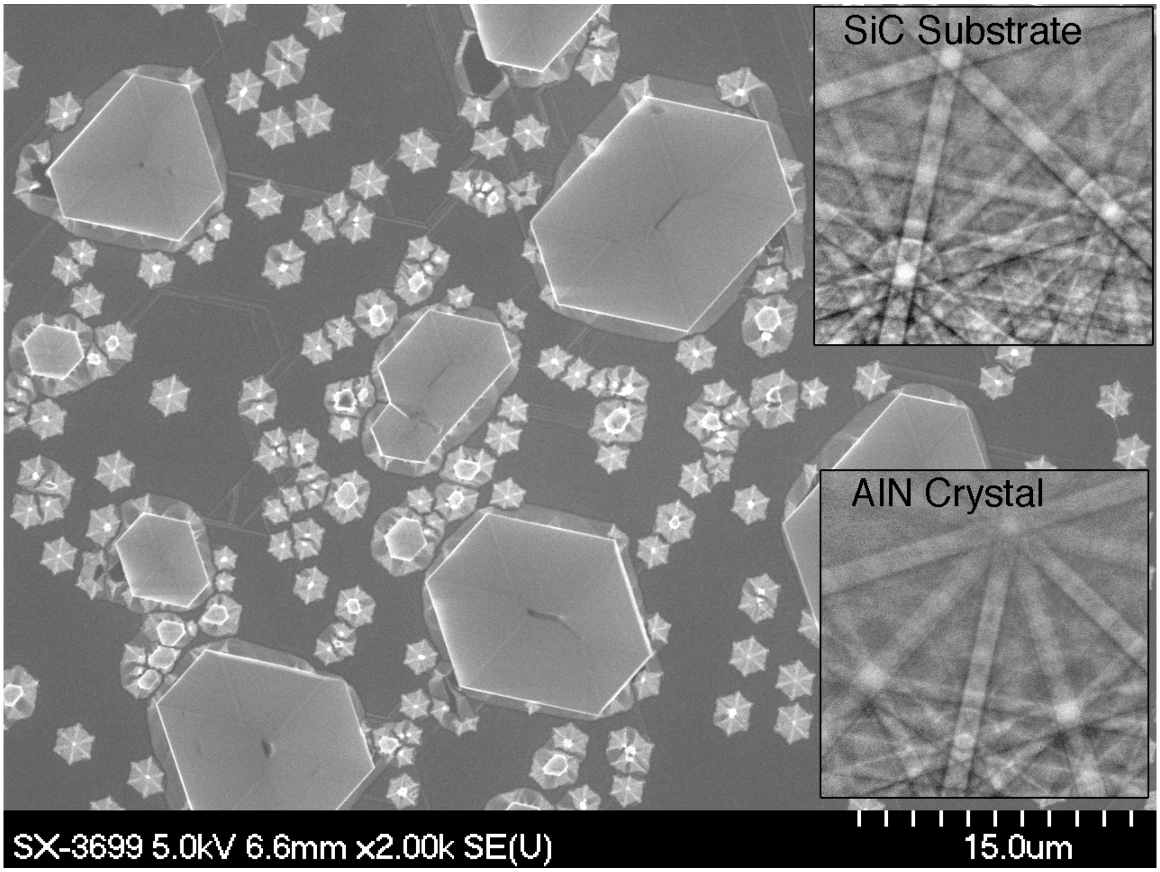


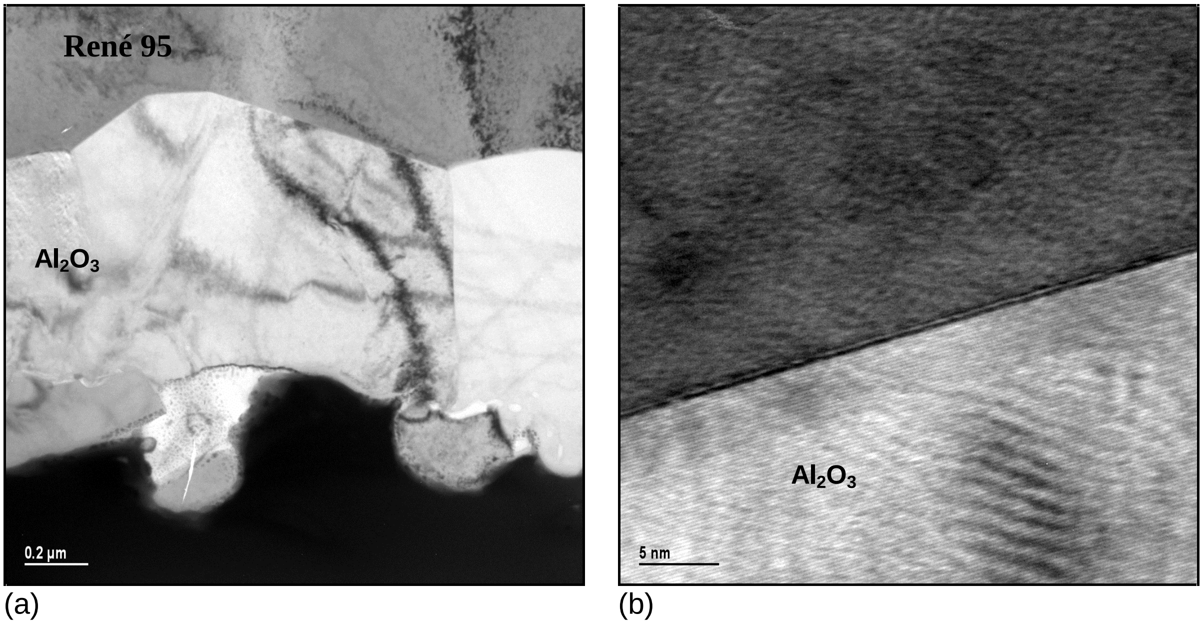
Fig. 1-4. Secondary electron image of a number of AlN crystals on a SiC substrate. *Inset:* EBSD patterns collected from the SiC (upper) and the AlN (lower) single-crystal “islands.”

Studies of Alumina "Template" Layers as Thermal Barrier Coatings on a Nickel-Based Superalloy—Stephens Institute of Technology

University Collaborators: W. Lee and Y.-F. Su
HTML: L. Allard

René N5, a nickel-based superalloy, is commonly used in the rotating airfoils of aircraft jet engines. Thermally grown oxides (TGOs) that develop on the surface of the alloy protect it against further oxidation. Prof. Woo Lee and graduate student Yi-Feng Su are studying the effect that a layer of alumina has on the development of the TGOs. They are using the Hitachi HF-2000 field-emission TEM to compare oxides produced on untreated samples of single-crystal René N5 with samples on which a layer of alumina has been deposited by chemical vapor deposition.

The Hitachi FB-2000 focused ion beam (FIB) mill was used to prepare thin specimens of the superalloy with two different as-deposited alumina layers and with several samples that had been subsequently oxidized for different times. The progress of growth of oxides on the alloy with the alumina template layer compared with growth on the alloy with no alumina layer is being studied by direct TEM imaging, electron diffraction, and energy-dispersive X-ray spectroscopy techniques. Figure 1-5 (a) shows an overall view of the alumina film (middle layer) on the René 95 alloy. Figure 1-5 (b) shows a high-resolution lattice image of a part of the interface between the alumina film and the alloy. The preserved ridges and the other structures were consistent with oxygen inward diffusion dominating in the alumina as the TGO grew in a columnar fashion. The lack of voids and delaminations at the interface suggests excellent adherence of the TGO to the base alloy. Much of the work on this project was conducted via remote microscopy sessions between Stevens Institute and the HTML.



(a) (b)

Fig. 1-5. (a) An overall view of the alumina film (middle layer) on the René 95 alloy. (b) A high-resolution lattice image of a part of the interface between the alumina film and the alloy, showing excellent adherence.

2. MACHINING, INSPECTION, AND TRIBOLOGY USER CENTER (MITUC)

Staff Members

Sam McSpadden, User Center Leader
Roxanne Raschke, Secretary

Peter Blau
Tyler Jenkins
Lawrence O'Rourke
Ron Ott
Randy Parten
Earl Shelton

The Machining, Inspection, and Tribology User Center (MITUC) provides basic facilities for investigation of grinding processes for high-performance materials, design and fabrication of mechanical property test specimens, dimensional characterization of test specimens and other components, and tribology.

Several types of numerically controlled grinders are available to guest researchers for their projects at MITUC. The grinders were selected for their similarity to those used in manufacturing facilities throughout the United States. Grinders are instrumented to permit real-time measurement of key grinding process parameters (including forces, spindle horsepower, spindle vibration, acoustic emission, and coolant temperature). Data may be collected, displayed, stored, and analyzed using specialized Labview programs and other analysis software. Dimensional inspection instruments are provided for the accurate measurement of the size, form, surface texture, and surface topography of geometric features on materials specimens and mechanical components. Specialized tribological instruments are provided for studying the friction and wear behavior of materials under controlled environmental conditions.

MITUC Instruments

MITUC Machining Instruments

- Chand-Kare grindability test system
- Cincinnati Sabre multi-axis grinder
- Harig surface grinders
- Instrumented K.O. Lee Vigor creep-feed grinder
- Instrumented Nicco creep-feed grinder
- Instrumented Weldon cylindrical grinder

MITUC Inspection Instruments

- EMD Legend integrated metrology center
- Mahr Formtester
- Nikon optical comparator
- Rodenstock 600-laser surface profile-measuring system (noncontact)
- Taylor Hobson Talysurf 120 stylus surface-profile-measuring system (contact)

MITUC Tribology Instruments

- Friction microprobe
- High-temperature pin-on-disk system
- Image analyzer
- Instrumented scratch tester
- Lubricant load-carrying-capacity screening rig
- Microindentation hardness tester
- Multimode friction and wear tester
- Pin-on-disk friction and wear testing station
- Portable scratch tester
- Reciprocating friction and wear tester
- Reciprocating sliding wear tester
- Repetitive impact testing system
- Stylus surface roughness measuring system

New MITUC Capabilities

K.O. Lee Vigor Creep-Feed Surface Grinder

The new K.O. Lee Vigor creep-feed surface grinder has been delivered and installed (Fig. 2-1). The grinder performed flawlessly in initial acceptance tests. Instrumentation has been added to the grinder, and it is now ready for HTML user projects. The instrumentation consists of a Kistler dynamometer, a wheel speed sensor, a spindle power sensor, an acoustic emission sensor, an accelerometer, and sensors for measuring coolant temperature, pressure, and flow rate. The grinder is capable of performing both conventional and creep-feed surface-grinding research. It features a horizontally mounted, high-speed, variable-speed spindle, fully enclosed hood, large work table with electromagnetic chuck, and a NUM Model 1040 controller with conversational programming capabilities. Capacities are shown in Table 2-1.

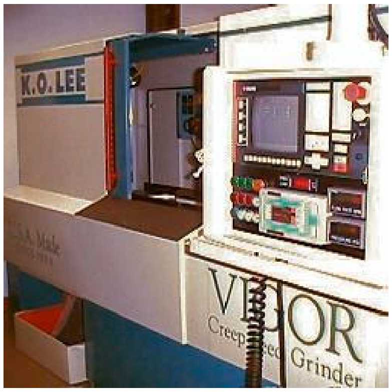


Fig. 2-1. The K.O. Lee instrumented creep-feed grinder.

Table 2-1. K.O. Lee Creep Feed Grinder Capabilities

Work surface	686 mm long × 254 mm wide (27 × 10 in.)
Spindle speed	500 to 7200 RPM
Power, spindle motor	7.5 kW (10 HP)
Down-feed resolution	0.1 µm (0.000004 in.)
Table speed	Variable from 0.004 to 305 mm/s (0.01 to 720 in./min)
Wheel specifications	200 mm diam, up to 38 mm thick, 76.2 mm bore (8 in. × 1.5 in., 3 in.) 300 mm diam, up to 50 mm thick, 127 mm bore (12 × 2 × 5 in.)

High-Speed Friction and Wear Testing System

As part of a research program on advanced brake materials sponsored by the DOE Office of Heavy Vehicle Technologies (OHVT), a new high-speed friction and wear testing system was designed and constructed in the HTML (Fig. 2-2). Equipped with friction force, vibration, and infrared temperature measurement systems, the new apparatus is capable of sliding speeds well in excess of 10 m/s. The original fixtures were designed to test brake pad sections, but a sphere holder has been fabricated to enable it to conduct high-speed pin-on-disk tests as well. This equipment is available through the User Program.

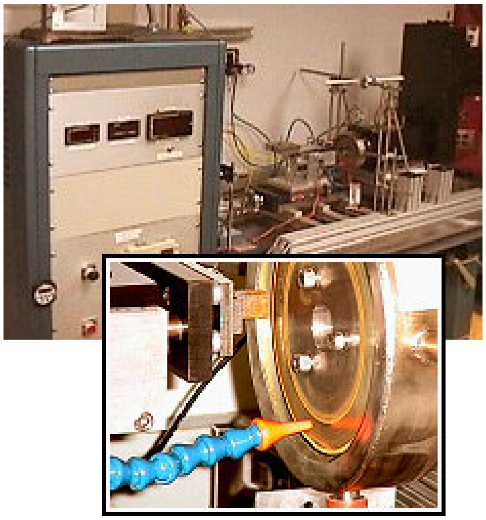


Fig. 2-2. The high-speed friction and wear testing system.

The Ernst Leitz Scanning Acoustic Microscope (ELSAM)

MITUC is developing methods for the nondestructive detection of subsurface damage in ceramic diesel engine components. Heavy-duty diesel engine manufacturers face fuel-economy and emission standards that will become increasingly stringent over the next six years. The allowable level of exhaust gas particulates will decrease by an order of magnitude, and there will be a mandated reduction in the sulfur content of diesel fuels with an attendant reduction in lubricity. This will dictate the use of high-performance, wear-resistant materials in key engine systems, such as fuel-injection and metering components.

Ceramic materials such as partially stabilized zirconia are being used successfully in current high-performance fuel systems, and their use is expected to increase significantly. The most common method of production of ceramic components is precision grinding, which is among the more costly and difficult-to-control machining processes. Ceramic components are susceptible to hidden, subsurface damage caused by the grinding process. This damage, which can lead to costly premature fuel system failures, is not detectable by either visual inspection or traditional nondestructive test methods, such as dye-penetrant testing.

Fig. 2-3. The Ernst Leitz Scanning Acoustic Microscope (ELSAM).

Scanning acoustic microscopy has been used successfully to detect subsurface machining damage in ceramic materials. The instrument used to demonstrate this technology was manufactured by Ernst Leitz, Wetzlar, GmbH, and is called the ELSAM (Ernst Leitz Scanning Acoustic Microscope) (Fig. 2-3). The ELSAM uses extremely high frequency (up to 2-GHz) acoustic waves to provide information on the surface and subsurface characteristics of materials. The acoustic signal is coupled to the specimen by a thin layer of deionized water as the conducting medium. Details such as grain boundaries, cracks, and flaws are clearly visible in the images provided. The feature that differentiates the ELSAM from conventional microscopy is the ability to detect both surface flaws and subsurface flaws. The depth of flaw detection is a function of the frequency of the acoustic signal.

The ELSAM is an important part of current MITUC machining research efforts, which are aimed at determining the envelope of grinding conditions that can be used to manufacture close-tolerance ceramic components with no subsurface damage. Acoustic microscopy is an enabling technology that should lead to the widespread use of ceramic components in future heavy-duty diesel engines. Among the expected benefits are

- reduced engine warranty costs;
- improved engine durability, performance, and fuel economy;
- better understanding of grinding process variables that cause subsurface damage; and
- less destructive testing required to ensure damage-free engine components.

Selected Highlights

Collaborations with North Carolina State University

University Collaborators: A. J. Shih, A. C. Curry, B. K. Rhoney, and W. Clark
HTML: T. Jenkins and S. McSpadden

MITUC is currently involved in numerous ongoing collaborative efforts with Dr. Albert J. Shih and his students in the North Carolina State University Materials Science and Engineering Department. Most of the projects involve the cost-effective machining of ceramic engine components.

Mr. Adam Curry completed the experimental portion of his master's thesis work at the HTML by developing methods of measuring flash temperatures in the grinding of MgO-doped partially stabilized zirconia. Mr. Curry's experiments were conducted in an attempt to better understand the underlying mechanisms that make it possible to grind zirconia with an inexpensive silicon nitride grinding wheel rather than a superabrasive wheel. Mr. Brian Rhoney completed a significant portion of his master's thesis experimental work at the HTML by investigating cylindrical-wire electrical-discharge-truing of metal-bond diamond grinding wheels. Both Mr. Curry and Mr. Rhoney have been awarded master of science degrees. Mr. William Clark investigated the use of an instrumented, computer numerically controlled, diamond wire saw (Fig. 2-4) to slice a wide variety of materials, ranging from silicon nitride to tool steel to wood.

Fig. 2-4. Instrumented diamond wire saw.

3. MECHANICAL CHARACTERIZATION AND ANALYSIS USER CENTER (MCAUC)

Group Members

Edgar Lara-Curzio, Group Leader
Paula D. Miller, Secretary

Ken C. Liu
Ralph L. Martin
Laura Riester
Christopher O. Stevens
Bob Swindeman

The Mechanical Characterization and Analysis User Center (MCAUC) specializes in the mechanical characterization of functional and structural materials. MCAUC performs mechanical testing and analysis and develops test methods, design codes, and supplemental analytical techniques. Numerous mechanical test frames with uniaxial and multiaxial capabilities are available to visiting researchers from industry and academia to conduct tests in tension, compression, flexure, torsion, shear, and internal pressurization on standard or customized specimens in controlled environments and at elevated temperatures. Facilities also include equipment for micromechanical testing and instrumented indentation. MCAUC staff has expertise with a wide range of materials, testing configurations, failure analysis, finite-element stress analysis, analytical modeling, and life-prediction analysis of materials and structures.

MCAUC Instruments

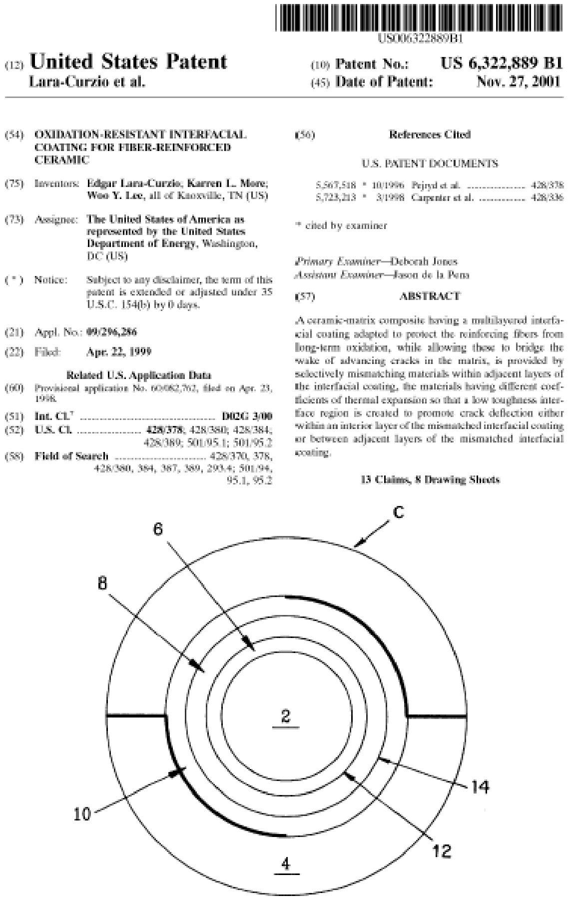
- Electromechanical, servohydraulic, pneumatic, and dead-weight testing machines for testing in tension, compression, torsion, flexure, axial/torsion, and other loading configurations
- Loading capabilities up to 500 kN and 3000 Hz
- High-temperature furnaces (resistance and induction heating, quartz lamps) with capabilities up to 1700°C in air and 3000°C in vacuum or inert environments
- Integral electronic controllers for load, displacement, and strain control, and for computerized data acquisition
- Fixtures for uniaxial and biaxial bending (ring-on-ring), Iosipescu shear testing, anti-buckling compression, and interlaminar shear by compression of double-notched specimens
- Experimental facilities for creep, stress rupture, and stress relaxation testing at ambient and high temperatures and in controlled environments
- Rotary bend fatigue machine equipped with a furnace for testing small cylindrical specimens in fully reversed cyclic loading
- Environmental test facility for testing in vacuum (vacuum levels of 1×10^{-10} torr) or inert gas environments to temperatures up to 3000°C, or in pressurized simulated industrial environments (e.g., steam)
- Microturbine test facility for assessing the effects of stress and temperature on the durability of materials for microturbine components

- Resonant ultrasound spectrometer for characterization of mechanical integrity and elastic properties of materials and components
- Electromechanical and micromechanical universal test machines for composites and their constituents
- Mechanical properties microprobe for measuring contact stiffness, elastic and plastic properties, and fracture toughness of thin films and small samples
- Interfacial test system for evaluation of composite interfacial properties by means of single-fiber push-in
- Instrumented indenters
- Workstation for life-prediction analysis (FEA + ERICA/CERAMIC + CARES)

MCAUC Staff News

MCAUC Initiates Renovation

MCAUC staff initiated a process that will lead to the renovation and consolidation of its facilities. This move was prompted by the need to optimize laboratory floor space and equipment usage. Associated with the renovation process is a donation program that will result in the transfer of more than 60 items (mechanical testing machines, furnaces, and other accessories) to Bradley University (Peoria, Ill.); Purdue University (West Lafayette, Ind.); Tennessee Technological University (Cookeville, Tenn.); Alfred University (Alfred, N.Y.); the University of Tennessee, Knoxville; the University of Alabama-Birmingham; and the University of Texas-Brownsville. This renovation process was made possible thanks to the financial support of program managers Mike Karnitz, Ray Johnson, and Dave Stinton. Other program managers have also pledged additional support to complete this effort.



US006322889B1

(12) **United States Patent**
Lara-Curzio et al.

(10) **Patent No.:** **US 6,322,889 B1**
(45) **Date of Patent:** **Nov. 27, 2001**

(54) **OXIDATION-RESISTANT INTERFACIAL COATING FOR FIBER-REINFORCED CERAMIC**

(75) Inventors: **Edgar Lara-Curzio; Karren L. More; Woo Y. Lee**, all of Knoxville, TN (US)

(73) Assignee: **The United States of America as represented by the United States Department of Energy**, Washington, DC (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **09/296,286**

(22) Filed: **Apr. 22, 1999**

Related U.S. Application Data

(60) Provisional application No. 60/082,762, filed on Apr. 23, 1998.

(51) **Int. Cl.**^{7} **D02G 3/00**
(52) **U.S. Cl.** **428/378**; 428/380; 428/384; 428/389; 501/95.1; 501/95.2
(58) **Field of Search** 428/370, 378, 428/380, 384, 387, 389, 293.4; 501/94, 95.1, 95.2

(56) **References Cited**

U.S. PATENT DOCUMENTS

5,567,518 * 10/1996 Pejryd et al. 428/378
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* cited by examiner

Primary Examiner—Deborah Jones
Assistant Examiner—Jason de la Pena

(57) **ABSTRACT**

A ceramic-matrix composite having a multilayered interfacial coating adapted to protect the reinforcing fibers from long-term oxidation, while allowing these to bridge the wake of advancing cracks in the matrix, is provided by selectively mismatching materials within adjacent layers of the interfacial coating, the materials having different coefficients of thermal expansion so that a low toughness interface region is created to promote crack deflection either within an interior layer of the mismatched interfacial coating or between adjacent layers of the mismatched interfacial coating.

13 Claims, 8 Drawing Sheets

Invention by Lara-Curzio and Co-Workers Receives Patent

Patent 6,322,889, “Oxidation-Resistant Interfacial Coating for Fiber-Reinforced Ceramic,” based on an invention by ORNL researchers Edgar Lara-Curzio, Karren L. More, and Woo Y. Lee, was issued on November 27, 2001. The patent describes a multilayered, multifunctional fiber coating for ceramic matrix composites capable of protecting the fibers from environmental attack while allowing them to deflect and bridge matrix cracks.

Lara-Curzio Receives Award of Merit and Becomes a Fellow of ASTM

Fig. 3-1. Lara-Curzio received his award from ASTM C28 chairman M. G. Jenkins.

On January 18, 2001, Edgar Lara-Curzio received the 2001 Award of Merit and the honorary title of Fellow of the American Society for Testing and Materials (ASTM) (Fig. 3-1). He was cited for "technical expertise, outstanding leadership, and distinguished service in committee C28 Advanced Ceramics demonstrated by his tireless commitment to the technical excellence and introduction of new test methods for ceramics and ceramic matrix composites." Lara-Curzio, who is one of the youngest recipients of this award, is currently serving as chairman of ASTM subcommittee C28.07 on Ceramic Matrix Composites, and as U.S. representative and convener, through the American National Standards Institute (ANSI) and ASTM, of Working Groups 20 and 21, Technical Committee 206 on Advanced Ceramics, of the International Standards Organization (ISO).

Lara-Curzio Appointed Associate Editor of the Journal of the American Ceramic Society

In February 2001 Edgar Lara-Curzio was appointed associate editor of the *Journal of the American Ceramic Society*.

Lara-Curzio Receives Certificate from the American Ceramic Society Engineering Ceramics Division

Edgar Lara-Curzio received a certificate from the Engineering Ceramics Division of the American Ceramic Society in recognition for having served as co-organizer of the Symposium on Mechanical Behavior/Design of Engineering Ceramics & Composites as part of the 25th Annual International Conference on Advanced Ceramics & Composites held in Cocoa Beach, Florida, January 21–26, 2001.

Selected Highlights

Mechanical Evaluation of Metallic Foams—Porvair Advanced Materials, Hendersonville, N.C.

Industrial Collaborator: S. Wagner
HTML: E. Lara-Curzio, R. Parten, and C. Stevens

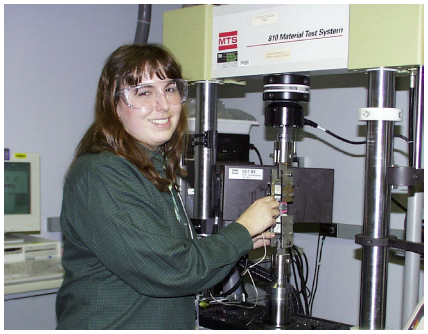

Fig. 3-2. Porvair engineer Stacia Wagner.

Metallic foams are currently being considered for fuel cells, catalytic converters, filters, and many other applications. To validate the structural integrity of components designed and fabricated with these materials, it is necessary to evaluate their mechanical properties. However, because currently there are no standardized test methods for the evaluation of metallic foams, Porvair Advanced Materials (Hendersonville, N.C.) sought the expertise of ORNL researchers to evaluate their mechanical properties. Porvair Engineer Stacia Wagner (Fig. 3-2) visited the HTML to work with Edgar

Lara-Curzio, Randy Parten, and Chris Stevens to determine the effect of temperature and foam porosity (3, 20, and 40 pores per linear inch) on the tensile and compressive properties of foams of 316 stainless steel, copper, and FeCrAlY. By adapting techniques originally developed for the manufacture of ceramic foams, Porvair can synthesize a wide variety of metallic foams (platinum, copper, brass, 316 stainless steel, nickel, silver, cobalt, rhodium, titanium, nichrome, Hastelloy X, and Inconel) that can be braised and/or sintered into more complex components.

Mechanical Evaluation of Chemical-Vapor-Deposited SiC Foams—Ultramet, Pacoima, Calif.

Industrial Collaborator: E. Stankiewicz
HTML: E. Lara-Curzio, T. Kirkland, and L. Riester

The development of chemical-vapor-deposited SiC foams is being driven by their potential use as catalytic substrates for pollution control devices, igniters for rocket engines, and advanced filters for hot gases and molten aluminum. In addition, when coated with a biocompatible material (e.g., niobium or tantalum), SiC foams can be used for structural in-body implants to promote the growth of bone and other tissue. These materials are synthesized by using high-deposition-rate (100–400 μm/h) chemical-vapor infiltration (CVI) of an extremely porous vitreous carbon body with SiC (Fig. 3-3).

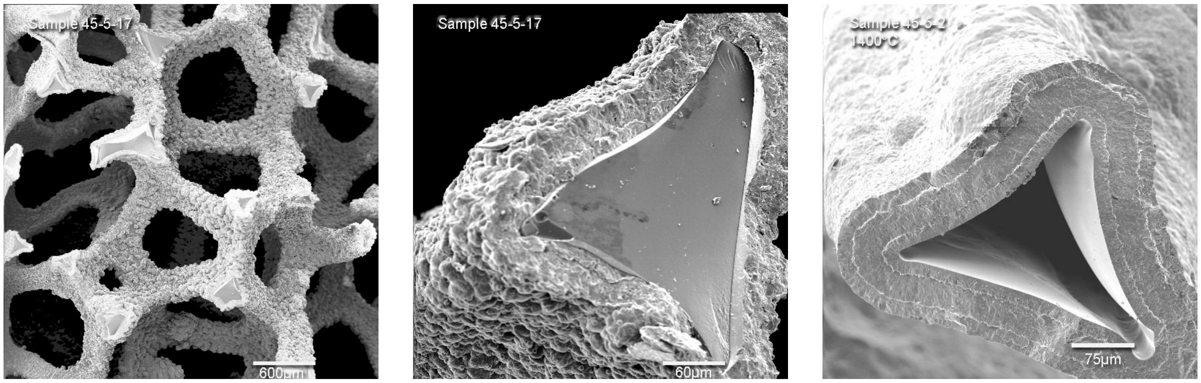

Fig. 3-3. Scanning electron micrographs of CVI-SiC foams after mechanical evaluation at ambient and elevated temperatures.

The resulting structures are thermally stable, low in density, and chemically pure; they have low thermal expansion, resist thermal shock, and are relatively inexpensive. Dr. Edwin Stankiewicz of Ultramet (Pacoima, Calif.) worked with Edgar Lara-Curzio, Tim Kirkland, and Laura Riester on the mechanical characterization of these materials. Specifically, this user project focused on assessing the effect of temperature (20–1400°C) and number of pores per inch (45 vs 60) of the foams, their strength, and their fracture toughness (Fig. 3-4).

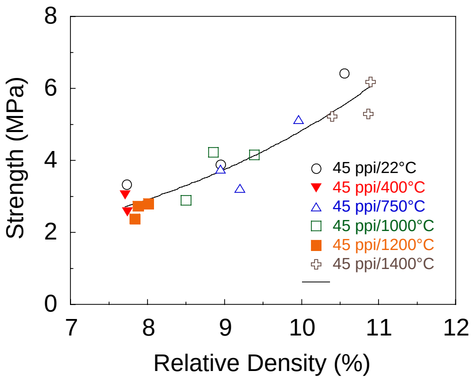

Fig. 3-4. Diametral tensile strength of SiC foams as a function of temperature and relative density.

Creep Behavior of Magnesium Alloys—Noranda Inc., Toronto, Ontario, Canada

Industrial Collaborators: E. Baril and E. Landriault
HTML: K. Liu, E. Lara-Curzio, and L. Riester

Noranda is working on the development of a new generation of creep-resistant magnesium alloys that could be used for the manufacture of drivetrain components and therefore could contribute to reducing the weight of vehicles. Noranda researchers Eric Baril and Emmanuelle Landriault (Fig. 3-5) are collaborating with Ken Liu, Edgar Lara-Curzio, and Laura Riester to study the mechanisms responsible for the creep deformation of magnesium alloys and its relationship to their microstructural evolution. Specifically, two magnesium alloys are being studied: Mg-Al-Zn (AZ91), a well-characterized alloy system for die-casting applications, and Mg-Al-Sr, a new family of elevated-temperature creep-resistant alloys. The analysis of recent creep tests results carried out at the HTML revealed that the family of Mg-Al-Sr alloys is at least two orders of magnitude more creep-resistant than AZ91. The mechanical properties of individual grains of the Mg-Al-Sr alloy samples were determined with a nanoindenter so that the role of Sr in improving creep resistance could be further understood (Fig. 3-6).

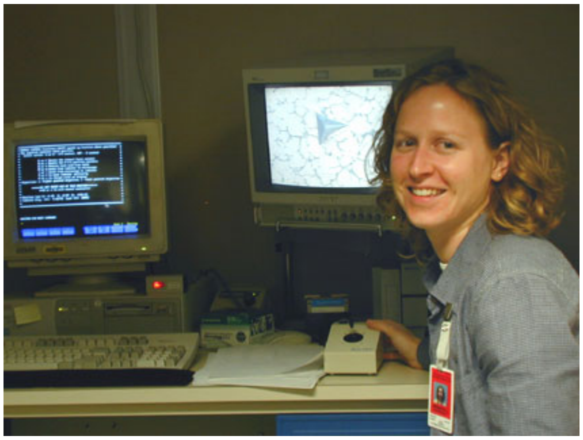

Fig. 3-5. Noranda researcher Emmanuelle Landriault.

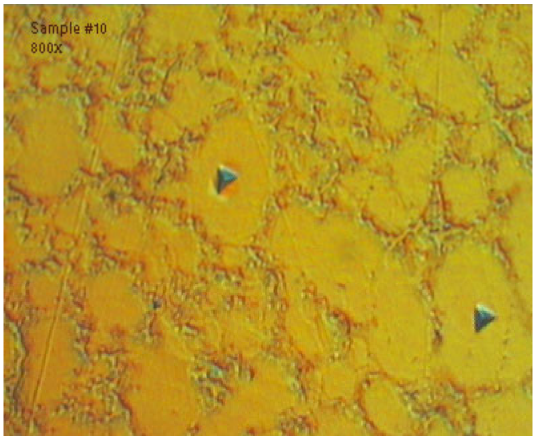

Fig. 3-6. Nanoindents inside individual grains of Mg-Al-Sr alloy that had failed during creep testing.

Although the results from nanoidentation tests revealed a significant amount of variability, preliminary analyses indicate that the hardness was highest for samples that had failed during the creep test. These results are consistent with the development of dislocations in this material during creep and will be coupled with microstructural information obtained by means of transmission electron microscopy (TEM).

Stresses in Ceramic Matrix Composite Combustor Liners—the Siemens Westinghouse Science and Technology Center, Pittsburgh, Pa.

Industrial Collaborator: E. Carelli
HTML: E. Lara-Curzio and R. Parten

Mr. Eric Carelli, an engineer with Siemens Westinghouse Science and Technology Center, worked with Edgar Lara-Curzio and Randy Parten (Fig. 3-7) to determine the state of residual stresses in continuous-fiber-reinforced oxide-oxide ceramic-matrix composite

Fig. 3-7. Siemens Westinghouse engineer Eric Carelli and Randy Parten.

combustor liners. In addition, using a push-out test, they determined the bond strength between the combustor liner and a thermal barrier coating developed at Siemens-Westinghouse that is being used to extend the range of service temperatures of the liners. The techniques used for these tests, which were developed by the HTML researchers, are providing important information for the optimization of these components.

Mechanical Evaluation of Neodymium-Iron-Boron Permanent Magnets—Magnequench Inc. and North Carolina State University

Industrial Collaborator: B.-M. Ma
University Collaborators: A. Shi and M. Garrell
HTML: E. Lara-Curzio and C. Stevens

Magnequench Inc. has developed a process to produce net-shape bonded isotropic neodymium-iron-boron (NdFeB) materials with isotropic magnetic properties by injection molding slurries of rapidly solidified powders and organic binders (Fig. 3-8). Considering that more than 150 magnets are used in a typical automobile, the permanent magnets being developed by Magnequench are attractive for automotive applications because of their large magnetic-strength-to-weight ratio. As part of a user project, Dr. Bao-Min Ma, director of research and technology for Magnequench Inc., and North Carolina State University graduate student Monika Garrell (Fig. 3-9) worked with Edgar Lara-Curzio and Chris Stevens to quantify the effect of binder type and to test the effects of temperature (between –40°C and 180°C) and magnetic-particle concentration on the mechanical properties of these materials.

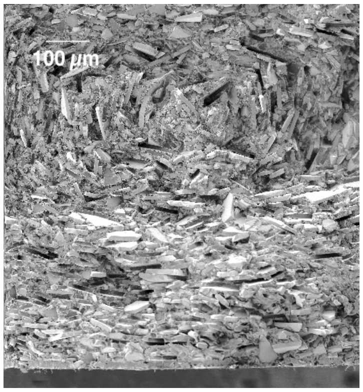

Fig. 3-8. Scanning electron micrograph of fracture surface of NdFeB/nylon material.

Fig. 3-9. N.C. State graduate student Monika Garrell.

Mechanical Properties of Laser-Synthesized Iron Oxide Coatings on Aluminum Alloys—University of Tennessee Space Institute

University Collaborators: N. B. Dahotre and S. Nayak
HTML: L. Riester

Dr. Narendra B. Dahotre and graduate student S. Nayak (Fig. 3-10) worked with Laura Riester to evaluate the mechanical properties of laser-synthesized iron oxide coatings on Al319, a hypereutectoid Al-Si alloy (Fig. 3-11). The purpose of these coatings is to improve the wear and corrosion resistance of this alloy, particularly if it is to be used for cylinders in an engine block. Dr. Dahotre, who is an associate professor of materials science and engineering at the University of Tennessee Space Institute (Tullahoma, Tenn.), developed the method for processing these coatings with NdYAG and CO_2 lasers. The objective of their HTML user project is to use the nanoindenter to determine the mechanical properties (stiffness, hardness, and fracture toughness) of the coatings.

Fig. 3-10. UT graduate student S. Nayak.

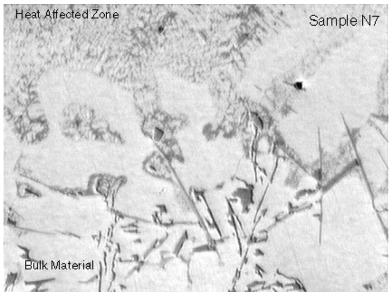

Fig. 3-11. FeO-coated Al 1319 alloy.

Creep Behavior of Si_3N_4/SiC Nanocomposites—University of California-Davis

University Collaborators: A. Mukherjee and M. Gasch
HTML: K. Liu, E. Lara-Curzio, and L. Riester

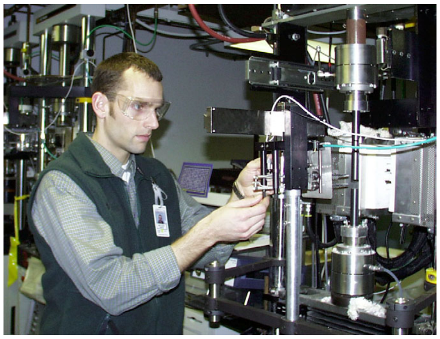

Fig. 3-12. UC-Davis graduate student Gasch.

Professor Amiya Mukherjee and graduate student Matt Gasch (Fig. 3-12) of the University of California-Davis are working with Ken Liu, Edgar Lara-Curzio, and Laura Riester to study the compressive creep behavior of silicon nitride nanocomposites. Professor Mukherjee and his team have recently consolidated dense nanostructured Si_3N_4/SiC ceramic composites following two routes: from amorphous polymer precursors by using electric-field-assisted sintering (EFAS) with oxide additives and by mixing the polymer powder with the liquid polymer as binder followed by cold isostatic pressing and heat treatment in a furnace to crystallization. TEM work performed at UC-Davis showed that the amorphous material crystallized into a fine-grained (300–500-nm) matrix of Si_3N_4 grains with nanocrystalline (100-nm) SiC particles at grain boundaries and within Si_3N_4 grains. The addition of $MgAl_2O_4$ as a sintering aid resulted in the formation of an intergranular amorphous oxide phase as expected. However, high-resolution TEM demonstrated that, as a result of the rapid consolidation possible with EFAS and in situ crystallization of the amorphous phase, several grain boundaries are partially or completely free of oxide grain-boundary phases. It is expected that microstructures of this type will show increased creep resistance. Currently, long-term creep tests are being conducted at ORNL on materials synthesized by the two different routes, and a nanoindenter was used to evaluate samples with various amounts of sintering additives (8%, 5%, 3%, 1%, and 0%). Significant differences in the value of the elastic modulus and hardness were found among the specimens evaluated (Fig. 3-13). These differences can be explained from the differences in their microstructure, which has been characterized by high-resolution electron microscopy and which has a significant effect on the mechanical behavior of the material at elevated temperatures.

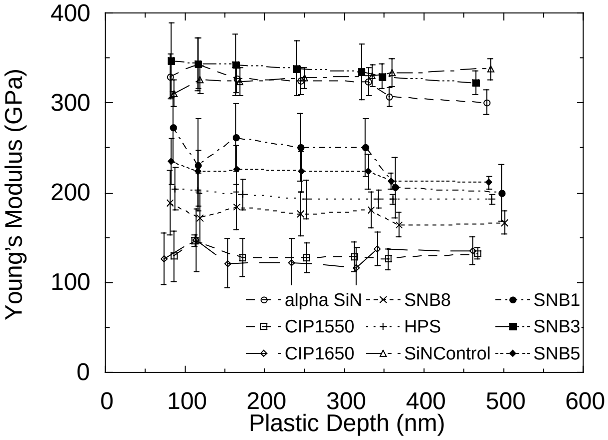

Fig. 3-13. Plastic depth compared with Young's modulus of Si_3N_4/SiC nanocomposites.

Georgia Tech Researchers Use Resonant Ultrasonic Techniques to Determine the Temperature Dependence of the Elastic Properties of TiAl—Georgia Institute of Technology

University Collaborators: T. R. Kurfess and W. Stone
HTML: E. Lara-Curzio and L. Riester
ORNL Researchers: M. K. Ferber and R. Carneim

Professor Thomas R. Kurfess and graduate student Wesley L. Stone (Fig. 3-14), of the School of Mechanical Engineering at the Georgia Institute of Technology in Atlanta, are developing analytical models to simulate the temperature distribution in materials during grinding. In particular, they are interested using cubic boron nitride to grind TiAl. TiAl is an intermetallic compound that has potential for high-temperature applications in the automotive and aerospace industries. To support their modeling efforts, the researchers need to know the thermal dependence of the elastic constants, hardness, specific heat, thermal expansion, and thermal diffusivity of these materials.

Fig. 3-14. Georgia Tech graduate student Wesley Stone.

Professor Kurfess and Mr. Stone are working with Edgar Lara-Curzio and ORNL researchers M. K. Ferber and Robert Carnhaim to determine the elastic constants between ambient temperature and 800°C using resonant ultrasound spectroscopy (RUS). RUS is based on the principle that mechanical resonances of a solid are dependent upon geometry, density, crystalline symmetry, and elastic constants. As part of this effort, graduate student Wesley Stone initiated a small separate study with Laura Riester to compare the values of the elastic properties obtained from RUS and those obtained during the unloading segment of nanoindentation tests. It was found that the value of Young's modulus measured by RUS agreed well with that calculated from nanoindentation tests (160 GPa).

MCAUC Supports Nation's Defense Programs—Westinghouse Savannah River Company

Industrial Collaborator: P. Korinko
HTML: L. Riester

Fig. 3-15. WSRC engineer Dr. Paul Korinko.

While production of new tritium will not be necessary for many years, recycling of tritium to keep the nation's supply of nuclear weapons ready is one of the missions of the Savannah River Site (SRS), managed by Westinghouse Savannah River Company (WSRC). WSRC engineer Paul Korinko (Fig. 3-15) is working with Laura Riester to assess microstructural changes and to determine the magnitude of residual stresses in welded stainless steel tubes used to store tritium. These tubes are sealed with a resistance pinch weld and are subsequently cut above the weld. After cutting, there is often some surface contamination that must be removed to ensure safe handling. This contamination is currently removed using a time-consuming and costly processing step in which heated air impinges on the surface and drives the contamination away. In an attempt to eliminate this decontamination step, engineers at WSRC have developed various processes that involve cutting the stem within the weld to reduce the effects of the contamination. The influence of the cutting process on the state of residual stresses in the pinch weld must be determined before the existing welding and cutting processes can be changed. Preliminary results of conventional microhardess testing (Knoop) and nanoindentation techniques with both spherical and Berkovich indenters have revealed differences in the hardness of the material around the weld. The results from this investigation will allow WSRC personnel to determine the feasibility of the alternative processes.

4. DIFFRACTION USER CENTER (DUC)

Group Members
Camden R. Hubbard, Group Leader
Geneva N. Worley, Secretary

Jianming Bai
E. Andrew Payzant
Roberta Peascoe
Claudia J. Rawn
Thomas R. Watkins

The Diffraction User Center (DUC) uses room- and high-temperature X-ray, synchrotron, and neutron diffraction methods to characterize crystalline phases and the stability of ceramics, alloys, catalysts, and other industrially relevant materials. The data, obtained under controlled environments as a function of temperature, are used to relate materials processing and performance with phase transformations, reactions (solid-solid, liquid-solid, and gas-solid), lattice expansion, atomic structure, crystallization from the melt, and phase stability.

In addition to supporting the diffraction needs of users from academia, industry, and DOE laboratories, the diffraction facilities are used extensively by qualified staff members in ORNL's Metals and Ceramics Division, who conduct a wide variety of ceramic and alloy research and development efforts sponsored by DOE and other federal agencies. DUC staff members also lead several DOE- and ORNL-funded projects.

The DUC continues to be home to a large number of user projects representing a broad range of materials science and characterization. Accomplishments in FY 2001 include the following:

- 3 "best poster" awards for projects undertaken at DUC,
- 18 new user proposals,
- more than 20 papers co-authored by DUC staff,
- an invited presentation by C. J. Rawn at the American Crystallographic Association 2001 meeting, and
- election of a DUC staff member, C.R. Hubbard, to the International Centre for Diffraction Data Board of Directors.

DUC staff members are principal investigators on two Laboratory Directed Research and Development (LDRD) projects involving real-time, in situ studies using X-ray, synchrotron, and (particularly) neutron facilities. These projects will lead to enhanced facilities and expertise at HTML and will lay a foundation for cooperative future use of the upgraded instruments at the High Flux Isotope Reactor (HFIR) and the revolutionary Spallation Neutron Source (SNS).

During this fiscal year, Philips Analytical Instruments completed the installation of an X'Pert Pro Diffractometer in the DUC. The consignment agreement provides HTML a top-of-the-line

Philips X-ray diffraction system capable of high-temperature X-ray diffraction (HTXRD) and high-temperature residual stress measurements while providing Philips with input from the experience of HTML staff and users. The instrument, valued at $345,000, includes interchangeable X-ray optics and a high-temperature furnace with sample spinner.

DUC Instruments

- Neutron powder diffractometer (high-temperature)
- Philips X'Pert Pro X-ray Diffractometer (high-temperature, controlled environment)
- Scintag PADV X-ray Diffractometer
- Scintag PADX X-ray Diffractometer (high-temperature, controlled environment)
- Scintag XDS2000 X-ray Diffractometer
- Synchrotron X-ray beamline X-14A (low- and high-temperature)

Selected Highlights

Ordering in Nanocrystalline Spinel Structures—Georgia Institute of Technology

University Collaborators: A. Wilkinson and A. Samia
HTML: J. Bai and C.J. Rawn

Researchers at Georgia Tech have been studying the relationship between synthesis conditions, particle size, and magnetic properties for a range of $Ni_{1-x}Fe_{2+x}O_4$ spinel compositions (Fig. 4-1). To fully interpret the properties of the nanoparticles synthesized with various heat treatments, the degree of inversion (interchange of iron and nickel) in the spinel must be determined. Since the contrast between Ni and Fe is negligible for both neutrons and nonresonant X rays, Prof. Wilkinson and Ms. Samia conducted resonant scattering experiments at HTML's synchrotron beamline X14A with Dr. Jianming Bai and Dr. Claudia Rawn of the DUC. To enhance the scattering contrast of the iron and nickel, data sets were collected near the iron and nickel K-edges, while a third high-energy data set was collected for each sample at approximately 17 keV to define a baseline of structural parameters. Currently, the data are being processed using Rietveld or whole-pattern-fitting structural refinement techniques, through which a powder pattern is calculated from a structural model and is compared to the data. Structural information such as the atomic positions and site occupations will be used to determine the degree of inversion.

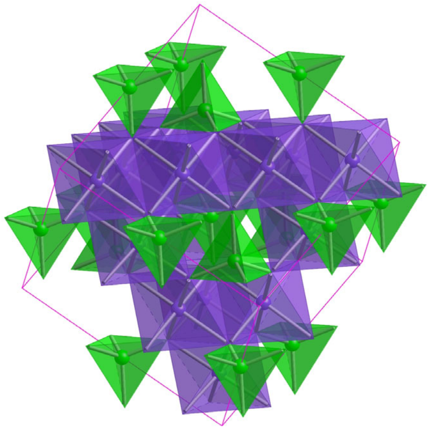

Fig. 4-1. The structure of $Ni_{1-x}Fe_{2+x}O_4$ spinel, in which the nickel atoms are only found in the octahedral (purple) sites, and the iron is divided between the tetrahedral (green) and octahedral sites.

Metal Hydrides for Hydrogen (Tritium) Storage—University of Nevada, Reno

University Collaborators: D. Chandra, J. Smith, and M. Coleman
HTML: E. A. Payzant

Researchers from the Metallurgical Engineering Department at the University of Nevada, Reno, visited the DUC to identify phase equilibria in zirconium iron alloy (SAES St198). This alloy consists mainly of Zr_2Fe, with some Fe_2Zr and zirconium. These intermetallic phases have important applications for the gettering of hydrogen and tritium. However, the phase equilibria under different atmospheres had not been fully evaluated or understood. X-ray diffraction (XRD) studies were undertaken using HTML's unique controlled-atmosphere HTXRD, which enabled comparative studies under nitrogen, hydrogen, and inert atmospheres. Preliminary results under nitrogen and hydrogen environments indicate that stable zirconium iron forms nitrides above 700°C. The Zr_2Fe phase was actually a supercooled phase, and the equilibrium Zr_3Fe phase was obtained after heating to 600°C. Heating the supercooled Zr_2Fe phase in a hydrogen environment showed that the Zr_3FeH_x phase formed. $Zr_3FeH_{5.6}$ remained stable between 300 and 400°C in pure hydrogen (Fig. 4-2). Experiments in 96% N_2-4% H_2 gas mixtures showed that the $Zr_3FeH_{5.6}$ disproportionates to ZrN at approximately 700°C. Thermal aging of $Zr_3FeH_{5.6}$ in 96% N_2-4% H_2 showed disproportionation to ZrN and α–Fe and other phases yet to be determined.

ORNL 2002-02835/gss

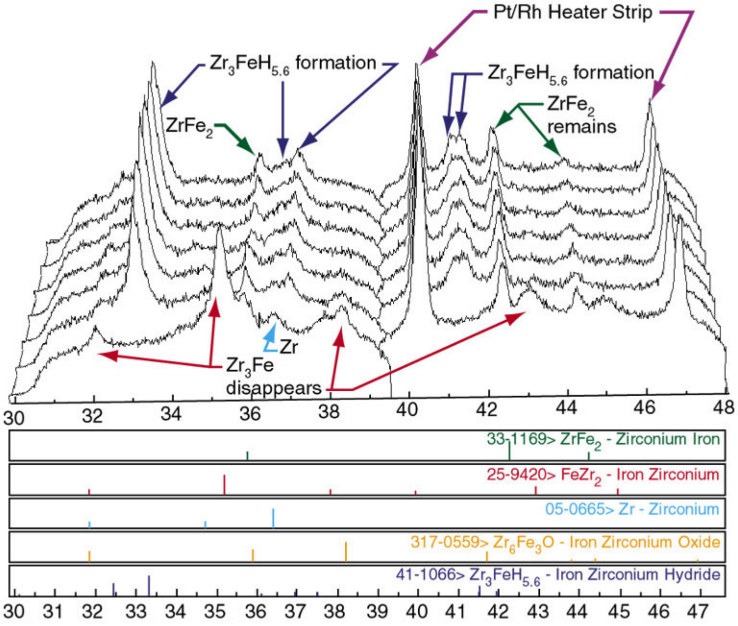

Fig. 4-2. The formation of zirconium iron hydride in alloy St198 observed by in situ HTXRD in H_2 atmosphere at 300°C.

The HTML shared in two "best poster" awards for presentations describing user projects completed with the University of Nevada, Reno. The winning posters were "High Temperature X-Ray Diffraction Studies during Hydriding of Zr_2Fe," by J. Smith, D. Chandra, J. R. Wermer, and E. A. Payzant; and "Solid State Phase Transitions of NH_4NO_3-KNO_3 Binary System," by W.-M. Chien, D. Chandra, J. Smith, C. J. Rawn, and A. K. Helmy.

Crystallite Size Analysis of Catalysts—Cummins Engine Co.

Industrial Collaborator: R. England
HTML: T. R. Watkins

Catalytic activity is to a large extent controlled by the surface area of the active components. After extended operation at high temperatures, the surface area of some of these components decreases, reducing the activity of the catalyst. Transition frequencies and bandwidths of Raman bands of support materials are sensitive to the surface area of these active components. One percent doping of platinum can significantly alter the Raman bands of the substrate. The band broadening is an indication of the relaxation process with surrounding lattice modes; the correlation length is a function of the crystallite size of the platinum Raman vibrational spectroscopy that has been used to characterize surface-adsorbing species.

In this study, the goal was to test the correlation of the Raman response with a physical measurement of the catalyst's crystal size in the bulk material. Calibration of the Raman response using XRD and transmission electron microscopy (TEM) will permit Cummins Engine to use the Raman technique as a particle-size probe to delineate catalyst degradation pathways that are critical in developing a fundamental understanding of aging mechanisms in NO_x/SO_x absorbers. The new Philips X'Pert Pro MPD Diffractometer was used to scan the (200) platinum peak on titania substrates, which were corrected for instrumental broadening by the (220) reflection of silicon. Good correlation was observed between the XRD and Raman techniques for specimens containing 3.62 wt % platinum on titania (Fig. 4-3). Raman data were also obtained for 1.66 wt % platinum. TEM results for one specimen confirmed the XRD crystallite sizes.

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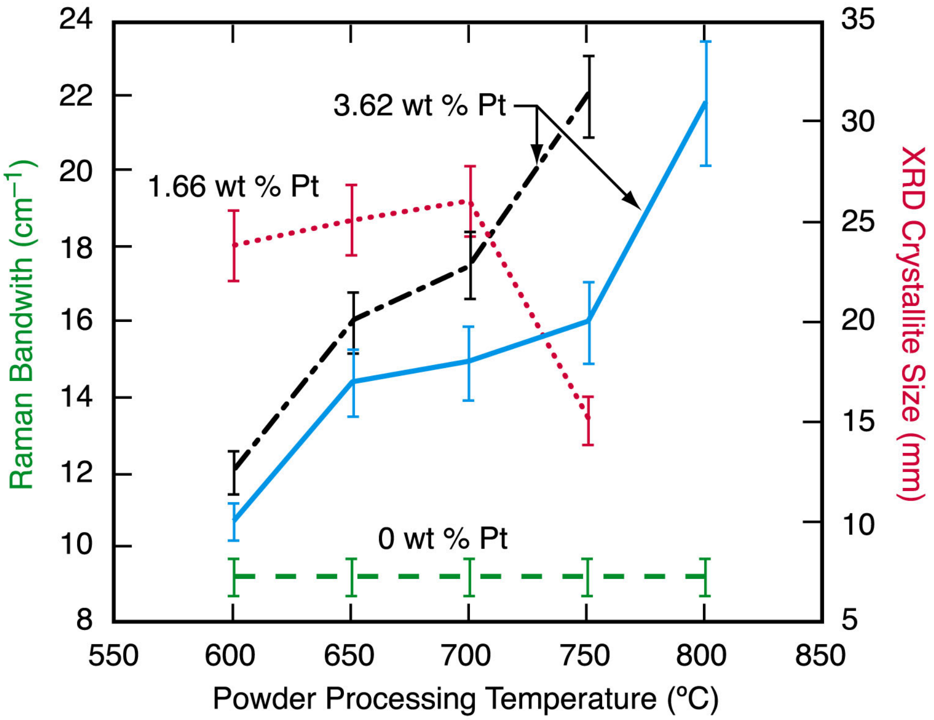


Fig. 4-3. Correlation of Raman (dashed lines) and XRD crystallite size (solid line) data.

Low-Temperature Structural Transformations—Solid State Division, ORNL

Laboratory Collaborators: L. Boatner and M. Farmer
HTML: J. Bai and C. J. Rawn

Matt Farmer, a graduate student at Baylor University, working at ORNL under the direction of Lynn Boatner of the Solid State Division, is studying alkali lanthanide double phosphates for radiation detection applications. Current research has focused on $K_3Lu(PO_4)_2$ (Fig. 4-4). Previous characterization with single-crystal XRD and neutron powder diffraction has provided evidence of low-temperature phase transitions. With the HFIR currently down for upgrades, low-temperature capabilities for studying structural information of powders is limited; however, the HTML beamline at the National Synchrotron Light Source offers such capabilities. Data were collected at 293, 230, 200, 100 and 30 K, and the low-temperature polymorphs have been confirmed. Rietveld refinements are being used to understand these low-temperature structures. A poster entitled "Polymorphism, Phase Transitions, and Thermal Expansion of $K_3Lu(PO_4)_2$," by J. Matt Farmer, Lynn Boatner, Bryan Chakoumakos, Claudia Rawn, and Jeff C. Bryan, received both the Pauling Poster Prize and the 2001 Oxford Poster Prize at the Annual ACA meeting in Los Angeles in July 2001. This is especially significant in that it is the first time that both awards have been given for the same poster.

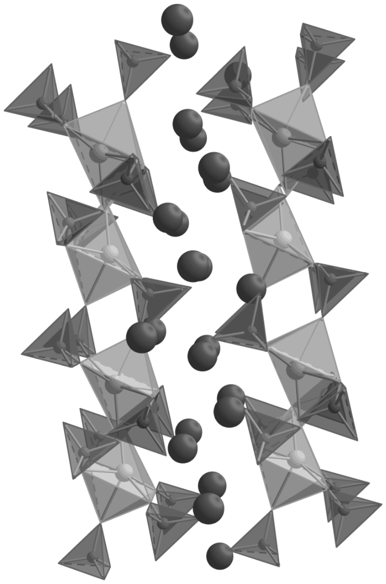


Fig. 4-4. The crystal structure of potassium lutetium phosphate.

Thermal Expansion Anisotropy—Metals and Ceramics Division, ORNL

Laboratory Collaborator: J. Schneibel
HTML: C. J. Rawn, T. R. Watkins, and E. A. Payzant

Critical issues for Mo_5Si_3, an ultrahigh-temperature material, include understanding the structure and the reduction of high anisotropy in the coefficient of thermal expansion (CTE). Dr. J. Schneibel, of ORNL, is studying these alloys under Office of Industrial Technologies (OIT) and Basic Energy Sciences (BES) funding using HTML's new Philips X'Pert Pro X-ray diffractometer system. This system provides parallel beam optics and a reduced temperature gradient in the furnace that has improved experimental precision dramatically over previous measurements.

In Mo_5Si_3 the CTE along the *c*-direction is more than twice that in the *a*-direction. The anisotropy is due to an elastically more rigid basal plane and a higher anharmonicity along the *c*-axis. The higher anharmonicity along the *c*-axis is attributed to the existence of [001] Mo chains in the $D8_m$ structure of Mo_5Si_3. As these chain structures were either modified by alloying or eliminated by structural modification (from $D8_m$ to $D8_l$), the researchers found significant changes in the CTE anisotropy. Additions of niobium up to approximately 40 at. % reduced the anisotropy (Fig. 4-5). At higher niobium concentrations, the CTE increased until the structure changed to that of Nb_5Si_3 and the CTE anisotropy decreased. These findings have been reported in papers submitted to *Intermetallics* and *Physical Review B*.

ORNL 2002-01851/gss

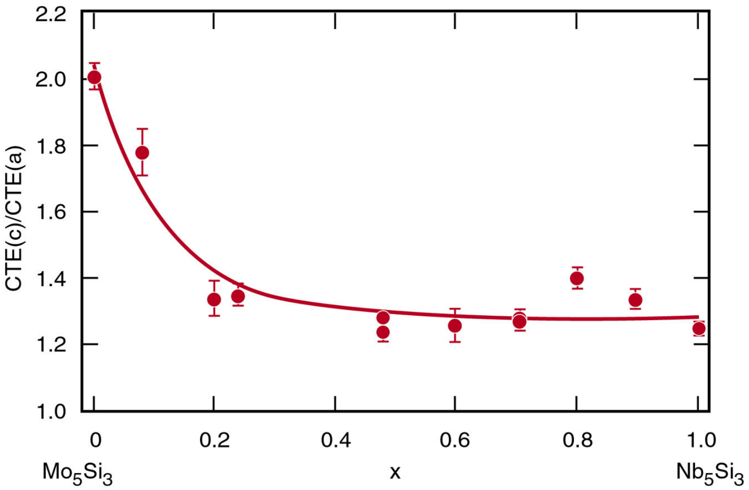


Fig. 4-5. The thermal expansion anisotropy of $(Mo_{1-x}Nb_x)_5Si_3$ is reduced with increasing Nb content.

Nitridation of Two-Phase Cr-Pt Alloys—University of Tennessee

University Collaborators: P. Liaw and S. Baham
ORNL Collaborators: M. P. Brady and D. A. Hoelzer
HTML: E. A. Payzant

A synthesis route based on internal oxidation reactions in Cr-Pt alloys was utilized for the controlled production of near-surface complex composite structures. The metal alloy microstructure was shown to act as a template for the nitrided composite microstructure. XRD analysis was performed on an as-cast nitrided coupon in which the surface Cr_2N layer was removed (Fig. 4-6). The diffraction pattern was indexed as a combination of Cr_3PtN, an unusual metal nitride perovskite, and Cr_2N, with trace amounts of Cr_2O_3 as a minor phase (likely present as impurity inclusions left over from the arc-casting step). Preliminary results have been reported in the *Journal of Materials Research*.

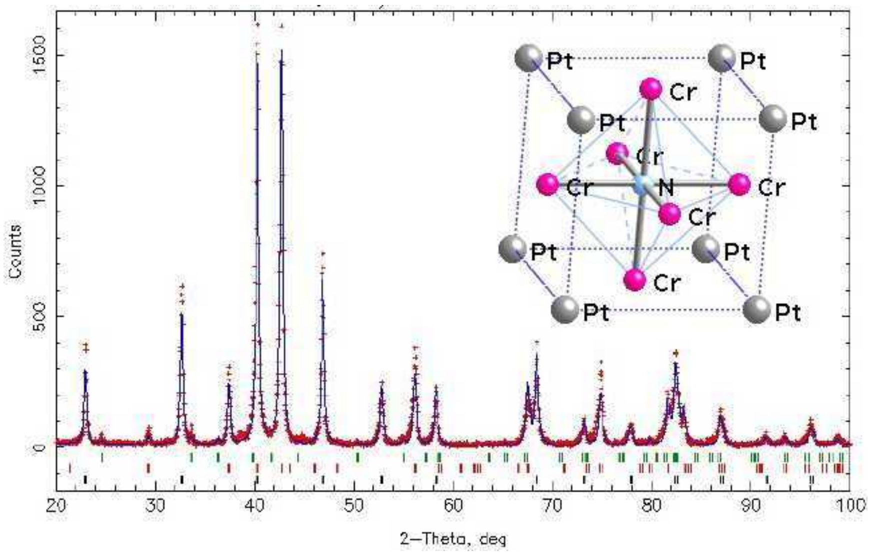


Fig. 4-6. Refined XRD pattern of a Cr_2N-Cr_3PtN composite. *Inset*: the crystal structure of the Cr_3PtN metal perovskite.

5. RESIDUAL STRESS USER CENTER (RSUC)

Group Members
Camden R. Hubbard, User Center Leader
Geneva N. Worley, Group Secretary

Jianming Bai
O. Burl Cavin
Tom Ely
Gerard M. Ludtka
E. Andrew Payzant
Roberta Peascoe
Stephen Spooner
David Q. Wang
Thomas R. Watkins

HTML user projects and DOE programs are increasingly concerned with life prediction and failure analysis of engineering structures and how to improve life via beneficial compressive stresses near the surface. In many cases, knowledge of residual stress gradients (sign and magnitude) as a function of location at both the surface and throughout the volume of a component is critical information for failure analysis and life prediction models. The Residual Stress User Center (RSUC) was established to meet this need and to provide a facility for research into controlling residual stresses, either through modifications in the forming, surface treating, and finishing processes; changes in the design; or stress-relief procedures.

RSUC provides three principal measurement capabilities at three locations: the HTML X-ray residual stress facilities; the ORNL synchrotron beamline X14A at the National Synchrotron Light Source; and the Neutron Residual Stress Mapping Facility (NRSF) at the High Flux Isotope Reactor (HFIR). Together, the three facilities, unique in themselves, make RSUC an unparalleled resource able to address a wide range of measurement needs of both industry and academia. These diffraction facilities are utilized to measure both macro (long-range) and micro (short-range) residual stresses in polycrystalline materials.

RSUC users also characterize nonrandom grain distribution, known as texture, in materials and relate the results to directionally dependent materials properties using the same facilities. Texture is very common in materials subjected to deformation and also in thin films and coatings, which are materials of increasing technological importance.

RSUC Instruments

- Powder-texture-stress (PTS) 4-axis goniometer with 18-kW rotating-anode X-ray generator
- PTS 4-axis goniometer with 2-kW X-ray tubes
- X-ray large-specimen stress analyzer
- Neutron Residual Stress Mapping Facility (NRSF), including remote access

- Neutron powder diffraction facility with high-temperature furnaces
- Synchrotron high-flux, highly parallel X-ray beam line X14A

New and Enhanced Instruments

The Technology for Energy Corporation's TEC X-ray diffraction large-specimen stress analyzer was heavily used in FY 2001 after becoming fully operational at the end of FY 2000. Besides supporting a number of user projects, this instrument is also used for several projects of DOE's Office of Energy Efficiency and Renewable Energy (EE/RE). Goals for FY 2002 are to further increase reliability and to fully automate the system to enable automated stress mapping of industrial-sized specimens. More detail is provided in the third highlight in the following section.

The NRSF began a major upgrade in July 2000, coinciding with the replacements of the beryllium reflector and the beam tube at HFIR. The HFIR was unavailable throughout the year, with operation scheduled to resume in early FY 2002. Following upgrades at the HB-2 beamline, the new NRSF will be installed toward the end of FY 2002. When completed, NRSF should provide an approximately tenfold improvement in measurement capability compared to the instrument available just two years ago. Upgrades to NRSF include a new monochromator system, multiple detectors, new goniometers with expanded capacity for large and small specimens, and enhanced automation. Finally, NRSF will be supported with remote collaboration tools.

New Staff

Dr. Gerry Ludtka joined the group staff in FY 2001. Dr. Ludtka has more than 25 years of experience in advanced manufacturing technology and R&D programs investigating aluminum, ferrous, titanium, nickel, copper, and uranium alloy behavior. These research efforts focused on studying the influence of processing variables and chemistry on microstructural evolution and how these impact subsequent mechanical behavior distortion and residual stress development. Dr. Ludtka has led and participated in multidisciplinary teams in the development of heat treatment distortion software that predicts microstructure evolution, distortion, and residual stress in ferrous alloys and weapons materials. A current project with the American Iron and Steel Institute (AISI) and 12 industrial partners is characterizing the phase transformation kinetics and dilations of ferrous alloys using high-speed quenching dilatometry in support of modeling endeavors that will predict residual stresses for heat treating, casting, welding, and forging applications. Also, this year he initiated a research project involving Cummins Engine Company to study the potential of magnetic processing for residual stress abatement and for tailoring the microstructural evolution and properties in ferromagnetic materials. In support of industrial residual stress modeling and manufacturing endeavors, Ludtka has been involved with a wide range of companies, including Caterpillar, Cummins, Dana, Deformation Control Technologies, Eaton, Ford, General Motors, Timken, and Torrington.

Selected Highlights

RSUC continues to address critical industrial and academic problems, typically using a combination of RSUC and other HTML facilities. Growth in the use of the unique flux, energy tunability, and parallel beam characteristics of synchrotron radiation continues. The following highlights were selected to display the scope of activities conducted in this user center and the growing use of a combination of the RSUC instruments to obtain a comprehensive mapping of stress.

Thermal Residual Stress in Directionally Solidified Alumina-Cubic Zirconia Eutectic—University of Kentucky

University Collaborators: E. Dickey and C. Frazer
HTML: A. Payzant

A directionally solidified eutectic rod of alumina-cubic zirconia (composition #184-2) was analyzed on the PTS/Tube XRD. The rod was mounted on a goniometer head for precise alignment of the growth direction with the *z*-axis of the goniometer. A 1.0-mm collimator was used to prevent the beam from missing the sample surface at lower angles. To characterize the crystallography, seven different pole figures were taken for the alumina phase and four for the stabilized zirconia phase. Additional pole figures were taken at smaller step sizes for greater detail. Twelve pole figures were taken on similar samples (#96-1, #97-1) of smaller dimensions to confirm their crystallographic similarities with the #184-2 sample. The alumina phase was shown to be essentially single-crystal in nature. Figure 5-1 shows the two grains, or twin-related variants, for the #184-2 rod.

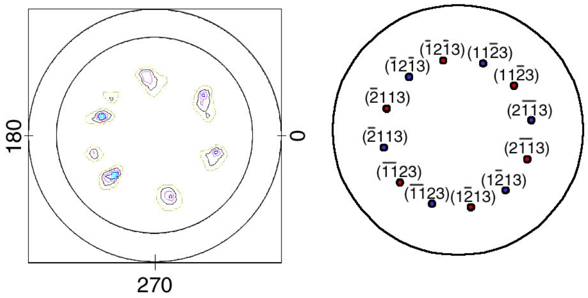


Fig. 5-1. Pole figure of (11•3) alumina (*left*) with stereographic projection (*right*).

The zirconia phase was originally thought to be highly textured with a random component, but further analysis revealed another possible explanation. The zirconia appears to maintain a consistent orientation relation with the alumina phase. The nominal orientation relationship between the phases is [001]Z || [2-1-10]A1 || [1-100]A2 with [110]Z || [0001]A || growth direction. As shown in Fig. 5-2, the (220) reflections can be rotated about the normal direction (along with the alumina twin) to account for the other points of intensity in the pole figure.

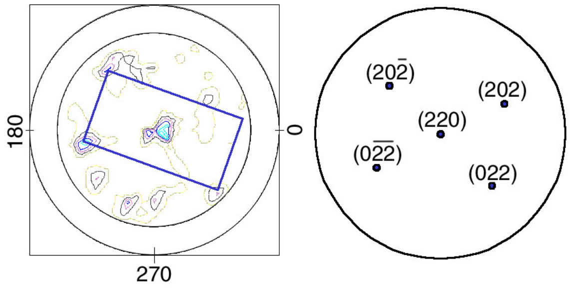


Fig. 5-2. Pole figure of (220) zirconia (*left*) with stereographic projection (*right*).

Texture analysis for the zirconia phase of #184-2 was done using popLA. The four pole figures taken were used to calculate a harmonic orientation distribution function (ODF). The ODF was then used to reconstruct the original pole figures to check the validity of the calculation. Finally, using an input stiffness tensor (Fig. 5-3), the polycrystalline stiffness tensor (Fig. 5-4), or weighted stiffness tensor, was produced using the Hill averaging

406	105	105	0	0	0
105	406	105	0	0	0
105	105	406	0	0	0
0	0	0	55	0	0
0	0	0	0	55	0
0	0	0	0	0	55

Fig. 5.3. Single-crystal stiffness tensor for zirconia. [R. P. Ingel and D. Lewis III, *J . Am. Ceram. Soc*. 71 (4), 265–71 (1998).]

322	147	147	–1	–1	2
147	321	148	1	0	–1
147	148	320	0	0	–1
–1	1	0	82	–1	0
–1	0	0	–1	81	–1
2	–1	–1	0	–1	81

Fig. 5.4. Weighted polycrystalline stiffness tensor for zirconia.

scheme. The quantification of the weighted stiffness tensor is vital in the calculation of thermal residual stress in the rod. The residual stress measurements will ultimately lead to improvements in the manufacturing process.

Neutron Diffraction Strain Tensor Measurements in Aluminum Friction-Stir Welds—University of South Carolina

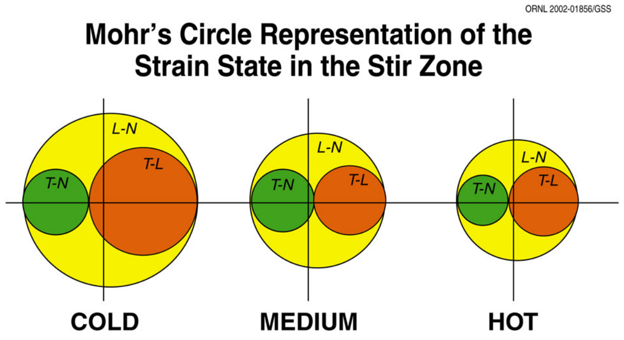
University Collaborator: M. Sutton
HTML: D. Q. Wang and S. Spooner

The friction-stir welding (FSW) method holds great promise for joining plates and bars of many alloys, including transportation-related aluminum alloys. In this project, researchers from the University of South Carolina have developed a model to predict the microstructure and residual stresses resulting from FSW joining of two aluminum 2024-T3 plates. Earlier measurements determined the residual stresses in three mutually perpendicular directions (longitudinal, transverse, and normal) relative to the direction of the FSW. However, more recent fracture studies and models of the FSW process indicated the possibility that the stress tensor's principal axes were not along these three primary directions. To investigate this hypothesis and to complete the strain mapping, the specimen was taken to the NRU reactor in Chalk River, Canada, for measurements, since HFIR is currently out of service while upgrades are being made.

FSW uses a high-rotary-speed stirring tool to heat and shear the material between the plates to form a seamless joint. Melting does not occur, and because the temperature rise is less than that in an arc weld, the resulting residual stresses are smaller. Because of the high-energy mechanical mixing action, it was anticipated that the stress state of the weld would be distinctly different from that of a conventional weld. Three welds differing in the energy input of the welding process were made at the University of South Carolina. An initial assessment of residual strains in one of the plates was completed before HFIR was shut down for renovations. Staff from HTML and Chalk River extended the prior measurements using the neutron strain mapping facilities at Chalk River, with emphasis on determining the orientation of the principal strain axes of the strain tensor.

The principal strain axes are orthogonal to one another and are usually related in a simple way to the geometry of the weld. The determination of the directions of the principal axes requires the measurement of strain in many directions—in this experiment, 11 directions. It was found that the principal axes for test locations within the stir zone were indeed simply related to the weld process, with the maximum longitudinal strain parallel to the weld line and a very compressive strain through the plate thickness. At the locations outside the stir zone, it was found that the principal strain axes were rotated up to 40° in the plane of the plate. The amount of principal axis rotation differed between the side of the weld in which the tool stirs into the material relative to the direction of the tool and the side in which the stir direction is opposite to the overall tool motion. The residual strains outside the stir zone are smaller than in the stir zone. These results are summarized in Figs. 5-5 and 5-6.

These neutron residual stress results are the first of their kind in FSW characterization and represent a powerful method of testing and validating residual stress calculations being made at the University of South Carolina. It is expected that the friction-stir methods can be optimized in this and other materials through the use of computation and neutron residual stress measurement. The measurements showed that there was indeed a tilt to the principal axes of stress that changed with distance from the edge of the FSW. X-ray surface stress measurements are also desired, and HTML staff have completed an assessment of the potential of successful measurements and have developed an experimental plan.



	T	*L*	*N*
COLD	−2.32	13.65	−11.86
MEDIUM	0.92	11.25	−8.55
HOT	−0.91	9.42	−8.37

Fig. 5-5. The strain state within the stir zone represented by a Mohr's Circle construction. The intersections of the circles with the horizontal line give the principal axis strains along the transverse (to the weld line), longitudinal (along the weld line), and normal or perpendicular (to the plate) directions. The vertical extreme in each of the circles gives the shear strain. It is apparent that the strains are largest for the cold stirring process and decrease as the process becomes hot.

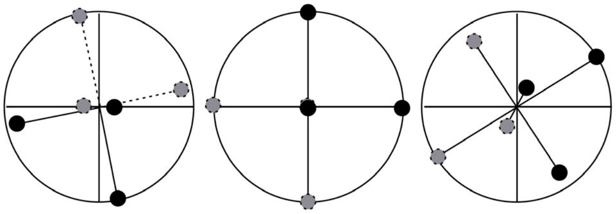


Fig. 5-6. The directions of the principal axes in base metal on the tool advancing side (*left*), the stir zone (*middle*), and the base metal (*right*) on the tool retreating side represented in stereogram plots. The dark circles are above the "equator" of the stereogram, and the gray circles are below. Note that the principal axes for the stir zone are simply related to the orthogonal axes of the weld geometry. The principal axes for the material just outside the stir zone are rotated away from the stir zone principal axes.

Large-Specimen X-Ray Stress-Mapping Gantry Facility Designed—University of South Carolina

University Collaborator: W. Bailey
HTML: T. R. Watkins and C. Hubbard

A new 1200-ft^2 laboratory module for the large-specimen X-ray residual stress-mapping facility and off-line alignment system is being refurbished with a new ceiling, cleaned and painted walls, and new floor tile. This laboratory will house the automated large-specimen X-ray residual stress-mapping facility that is under development and a planned second instrument for off-line alignment of neutron and X-ray strain-mapping specimens.

HTML User Program projects, using the large-specimen X-ray residual stress-mapping facility will be greatly enhanced via automation. This new overhead translation or gantry system (see Fig. 5-7) was designed by a student engineer from the University of South

Carolina and is presently being fabricated. The large-specimen system will enable automated X-ray residual stress mapping of actual components such as engine blocks. The expanded facility will include

- an X-ray enclosure;
- a gantry system consisting of an overhead translation system to which the existing TEC X-ray source and goniometer will be mounted;
- a *Z* motor for vertical movement of the TEC unit;
- two automated *XY* sample translation stages, one capable of mapping stresses in coffee-cup-sized parts and the other able to map stresses in parts the size of an engine block; and
- PC-based Labview software compatible with NRSF systems to interface between the translation stages and the preexisting data collection software.

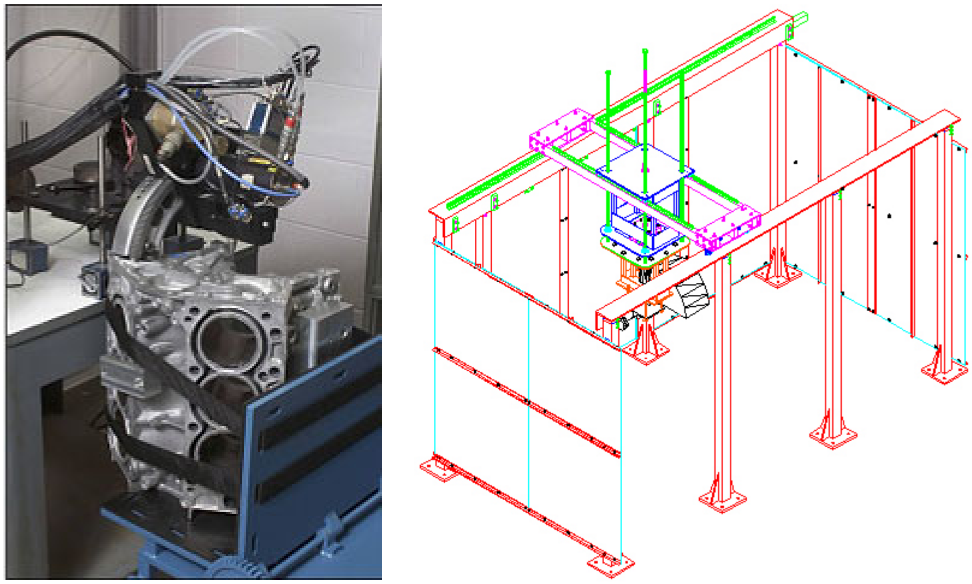

(A) (B)

Fig. 5-7. (A) The current large-specimen unit examining an engine block. (B) The final design of the large-specimen gantry system.

The second instrument planned for the new laboratory will be an off-line alignment system comprising operable translation stages arranged so as to emulate both the large-specimen X-ray and the NRSF. This instrument will allow specimens with complex geometries to be mounted and prealigned on a transportable mounting stage, saving precious actual instrument time. This will significantly enhance the efficiency of the neutron measurements, as alignment often takes 50% of the available beam time.

Residual Stresses in Laser-Bonded TiC Coating—University of Tennessee Space Institute

University Collaborators: N. Dahotre and P. Kadolkar
HTML: T. R. Watkins

Researchers from the University of Tennessee Space Institute (UTSI) used the facilities in RSUC to characterize specimens with TiC and silicon particles laser-bonded to an aluminum substrate. The coatings consist of discrete TiC particles fused to a continuous aluminum matrix. The 10 wt % silicon addition increases the wettability of TiC with aluminum and also increases the fluidity of aluminum. This relatively new surface modification method has garnered some interest for wear applications, although initial observation of some coatings showed adhesion problems.

Since residual stress can be an important part of coating adhesion, a set of samples were processed at three different table speeds with two different aluminum substrates using a common 2-kW laser power setting. Visual inspection of these samples clearly showed adhesion decreasing with increasing table speed for either alloy. The coating on the 2024 alloys demonstrated better adhesion than the coating on the 6061 alloys. The better coating performance on the 2024 alloys was attributed, in part, to the 4 wt % Cu additions, which increased bonding of the 2024 alloy to the TiC.

Because the resulting coating surfaces were very rough (see Fig. 5-8), the researchers used HTML's new Philips X'Pert Pro X-ray Diffractometer to take advantage of the parallel beam optics. These optics remove the confounding effect of sample surface roughness on the observed peak position, which is necessary to make interpretable measurements. While some point-to-point variability was observed in residual stresses within a particular coating, data trends indicate that the compressive residual stress decreases (or becomes more tensile) as the table speed increases in both the TiC component of the coatings and the aluminum coating matrix/substrate (Fig. 5-9). These trends in residual stress magnitude are consistent with the adhesion trends observed visually. Thus, the best coatings are made with 2024 alloy laser-bonded at slower table speeds, allowing for more complete aluminum melting and TiC particulate bonding.

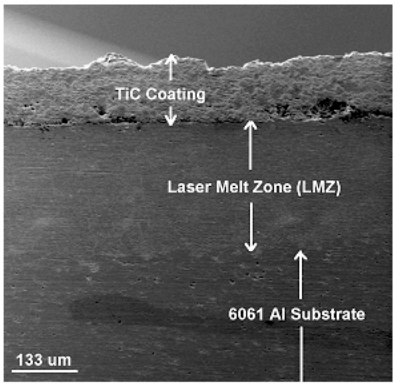
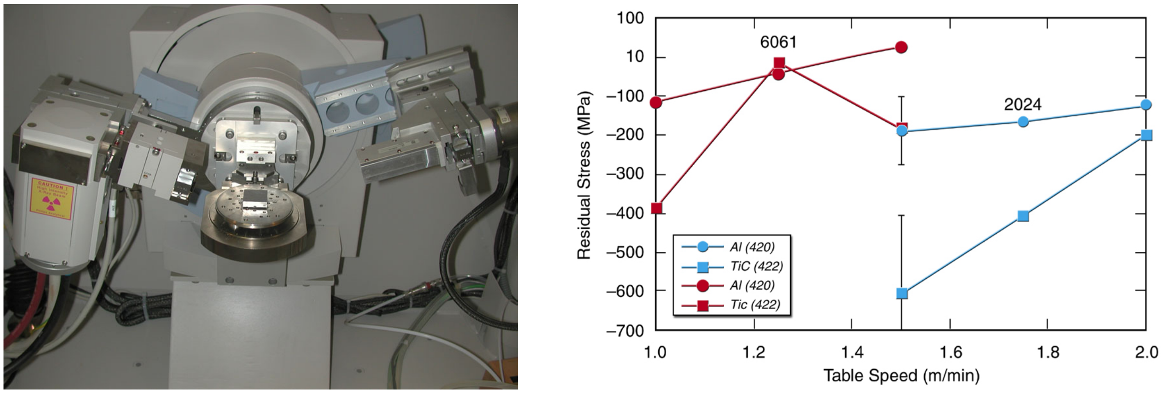


Fig. 5-8. Cross section of a 6061 alloy sample showing the microstructure at the coating-substrate interface.



(A) **(B)**

Fig. 5-9. (A) Philips X'Pert Pro Diffractometer in the parallel beam configuration to examine one of the TiC coatings. (B) The measured residual stresses as a function of table speed and substrate. Error bars represent the standard deviations for all data from a particular reflection.

Residual Stresses in Hard Turned Steels—Georgia Institute of Technology

University Collaborators: S. Melkote and S. Smith
HTML: T. R. Watkins

A researcher from Georgia Tech visited RSUC to identify residual stress profiles of various machined steel components for use in analysis of the resulting fatigue performance and service life. Hard turning is the process of machining hardened steels using single-point cutting tools (as opposed to grinding). There are a number of advantages in hard turning, including cheaper machine tools and tooling, versatility, less environmentally unfriendly waste, and streamlined production cycles. However, for hard turning to be accepted as an alternative process, the effects of the process on the microstructure and properties of the near surface must be evaluated and understood. In conjunction with fatigue testing, the residual stress depth profiles were obtained using X-ray diffraction methods and electropolishing for removal of layers. Results show that the compressive residual stress decreases with depth. Results were combined with fatigue life studies to fully characterize the effects of hard turning.

Residual Stresses in Aluminum with Cold Expanded Fastener Holes—Georgia Institute of Technology

University Collaborators: S. Johnson and D. A. Clark
HTML: S. Spooner, D. Q. Wang, T. R. Watkins, and C. Hubbard

Dave Clark, a master's degree student in mechanical engineering at Georgia Tech, visited RSUC to measure the residual stresses in 7050-T7451 aluminum plates with 0.25-in. holes given a cold expansion treatment (Fig. 5-10). Cold expansion is done by drawing an oversized mandrel through the holes. The resulting plastic deformation leaves the surface of the hole in a condition of high compressive residual stress. The stresses induced by cold expansion inhibit fatigue cracking; thus, the process significantly extends the useful life of aircraft structures. Fatigue tests were done on as-prepared test bars and on test bars annealed at 104°C to explore the possibility of relaxation of the compressive stresses caused by annealing. Although the fatigue crack geometry and surface condition of cracks were altered by annealing, the residual strains measured with neutron scattering showed no significant response to annealing. With the use of X rays, the compressive residual stress increased slightly and then decreased as a function of distance from the holes. These results will be correlated with ongoing mechanical fatigue testing at 27 and 70°C, as the purpose of this work is to determine whether the beneficial compressive residual stresses relax with time at these slightly elevated temperatures.

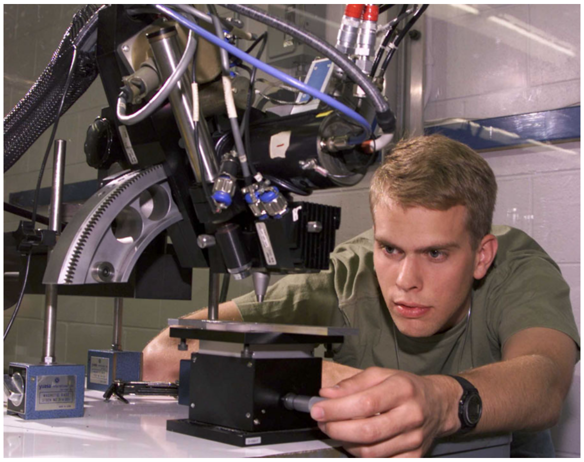


Fig. 5-10. Dave Clark positioning a sample underneath the X-ray large-specimen stress analyzer.

6. THERMOPHYSICAL PROPERTIES USER CENTER (TPUC)

Group Members

Camden R. Hubbard, Group Leader
Geneva N. Worley, Secretary

Ralph B. Dinwiddie
Wallace D. Porter
Hsin Wang

The Thermophysical Properties User Center (TPUC) is dedicated to measuring thermophysical properties as a function of temperature and correlating these properties with the processing, the microstructure, and the performance of materials. Specifically, TPUC staff work with users to determine thermophysical properties such as thermal diffusivity, thermal conductivity, specific heat, and thermal expansion and to characterize the thermal stability, high-temperature reactions and compatibility, and high-temperature oxidation and corrosion properties of materials. The materials studied include structural ceramics, engineering alloys, ceramic and metal matrix composites, ceramic precursors, superconducting materials, carbon materials, and carbon fiber composites.

TPUC continues to develop capabilities in the field of infrared (IR) imaging and sensing using focal-plane-array IR cameras and fast IR point detectors coupled with IR fibers and light pipes. These capabilities have been demonstrated on a wide variety of materials processes, in service performance characterizations, and in nondestructive evaluation (NDE) inspections.

TPUC Instruments and Capabilities

- Laser flash thermal diffusivity system
- Xenon flash thermal diffusivity system
- Hot disk thermal constants analyzer
- Three-omega thermal diffusivity system
- Radiance-HSX IR camera
- Alpha IR camera
- High-speed two-color single-point IR detectors
- Netzsch differential scanning calorimeter
- Dual-push-rod dilatometer
- Simultaneous thermal analysis
- Thermogravimetric analysis

Fast Weld Inspection Technique for Electronic Circuits—Motorola

Corporate Collaborator: H. Maleki
HTML: H. Wang and R. B. Dinwiddie

Researchers from Motorola worked with TPUC staff to develop a fast technique for inspecting welds within a ball grid array (BGA) for electronic circuits. The defective (weak) welds are between the circuit board and ceramic substrate. If the component passes the electrical continuity test, locating the weak welds is very difficult. The only techniques previously known to be useful are X-ray tomography or destructive inspection. Utilizing the IR imaging facility at TPUC, the researchers developed a new NDE technique using IR imaging. The traditional flash lamp heating and step heating methods could not locate the welds. However, with a high-voltage pulser, the welds can be seen clearly after a 1-ms, 150- to 300-V pulse. As shown in Fig. 6-1, the hot spots revealed missing connections that heated up after the pulse. For BGAs that passed continuity tests, the welds showed up nicely when 1-kV pulses were applied. More tests are planned at TPUC when a series of weak welds are prepared at Motorola.

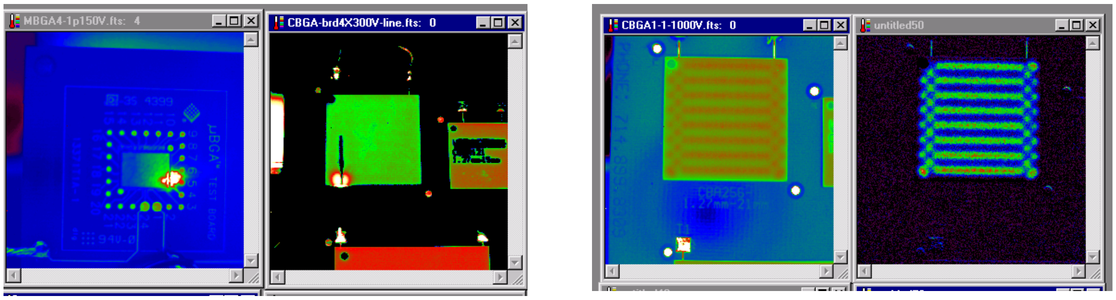
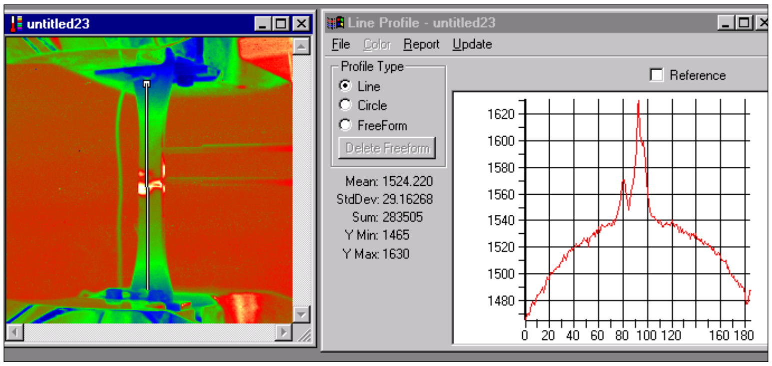


Fig. 6-1. Infrared images of welds produced by a nondestructive pulsed heating technique. *Left*: Bad welds heated up after 200-V pulses. *Right:* The entire BGA heated up after 1-kV pulses.

Infrared Monitoring of SiC-SiC Composites During Tensile Testing—University of Tennessee, Knoxville

University Collaborators: J. G. Kim and P. Liaw
HTML: H. Wang

TPUC worked with UTK Materials Science Department researchers on mechanical properties of SiC-SiC composites. An IR camera was used to monitor temperature change during tensile testing. As shown in Fig. 6-2, a localized temperature rise of 5 to 10°C was found at the moment of failure. No change was observed as close as 10 ms before the failure. This indicates that SiC-SiC composites experience brittle fracture. Sliding of fibers and fiber pullout were observed during the failure. Stress-strain curves and acoustic emission were also recorded during the tensile tests. The acoustic emission results are being analyzed in order to correlate the temperature changes captured by the IR camera.



Profile Type
● Line
○ Circle
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☐ Reference

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Mean: 1524.220
StdDev: 29.16268
Sum: 283505
Y Min: 1465
Y Max: 1630
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Fig. 6-2. Infrared monitoring of temperature changes in SiC-SiC composite during tensile testing. *Left:* Infrared image showing the final failure of a SiC-SiC composite specimen. *Right:* Line profile with *x*-axis showing pixels and *y*-axis showing IR intensity counts (1 K = 30 counts).

Thermal Interaction Between SiC Grinding Wheels and Zirconia—North Carolina State University

University Collaborators: A. J. Shih and J. Kong
HTML: R. B. Dinwiddie and H. Wang

North Carolina State University is combining the grinding capabilities of MITUC and the high-speed temperature measurement capabilities of TPUC to study ceramic grinding temperatures. Specifically, university researchers are investigating the thermal interactions between vitreous-bonded SiC grinding wheels and zirconia. The setup is designed to "look through" the zirconia and measure the contact temperature of the grinding wheel. These measurements have been made with and without lubricant. An IR fiber optic is positioned to view the grinding wheel from below the zirconia plate being machined. The fiber optic transmits the IR radiation to a two-color detector (3–5 and 8–11 µm). The detector can make measurements at a rate of 100 kHz.

Synthesis of High-Surface-Area Nanostructures—University of Tennessee, Knoxville

University Collaborator: C. Barnes
HTML: W. D. Porter

The TPUC simultaneous thermal analyzer was used to investigate the reaction steps involved during controlled heating of mixed metal oxides synthesized by a nonhydrolytic linking and condensation of nanosized building blocks consisting of silicates and metal alkoxides. These nanostructured solids are expected to be porous, high-surface-area materials that have homogeneous distributions of well-defined metal oxide clusters within the support matrix. One proposed use for these materials is as catalysts in the oxidative decomposition of volatile organic compounds. Results obtained from the STA investigations of $Si_8O_{12}(OSnMe_3)_8 + WCl_6$ were recently used to obtain funding of a proposal to DOE.

Testing the Effectiveness of Diamond and Diamond Composite Thermal-Management Substrate Designs—North Carolina State University

University Collaborators: Y. N. Saripalli and J. Kasichainula
HTML: R. B. Dinwiddie and H. Wang

North Carolina State University is developing single-layer and multilayer diamond and diamond composite substrates for improved thermal management in electronic packaging applications. The purpose of these substrates is to spread the heat away from hot areas within an integrated circuit and to transport the heat out of the package. These substrates are applied to the noncircuit side of the silicon wafer. Students from North Carolina State are using the thermal-imaging capabilities of TPUC to investigate the effectiveness of various heat spreader designs.

TPUC developed a new technique to compare the effectiveness of different substrate designs on spreading the heat generated on the active side of a silicon wafer. A small surface-mount resistor (2.0 × 1.2 mm) is pressed onto the silicon surface. Micropositioners are used to attach current probes to the resistor. A sequence of thermal images is then recorded while a constant current heats the resistor. The relative effectiveness of a heat spreader design can then be determined by measuring the temperature (IR signal) at a location away from the resistor as a function of time.

Figure 6-3 shows the preliminary results of a recent test comparing a diamond-coated substrate to a diamond + AlN-coated substrate. In this case, the diamond-coated substrate was more effective at spreading the heat over a wider area than the substrate with the composite coating. This technique takes into account any thermal resistance that may be present between the coating and the substrate as well as the difference in thermal properties of the substrates.

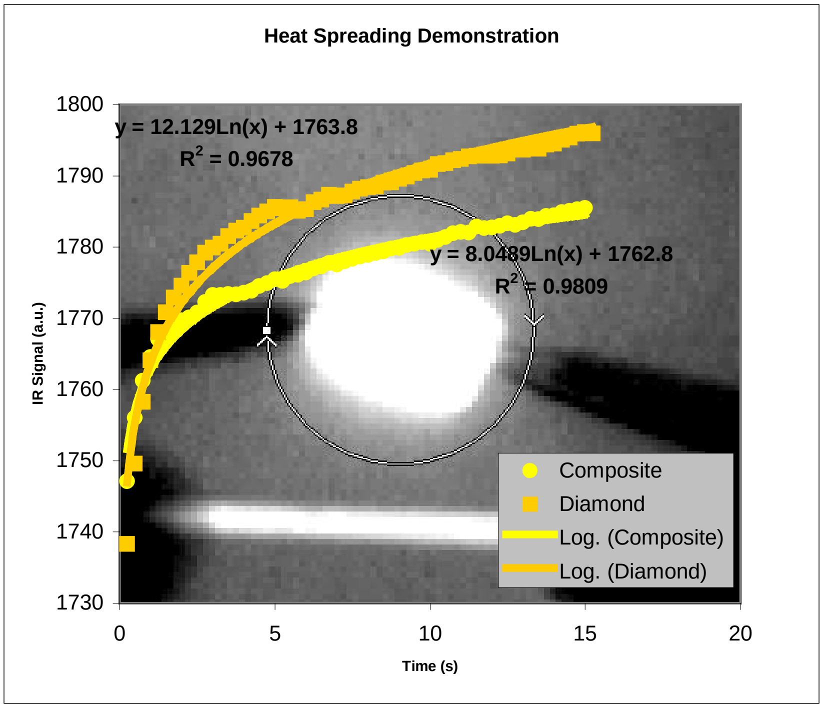

Fig. 6-3. Comparison of the thermal signature of a diamond-coated substrate to that of a substrate coated with diamond + aluminum nitride (composite).

Long-Term Aging of Thermal Barrier Coatings—Siemens Westinghouse

Corporate Collaborator: A. Burns
HTML: H. Wang

The thermal conductivity of electron-beam physical vapor deposition (EB-PVD) thermal barrier coatings (TBCs) has long been a challenge to researchers. Due to the columnar structure of the grains, it is very difficult to measure free-standing coatings, especially after extensive thermal aging. Although experiments have been done with EB-PVD TBCs on superalloy substrates, one of the goals of the DOE Advanced Turbine System requires that the coating be thermally aged at 1400°C for thousands of hours. No superalloy can be exposed to this high temperature in air for such a long time. To overcome this technical barrier, TPUC worked with Siemens Westinghouse on a traditional yttria-stabilized zirconia (YSZ) system. The researchers used alumina as substrates and the two-layer analysis capability of the laser flash thermal diffusivity system to obtain the thermal diffusivity of the TBC. The two-layer specimens survived the thermal aging treatment.

Measurement of Thermal Conductivity in Carbon Nanotube Composites—Rice University

University Collaborator: L. Yowell
HTML: H. Wang

Rice University continued to study carbon nanotube composites at TPUC. YSZ specimens with 1 to 5 vol % carbon nanotubes were prepared by tape casting and vacuum sintering. The thermal conductivities of over 70 specimens were measured at temperatures up to 1100°C. The thermal conductivity values are about 0.2–0.4 W/m K, making this material comparable to the lowest-thermal-conductivity thermal barrier coatings made by the air plasma spray technique. The thermal conductivity of polymers containing carbon nanotubes was also tested. The addition of carbon nanotubes was found to increase the thermal conductivity of the composites.

Infrared Imaging of Local Heating in HFETs—University of South Carolina

University Collaborator: J. A. Khan
HTML: H. Wang and R. B. Dinwiddie

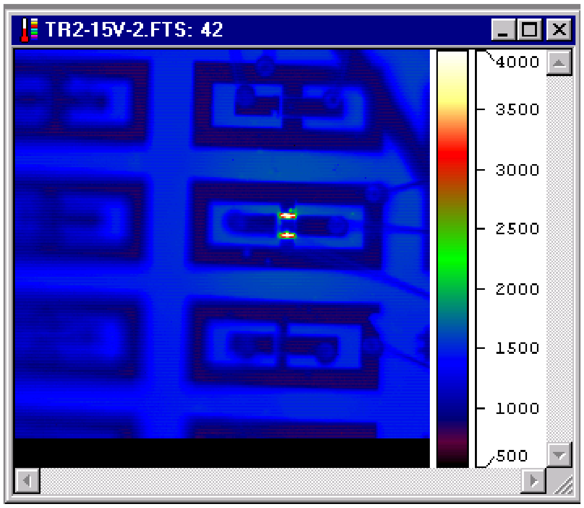

Fig. 6-4. Temperature map of HFET showing the gate temperature over 150°C (15 V dc).

Temperatures at GaN heterostructure field-effect transistors (HFETs) can reach as high as 330°C locally, according to finite-element analysis conducted at the University of South Carolina. The IR imaging system at TPUC was used to study this device. An IR microscope lens at TPUC gives a spatial resolution of 5.4 µm per pixel. The device was wire-bonded and energized by a dc power supply. Up to 15 V was applied to the gate (GaN strip), and temperature change as a function of time was recorded. The device temperature was found to be around 150–180°C, depending on the gate structure (Fig. 6-4). Since the actual area of the HFET is on the order of a few microns, the 5.4-µm resolution may have collected only the average temperature of that area; the modeling result of a local temperature of over 300°C is possible. Localized cooling may be needed for the HFET devices.

Characterization of Thermal Transport in Aircraft Brake Carbon-Carbon Composites—Southern Illinois University

University Collaborator: D. Marx
HTML: H. Wang and R. B. Dinwiddie

A researcher from the Center for Advanced Friction Studies at Southern Illinois University (SIU) worked with TPUC staff on thermal transport characterization of carbon-carbon composites used in aircraft brakes. Polyacrylonitrile- (PAN)-based carbon fiber bundles are believed to conduct heat more efficiently in this material. Thermal images at a microscopic level were needed to develop a heat-conduction model for this composite. The lock-in thermography technique developed from an ORNL Seed Money project was used in this study. An IR line laser was used to periodically heat one edge of a plate sample (dimension 1 × 1 × 0.25 in.) at 1 Hz. An IR camera with a microscope attachment was used to monitor the temperature waves on the plate surface (perpendicular to the laser line). Four images were taken within each heat cycle. The amplitude and phase maps (Fig. 6-5) show the heat conduction pattern in the composite. These maps reveal that the fiber bundles conduct more heat than the matrix. A computer-controlled stage is being built at SIU to improve the sample positioning and camera stability.

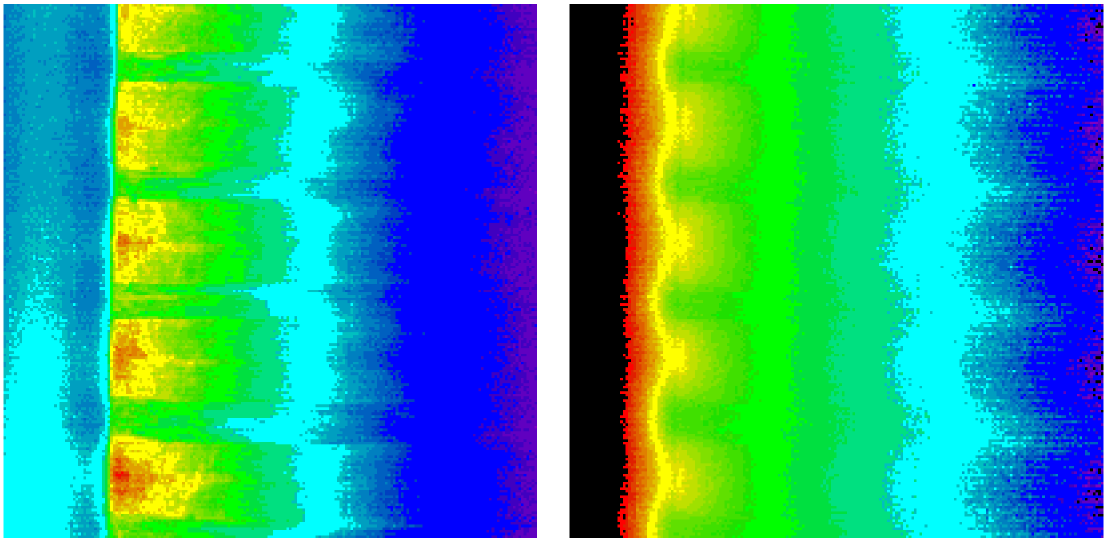

Fig. 6-5. Carbon composite during 1-Hz lock-in thermography test; 1 pixel = 5.4 µm.
***Left*: Amplitude image at 1 Hz. *Right*: Phase image at 1 Hz.**

Standard Nonproprietary User Agreements

The next several pages comprise a listing of universities and industries that have entered into standard nonproprietary user agreements with the HTML. These include 353 industries, 19 government facilities, and 159 universities from across the United States.

U.S. INDUSTRY—353

Alabama

Citation Corp. (Birmingham)
Monarch Tile, Inc. (Florence)
Southern Research Institute (Birmingham)
United Defense LP (Anniston)

Arizona

Advanced Ceramics Research (Tucson)
AlliedSignal (Phoenix)
Materials Focus Inc. (Tucson)
Motorola (Tempe)
RASTRA of the Americas (Litchfield Park)

California

AlliedSignal, Inc., Ceramic Components (Torrance)
AlliedSignal EMRC (Los Angeles)
Alzeta Corp. (Santa Clara)
Amercom Inc. (Chatsworth)
Ceradyne, Inc. (Costa Mesa)
CERCOM, Inc. (Vista)
Electroglas, Inc. (Santa Clara)
Ensei, Inc. (Pismo Beach)
FMC Corp. (Santa Clara)
Guidance & Control Systems/Litton Ind. (Woodland Hills)
IBM Almaden Research Center (San José)
Intel Corp. (Santa Clara)
Lockheed Martin Skunk Works (Palmdale)
Membrane Technology Research (Menlo Park)
M. J. Schiff & Associates, Inc. (Upland)
Northrop Corp. (Pico Rivera)
Nuclear & Aerospace Materials Corp. (Poway)
Rohr Inc. (Chula Vista)
Solar Turbines, Inc. (San Diego)
SRI International (Menlo Park)
Sullivan Mining Corp. (San Diego)
Sundstrand Power Systems (San Diego)
Tylan General (Torrance)
Ultramet (Pacoima)
Unit Instruments (Yorba Linda)
X-Ray Instrumentation Assoc. (Mountain View)

Colorado

Coors Ceramics Company (Golden)
Golden Technologies Co. (Golden)
Johns Manville (Littleton)
Material Physics Research (Highlands Ranch)
NA Technologies (Golden)
Quantum Peripherals (Louisville)
Schuller Int’l Inc. (Littleton)
TDA Research Inc. (Wheat Ridge)

Connecticut

ABB C-E Services, Inc. (Windsor)
Cytec Industries, Inc. (Stamford)
Plasma Coatings, Inc. (Waterbury)
Steven Winter Associates, Inc. (Norwalk)
Torrington Co. (Torrington)
United Technologies/Pratt & Whitney (East Hartford)

Delaware

E. I. du Pont de Nemours (Wilmington)
E. I. du Pont de Nemours Fluorochemicals (Wilmington)
Guidance & Control Systems-Litton Hercules, Inc. (Wilmington)
Rodel, Inc. (Newark)

District of Columbia

American Iron & Steel Institute
Appliance Industry/Government CFC Replacement Consortium, Inc.
SPI/SPFD
Structural Insulated Panel Assoc.

Florida

American Boarts Crushing Co. (Boca Raton)
Lockheed Martin Elect. Info & Missile–LMES (Winter Garden)
National High Magnetic Field Laboratory (Tallahassee)
Pratt & Whitney (W. Palm Beach)
Siemens Westinghouse Power Corp. (Orlando)
U.S. Filter Corp. (Deland)
Westinghouse Electric Corp. (W. Palm Beach)

Georgia

Advanced Engineered Materials (Atlanta)
AMERCORD, Inc. (Lumber City)
Ceradyne, Inc. (Scottdale)
Healthdyne Technologies (Marietta)
Hebel Building Systems (Smyrna)
Ionic Atlanta, Inc. (Atlanta)
Institute of Paper Science & Tech. (Atlanta)
Microcoating Technologies (Chamblee)
Motorola (Lawrenceville)
RCF Seals (Vidalia)
Rolls Royce, Inc. (Atlanta)
Thermal Ceramics (Augusta)

Illinois

A.E. Staley Manufacturing Co. (Decatur)
A. Finkl & Sons (Chicago)
Adtech Nepth, Inc. (Oak Park)
AlliedSignal (Des Plains)
Alloy Eng. & Casting (Champagne)
Belcan Corp. (Peoria)
Caterpillar, Inc./Tech. Ctr. (Peoria)
Insulating Concrete Form Assoc. (Glenview)
Metal Constructing Assoc. (Glenview)
Wagner Castings Co. (Decatur)

Indiana

AlliedSignal (South Bend)
Allison Engine Co. (Indianapolis)
Allison Gas Turbine (Indianapolis)
Cummins Engine Co. (Columbus)
Dana Corp. (Richmond)
Firestone Building Products Co. (Camel)
Guidance & Control System (Litton)
GM Corporation/Delco Remy (Andersonville)
Haynes International (Kokomo)

Iowa

Composite Technologies Corp. (Ames)

Kentucky

ARCO Aluminum, Inc. (Louisville)
Florida Tile Industries (Lawrenceburg)
Lexmark (Lexington)
Logan Aluminum (Russellville)
Machining Research, Inc. (Florence)
Stoody Co. (Bowling Green)
United Catalysts (Louisville)

Louisiana

Dow Chemical Co. (Plaquemine)
Lockheed Martin Michould Space Systems (New Orleans)

Maine

Surmet Corporation (Burlington)

Maryland

Krispin Technologies (Rockville)
RCMA (Rockville)
Refractory Composites Inc. (Glen Burnie)
W. L. Gore & Associates (Elkton)
W. R. Grace & Co./Conn. (Columbia)

Massachusetts

American Superconductor Corp. (Westborough)
Brigham and Women’s Hospital (Holyoke)
Busek Co. (Natick)
Ceramics Process Systems Corp. (Cambridge)
ChandKare Tech. Ceramics (Worcester)
Dynamet Technology (Burlington)
Foster-Miller, Inc. (Waltham)
Giner, Inc. (Waltham)
GTE Laboratories, Inc. (Waltham)
Hydrogen Microplasmatron Technologies, LLC (Cambridge)
JPS Elastomerics Co. (Holyoke)
Niton Corp. (Bedford)
Norton Co. (Northboro)
Norton/TRW Ceramics (Northboro)
Osram Sylvania/Univ. of Massachusetts (Lowell)
Phillips Analytical, Inc. (Natick)
Refractory Testing Associates (Chestnut Hill)
Rohm & Haas (Woburn)
Sarnafil, Inc. (Canton)
Single Ply Roofing Institute (Needham)
St. Gobain Norton (Northboro)
Textron Specialty Materials (Lowell)
Uniform Metal Tech. LLC (Watertown)

Michigan

Bosch Braking Systems (Farmington Hills)
Chrysler Corporation (Highland Park)
Detroit Diesel Corp. (Detroit)
Dow Corning Corp. (Midland)
Duro-Last Roofing, Inc. (Saginaw)
Eaton Corp. (Southfield)
Energy Conversion Devices, Inc. (Troy)
Ford Motor Company (Ann Arbor)
GM AC-Rochester (Flint)
GM Powertrain Group (Pontiac)
GM Research & Development (Warren)
Hoskins Mfg. (Hamburg)
Howmet (Whitehall)
Metal Building Manufacturers Assoc. (Traverse City)
Modern Alloying Technologies, LLC (West Bloomfield)
Parker Abex NWL (Kalamazoo)
Thixomat (Ann Arbor)
Valenite, Inc. (Troy)
Visteon Corp. (Allen Park)

Minnesota

3M (St. Paul)
FMC Naval Systems Division (Minneapolis)
Seagate Technology (Minneapolis)

Mississippi

Alpha Optical Systems (Ocean Springs)
Richard Knof McMullan (Decatur)

Missouri

McDonnell Douglas Corp. (St. Louis)
SB&TD Business Systems (Lancaster)
TAMKO Roofing Products, Inc. (Joplin)

Montana

Anaconda Foundry Fab. (Anaconda)
Columbia Falls Alumin. (Columbia Falls)

Nevada

DRI Institute (Reno)

New Hampshire

FLUENT Inc. (Lebanon)
Miniature Precision Bearings (Keene)

New Jersey

AlliedSignal (Morristown)
AT&T Bell Laboratories (Murray Hill)
Ceramic Magnetics, Inc. (Fairfield)
Certech, Inc. (Wood Ridge)
Engelhard Corp. (Edison)
Exxon Research & Eng. Co. (Annadell)
International Paper (Princeton)
INRAD Inc. (Northvale)
Lucent Technologies (Murray Hill)
Materials Technology (Shresburg)
Mobil Research & Development Corp. (Pennington)
Mobil Technical Co. (Paulsboro)
Nanopowder Enterprises, Inc. (Piscataway)
NEC Research Inst. (Princeton)
Phone-Poulenc, Inc. (Cranbury)
Stryker Howmedica Osteonics (Rutherford)

New Mexico

American Polysteel Forms (Albuquerque)
Eberline Instruments (Santa Fe)
Environmental Tech. & Education (Albuquerque)
TPL, Inc. (Albuquerque)

New York

Advanced Refractory Tech., Inc. (Buffalo)
AKZO Nobel Chemicals, Inc. (Dobbs Ferry)
Applied Nano Metrics, Inc. (Stormville)
CMP Industries, Inc. (Albany)
Carborundum Co. (Niagara Falls)
CDH Energy Corp. (Cazenovia)
Corning Inc. (Corning)
Donald T. Schmid (Clarence)
Eastman Kodak Co. (Rochester)
General Electric (Schenectady)
Goldman Oved Diamond Co. (NYC)
Monofrax, Inc. (Falcomer)

MRC, Division of Praxair Surface Technologies (Orangeburg)
ReMaxCo Technologies, Inc. (Kenmore)
Sulzer-Metco (Westbury)
T. J. Watson Research Center (Yorktown Heights)
U.K. Software Services, Inc. (Grand Island)
X-Ray Optical Systems (Albany)

North Carolina

Advanced Energy (Raleigh)
Cree Research, Inc. (Durham)
Magnequench Technical Center (Research Triangle Park)
MicroMet Technology, Inc. (Matthews)
PCC Airfoils (Beachwood)
Porvair Fuel Cell Technology (Hendersonville)
Selee Corp. (Hendersonville)
Syngenta Agribusiness Biotechnology Research (Research Triangle Park)
Teledyne Allvac (Monroe)
Unimin Corp. R&D (Bakersville)

Ohio

Advanced Ceramics Corp. (Lakewood)
Ashland Chemical (Dublin)
Eaton Corp. (Willoughby Hills)
Edison Welding Institute (Columbus)
Engineering Mechanics Corp. of Columbus (Columbus)
Equistar Technology Center (Cincinnati)
Doehler-Jarvis Tech. (Toledo)
GE Aircraft Engines (Cincinnati)
Goodyear Tires & Rubber Co. (Akron)
Libbey Owens Ford Co., Inc. (Toledo)
Lincoln Electric (Cleveland)
LTV Steel Co. (Independence)
Mead Research (Chillicothe)
Milacron, Inc. (Cincinnati)
Metal Building Manufacturers Assoc. (Cleveland)
Owens Corning Tech. Ctr. (Granville)
Park-Ohio Trans. (Cleveland)
PCC Airfoils (Beachwood)
Proctor & Gamble (Cincinnati)
Sandusky Int’l. (Sandusky)
Republic Technologies, International (Lorain)
Rhenium Alloys, Inc. (Elyria)
Tosoh SMD, Inc. (Grove City)
Universal Energy Systems, Inc. (Dayton)
Western Environmental (Franklin)

Oklahoma

MH Sukkar Research (Tulsa)

Pennsylvania

AHT, Inc. (Chicora)
Advanced Technology Materials, Inc. (University Park)
Alcoa Tech. Center (Alcoa Center)
Aluminum Co. of America (Alcoa Center)
Armstrong World Industries (Lancaster)
Bethlehem Steel Corp. (Bethlehem)
Calgon Corp. (Pittsburgh)
Carlisle Syntec, Inc. (Carlisle)
Certainteed Corp. (Valley Forge)
Concurrent Technologies Corp. (Johnstown)
IBACOS (Pittsburgh)
J&L Specialty Steel (Pittsburgh)
Kennametal, Inc. (Latrobe)
Leroy A. Landers (Philadelphia)
PPG Industries, Inc. (Lancaster)
Materials Resources International (North Wales)
SB&TD Business Systems (Lancaster)
Thermacore, Inc. (Lancaster)
Westinghouse Science & Tech Ctr (Pittsburgh)

Rhode Island

Quadrax Corp. (Portsmouth)

Tennessee

American Magnetics (Oak Ridge)
American Matrix, Inc. (Knoxville)
AMS (Knoxville)
Atlantic Research Corp. (Knoxville)
Browne Tech. (Nashville)
BTR Sealing Systems (Rockford)
Carroll Kenneth Johnson (Oak Ridge)
Cavin Consulting Services (Knoxville)
Church & Dwight Co., Inc. (Knoxville)
Complete Machine Co. (Clinton)
Computational Mechanics Corp. (Knoxville)
Computational Systems (Knoxville)

CTI, Inc. (Knoxville)
Cummins Engine Co., Inc. (Memphis)
DG Trim Products (Alcoa)
Dow Corning Wright Corp. (Arlington)
Eagle Racing (Loudon)
Eastman Kodak/Chemical (Kingsport)
Environmental Engineering Group, Inc. (Knoxville)
Forged Performance Products, Inc. (Oak Ridge)
GENASE, LLC (Loudon)
Goal Line Co. (Knoxville)
Great Lakes Research (Elizabethton)
H. R. DeSelm (Knoxville)
Herbert E. McCoy, Jr. (Clinton)
IMTech Company (Knoxville)
IntraSpec, Inc. (Oak Ridge)
J. A. Martin (Knoxville)
James Williams (Powell)
Jeffrey Chain Corp. (Morristown)
Microbial Insight, Inc. (Knoxville)
MINCO Acquisition Co. (Midway)
MMPact, Inc. (Oak Ridge)
M4 Environmental Systems, L. P. (Oak Ridge)
Nano Instruments, Inc. (Knoxville)
Noranda Magnesium, Inc. (Franklin)
Oxyrase (Knoxville)
PCC Enterprises (Oak Ridge)
Pyrotec, Inc. (Trenton)
ReMaxCo Technologies (Kingston)
Ronald K. McConathy (Kingston)
SENES Oak Ridge, Inc. (Oak Ridge)
Singleton Labs (Louisville)
Smelter Service Corp. (Mt. Pleasant)
Smith & Nephew (Memphis)
Stirling Technologies, Inc. (Oak Ridge)
Technology for Energy Corp. (Knoxville)
Textron Specialty Materials Div. Avco (Nashville)
William Thomas Pope (Clinton)
Tennessee Center for R&D (Knoxville)
Third Millenium Tech., Inc. (Knoxville)
TTE Diecasting (Oak Ridge)
Vamistor Corp. (Sevierville)

Texas

Aera Corp. (Austin)
Agriboard Industries (Electra)
CarboMedics, Inc. (Austin)
Dallas Optical Systems, Inc. (Rockwell)
Exxon Chemical Co. (Baytown)
Exxon Corp. (Houston)
Exxon Research & Engineering Co. (Annadale)
Ludlum Measurement Inc. (Sweetwater)
Poco Graphite, Inc. (Decatur)
Robert Hageman (Austin)
Smith International, Inc. (Houston)
Southwest Research Institute (San Antonio)
Stone & Webster Eng. (Houston)
Texas Instruments (Dallas)
TSC Thermo Sensors Corp. (Garland)
Tycom Corporation (Austin)

Utah

LoTEC, Inc. (Salt Lake City)
Mantic Corp. (Salt Lake City)

Virginia

B&W Nuclear Technologies (Lynchburg)
Babcock & Wilcox (Lynchburg)
Institute for Defense Analyses (Alexandria)
Energy Recovery, Inc. (Virginia Beach)
E. R. Johnson Associates, Inc. (Fairfax)
Hy-Tech Res. Corp. (Radford)
Materials Technologies of Virginia (Blacksburg)
Philip Morris (Richmond)
Reynolds Metals Company (Richmond)
Soil and Land Use Tech. (McLean)
Synterials, Inc. (Herndon)

Washington

Chiroscience R&D Inc./Darwin Molecular (Bothell)
Galvalume Sheet Producers of North America (Kalama)
Kyocera Industrial Ceramics Corp. (Vancouver)
The Boeing Co. (Seattle)
Weyerhaeuser Co. (Tacoma)

West Virginia

Huntington Alloys/Special Metals Division (Huntington)
INCO Alloys (Huntington)
Special Metals Corp. (Huntington)
Weirton Steel Corp. (Weirton)

Wisconsin

Federal Mogul Power Train Systems (Manitowoc)
Tower Automotive (Milwaukee)
Waukesha Electric Systems (Waukesha)

OTHER GOVERNMENT FACILITIES—19

Federal Highway Administration (Va.)
Idaho National Engineering and Environmental Laboratory (Idaho Falls)
Institute for Defense Analyses (Del.)
Lockheed Martin Aero Naval Systems (Md.)
Los Alamos National Laboratory (Los Alamos, N.M.)
NASA Glenn Research Center (Cleveland)
NASA Langley Research Center (Va.)
NASA Lewis Research Center (Ohio)
NASA/Marshall Space Flight Center (Huntsville, Ala.)
National Highway Traffic Safety (DC)
Naval Post Graduate School (Calif.)
Naval Research Laboratory (DC)
NIST (Md.)
Sandia National Laboratories (Livermore, Calif.)
Space and Naval Warfare Systems Center (Calif.)
U.S. Army Research Lab (Va.)
U.S. Bureau of Mines (New York)
U.S. FDA (Md.)
U.S. Naval Academy (Md.)

UNIVERSITIES—159

Alabama

Alabama A&M (Normal)
Auburn University (Auburn)
Tuskegee Univ. (Tuskegee)
University of Alabama (Birmingham/Tuscaloosa)
University of Alabama (Huntsville)

Arizona

Arizona State (Tempe)
Univ. of Arizona (Tucson)

California

California Inst. of Tech. (Pasadena)
California State Univ. (Los Angeles)
Stanford Univ. (Stanford)
Univ. of Calif., Berkeley
Univ. of Calif, Davis
Univ. of Calif., Irvine
Univ. of Calif., Los Angeles
Univ. of Calif., San Diego
Univ. of Calif., Santa Barbara
Univ. of Calif., Santa Cruz
Univ. of S. Calif. (Los Angeles)

Colorado

Colorado School of Mines (Golden)
Univ. of Denver (Denver)

Connecticut

Univ. of Connecticut (Storrs)
Yale Univ. (New Haven)

Delaware

Univ. of Delaware (Newark)

District of Columbia

George Washington Univ.
Howard University

Florida

Florida A&M Univ. (Tallahassee)
Florida Atlantic Univ. (Boca Raton)
Florida International Univ. (Miami)
Florida Solar Energy Center (Cape Canaveral)
Florida State Univ. (Tallahassee)
Univ. of Central Fl. (Orlando)
Univ. of Florida (Gainesville)
Univ. of Salford (West Palm Beach)

Georgia
Georgia Inst. of Tech. (Atlanta)

Hawaii
Univ. of Hawaii (Honolulu)

Illinois
Illinois Inst. of Tech. (Chicago)
Northwestern Univ. (Evanston)
S. Ill. Univ. (Carbondale)
Univ. of Ill. (Urbana)

Indiana
Indiana University (Indianapolis)
Purdue Univ. Calumet (Hammond)
Purdue Univ. (West Lafayette)
Univ. of Notre Dame (South Bend)

Iowa
Iowa State Univ. (Ames)

Kansas
Kansas State Univ. (Manhattan)
Wichita State University (Wichita)

Kentucky
Berea College (Berea)
Eastern Kentucky State (Richmond)
Univ. of Kentucky (Lexington)
Univ. of Louisville (Louisville)
Western Kentucky Univ. (Bowling Green)

Louisiana
Louisiana State Univ./A&M College
(Baton Rouge)
Southern Univ. (Baton Rouge)
Univ. of New Orleans (New Orleans)

Maine
Univ. of Maine (Orono)

Maryland
Johns Hopkins Univ. (Baltimore)
Univ. of Maryland (College Park)

Massachusetts
Boston Univ. (Boston)
Clark Univ. (Worcester)
Harvard Univ. (Cambridge)
Mass. Inst. of Tech. (Cambridge)
Mt. Holyoke College (South Hadley)
Northeastern (Boston)
Tufts Univ. (Medford)
Univ. of Mass. (Amherst)
Univ. of Mass. (Lowell)

Michigan
Michigan State Univ. (East Lansing)
Michigan Tech. Univ. (Houghton)
Univ. of Michigan (Ann Arbor)
Wayne State Univ. (Detroit)
Western Michigan Univ. (Kalamazoo)

Minnesota
Univ. of Minnesota (Minneapolis)

Mississippi
Mississippi College (Clinton)
Mississippi State Univ. (Miss. State)
The University of Mississippi (Oxford)

Missouri
Lincoln Univ. (Jefferson City)
Univ. of Missouri (Columbia)
Univ. of Missouri (Rolla)
Washington Univ. (St. Louis)

Montana
Univ. of Montana (Missoula)

Nebraska
Univ. of Nebraska-Lincoln (Lincoln)

Nevada
Univ. of Nevada (Reno)

New Hampshire
Dartmouth College (Hanover)

New Jersey
New Jersey Inst. of Tech. (Newark)
Princeton Univ. (Princeton)
Rutgers Univ. (Piscataway)
Stevens Inst. of Tech. (Hoboken)

New Mexico
New Mexico Tech. (Socorro)
New Mexico State (Las Cruces)
Univ. of NM (Albuquerque)

New York

Alfred Univ. College of Ceramics (Alfred)
Clarkson Univ. (Potsdam)
Cornell Univ. (Ithaca)
Polytechnic Univ. (Brookland)
Rensselaer Polytechnic Inst. (Troy)
Rochester Inst. of Tech. (Rochester)
State Univ of New York (Stonybrook)
Univ. of Rochester (Rochester)

North Carolina

Appalachian State Univ. (Boone)
Duke Univ. (Durham)
North Carolina A&T State Univ. (Greensboro)
North Carolina State Univ. (Raleigh)
Univ. of North Carolina (Chapel Hill)
Univ. of North Carolina (Charlotte)
UNC School of Dentistry (Chapel Hill)

North Dakota

Univ. of North Dakota/Energy & Environmental Research (Grand Forks)

Ohio

Case Western Reserve Univ. (Cleveland)
Denison Univ. (Granville)
John Carroll Univ. (University Heights)
Kent State Univ. (Kent)
Ohio State Univ. (Columbus)
Ohio Univ. (Athens)
Univ. of Cincinnati (Cincinnati)
Univ. of Akron (Akron)
Univ. of Dayton (Dayton)
Univ. of Toledo (Toledo)
Wright State Univ. (Dayton)

Oklahoma

Oklahoma State Univ. (Stillwater)
Univ. of Oklahoma (Oklahoma City)

Oregon

Oregon Graduate Institute (Portland)
Oregon State Univ. (Corvallis)
Portland State Univ. (Portland)

Pennsylvania

Carnegie Mellon Univ. (Pittsburgh)
Drexel (Philadelphia)
Lehigh Univ. (Bethlehem)
Pennsylvania State Univ. (University Park)
Univ. of Pennsylvania (Philadelphia)
Univ. of Pittsburgh (Pittsburgh)

Rhode Island

Brown Univ. (Providence)

South Carolina

Clemson Univ. (Clemson)
Furman University (Greenville)
Univ. of S. Carolina (Columbia)

South Dakota

S. Dakota State Univ. (Brookings)

Tennessee

East Tennessee State Univ. (Johnson City)
Fisk Univ. (Nashville)
Maryville College (Maryville)
Tennessee State Univ. (Nashville)
Tennessee Tech. Univ. (Cookeville)
Tennessee Tech. Univ. (Jacksboro)
Univ. of Memphis (Memphis)
Univ. of Tennessee (Knoxville)
Vanderbilt Univ. (Nashville)

Texas

Rice Univ. (Houston)
Texas A&M Univ. (College Station)
Univ. of Houston (Houston)
Univ. of North Texas (Denton)
Univ. of Texas (Arlington, Austin, El Paso)
Univ. of Texas-Pan American (Edinburg)

Utah

Univ. of Utah (Salt Lake)

Virginia

Norfolk State Univ. (Norfolk)
VPI & State Univ. (Blacksburg)
Univ. of Virginia (Charlottesville)

Washington

Gonzaga University (Spokane)
Univ. of Washington (Seattle)
Washington State Univ. (Pullman)

West Virginia

West Virginia Univ. (Morgantown)

Wisconsin

Marquette Univ. (Milwaukee)
Univ. of Wisconsin (Madison)

International Universities—1

University of British Columbia
(Vancouver, Canada)

HTML Proposal List

HTML Proposal List

Proposal No.	Organization	Title	Lead Center	Spokesperson	Staff Contacts
2001-001	Intel Corporation (2)	Dopant Mapping of Transistor Devices with Electron Holography	MAUC	Kevin D. Johnson	E. Voelkl
2001-002	Techmer PM, LLC (1)	Investigation of Diffusion of Low Melt Additives Into Titanium Dioxide Pigment Particles	MAUC	Erik Nielsen	L. Walker
2001-003	Lear Corporation (1)	Fracture of Powder Metallurgy Bushings Under No Load	MAUC	Russell House	T. Nolan L. Walker
2001-004	Poco Graphite, Inc. (2)	Characterization of Poco's ACF-10Q and ACF-10QE2 Graphite Grades for Aircraft Engine Applications	MITUC	Abuagela H. Rashed	P. Blau
2001-005	Philips Analytical Inc. (1)	High-Temperature Residual Stress Measurements in Alumina Scales	RSUC	Rik Kerstens	T. Watkins
2001-006	U of Kentucky (16)	Silicon Diffusion Across Clad-Core Interface of an Aluminum Brazing Sheet	MAUC	Dusan P. Sekulic	A. Pasto T. Nolan
2001-007	PPG Industries (6)	Characterization of the Changes in Layer and Interfacial Microstructure in Heated PVD Sungate Coatings on Glass	MAUC	Scott D. Walck	L. Allard
2001-009	U of Tenn.(86)	The Influence of Residual Stress on Fatigue Life Under Constant and Variable Amplitude Loading Conditions	RSUC	Waldek Wladimir Bose	C. Hubbard
2001-010	ORNL (11)	The Polymorphism Phase Transition and Thermal Hysteresis of Potassium Rare Earth Double Phosphate Complexes	DUC	Lynn Boatner	C. Rawn
2001-011	Western Kentucky U	The Effect of Artificial Aging and Mg Content on Work Hardening Characteristics of Cast Al-Si-Mg Alloys in Tension and Compression	MCAUC	Murat Tiryakioglu	E. Lara-Curzio K. Liu S. Viswanathan
2001-013	Ultramet	Measurement of O-Ring Tensile Strength and Fracture Toughness of CVD-SiC Open-Cell Foam at Elevated Temperatures	MCAUC	Edwin Stankiewicz	E. Lara-Curzio
2001-014	U of South Carolina (3)	A Study of Temperature Field in a GaN Hetrostructure Field-Effect Transistor	TPUC	Jamil A. Khan	R. Dinwiddie
2001-015	North Carolina State U (20)	Study of the Wear of EDM Recast Layer in Metal Bond Diamond Grinding Wheel	MITUC	Albert J. Shih	S. McSpadden R. Ott
2001-016	North Carolina State U (21)	Cylindrical Wire EdMed Surface	MITUC	Albert J. Shih	S. McSpadden L. Allard

HTML Proposal List (continued)

Proposal No.	Organization	Title	Lead Center	Spokesperson	Staff Contacts
2001-017	U of Houston (1)	Structural Analysis of Ordered Semiconductor Alloy Films	DUC	Simon C. Moss	C. Hubbard J. Bai
2001-018	Georgia Tech (39)	Thermomechanical Properties of Metallic Linear Cellular Materials	TPUC	David L. Mcdowell	H. Wang
2001-019	U of Kentucky (17)	Investigation of Thermal Residual Stress in Directionally Solidified Alumina-Cubic Zirconia Eutectics	RSUC	Elizabeth C. Dickey	E. Payzant
2001-020	U of Cincinnati (8)	Orientation Relations and Interfacial Structure During Nucleation in the Alpha to Massive Gamma and Transformation in Ti-Al Alloys.	MAUC	Vijay K. Vasudevan	L. Allard
2001-021	U of Tenn. (87)	Electron Microscopy Investigations of Porous Building Block Metal Oxides	MAUC	Craig Barnes	L. Allard K. More
2001-022	Porvair Fuel Cell Technology	Mechanical & Thermal Characterization of Metallic Foams	MCAUC	Ken Butcher	E. Lara-Curzio C. Hubbard
2001-023	Brookhaven National Laboratory	In Situ Resonant X-Ray Diffraction Studies of Li-Ion Battery Materials	DUC	Mahalingam Balasubramanian	C. Hubbard J. Bai
2001-024	Noranda Magnesium, Inc. (1)	Study of Creep Mechanisms in Magnesium Alloys	MAUC	Carl Lee	E. Lara-Curzio L. Allard
2001-025	U of California - Davis (3)	High-Temperaure Testing of Si_3N_4/SiC Ceramic Nanocomposites Prepared from Polymer Precursors	MCAUC	Amiya K. Mukherjee	E. Lara-Curzio K. Liu
2001-026	U of Tenn. Space Inst. (81)	Evaluation of Mechanical Properties of Laser Coated Iron Oxide Al-Alloy Using Nano-indentation Technique	MCAUC	N. B. Dahotre	L. Riester
2001-030	Magnequench (1) North Carolina State U	Mechanical Properties Characterization of Nd-B-Fe-Permanent Magnetic Materials	MCAUC	Bao-Min Ma	E. Lara-Curzio
2001-031	ORNL (13) Chem Tech	Development of Nanocrystalline Ceramic Particles and Thin Films	DUC	Michael Z. Hu	E. Payzant L. Allard
2001-032	Magnequench (2) North Carolina State U	Machine of Nd-B-Fe Permanent Magnetic Materials	MITUC	Bao-Min Ma	S. McSpadden

HTML Proposal List (continued)

Proposal No.	Organization	Title	Lead Center	Spokesperson	Staff Contacts
2001-033	Ohio State U (07)	Fabrication of Near Net-Shaped, Dense Carbide/Refractory Metal Composites by the Low Temperature Prima-DCP Process	MAUC	Ken H. Sandhage	L. Allard L. Walker
2001-034	Southern Illinois U (05)	High-Temperature Studies of Automotive Friction Materials After FIeld and Laboratory Friction Tests	DUC	Peter Filip	C. Hubbard T. Watkins
2001-035	U of Tenn. (88)	Superhardness in Steel	RSUC	Charlie R. Brooks	T. Watkins
2001-036	Penn State U (28)	The Characterization of Crystalline Interphase Formed in Polyurethane/Al Bond	DUC	Earl R. Ryba	T. Watkins
2001-037	North Carolina State U (22)	Temperature and Wear Measurement in Diamond Wire Saw Machining of Wood and Ceramics	MITUC	Albert J. Shih	S. McSpadden R. Ott R. Dinwiddie
2001-038	E Spin Technologies, Inc.	Characterization of Structures of Nanofibers	MAUC	Jayesh Doshi	T. Nolan E. Lara-Curzio C. Hubbard
2001-039	Ohio U (8)	Thermal Behavior of Ion-Exchanged Iron Thiogermanates	TPUC	David Young	C. Hubbard
2001-040	U of Tenn. Space Inst. (89)	Transmission Electron Microscopy of Laser Coated Iron Oxide on Al-Alloy	MAUC	N. B. Dahotre	L. Allard
2001-041	ORNL (14)	Thermal Expansion Study of Rare-Earth Titanates	DUC	Lynn Boatner	C. Rawn
2001-042	U of South Carolina (4)	Residual Stress Determination in Friction Stir Welded 2024-T3 Aluminium Under Hot and Cold Welding Conditions.	RSUC	Michael A. Sutton	C. Hubbard D. Wang
2001-043	U of Tenn. Space Inst. (90)	Evaluation of Residual Stress in Laser Surface Engineered Carbide Coating on Aluminum	RSUC	N. B. Dahotre	T. Watkins
2001-044	Southern Illinois U (06)	Thermal Diffusivity of Carbon Composite Materials	TPUC	David T. Marx	R. Dinwiddie H. Wang
2001-045	Ford Motor Company (13)	Physical, Mechanical, and Microstructural Characterization of Ultrasonic Welding of Aluminum Components	MCAUC	Oludele Popoola	E. Lara-Curzio
2001-046	U of Tenn. (91)	Study of the Fundamentals of Oxidation and Nitridation of Two-Phase Cr-Pt Based Alloys	MAUC	Peter K Liaw	E. Payzant W. Porter L. Walker

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Proposal No.	Organization	Title	Lead Center	Spokesperson	Staff Contacts
2001-048	U of Tenn. (92)	X-Ray Diffraction Characterization of Carbon Fibers	RSUC	Thomas T. Meek	T. Ely
2001-049	U of Washington (06)	Comparison of Actual and Empirically/Analytially Examined Interfacial Shear Stresses and Matrix Residual Stresses in Ceramic Matrix	MCAUC	Michael G. Jenkins	E. Lara-Curzio
2001-050	ORNL CASD (15)	Crystallinity of Amorphous and Semicrystalline Syndiotactic Polypropylene	DUC	Brian K. Annis	E. Payzant
2001-051	ORNL Metals and Ceramics Division (16)	A TEM Study of Gas Absorbent Carbon Monoliths	DUC	Timothy D. Burchell	L. Allard D. Blom
2001-052	Siemens Westinghouse Power Corp. (14)	Evaluation of Properties of Thermal Barrier Coating Materials	TPUC	Andrew J. Burns	M. Ferber H. Wang W. Porter
2001-053	U of Tenn. (92)	Fatigue Damage Assessment of Nanocrystalline Copper	DUC	Peter K. Liaw	H. Wang C. Hubbard L. Allard W. Porter
2001-054	North Carolina State U (23)	Raman Study of Neutron Transmutation Doping of Diamond	MCAUC	Jagannadham Kasichainula	E. Lara-Curzio M. Lance
2001-055	Special Metals Corporation (2)	The Effect of Nb on the Sulfidation Resistance of Ni-Base Alloys	MAUC	Mark Andrew Harper	L. Walker H. M. Meyer
2001-056	Case Western Reserve U (02)	Evaluation of Doped Diamond-Like Carbon for Wear Resistant Applications and Doped Diamond for Electronic Applications	MAUC	Jeffrey T. Glass	K. More
2001-057	Stevens Institute of Technology (4)	The Influence of an Alpha-Alumina Template Layer, Deposited on Ni-Based Airfoil Alloys, on the Structure and Behavior of Thermally-Grown Oxides and EB-PVD YSZ Coatings	MAUC	Woo Young Lee	L. Allard
2001-058	Cummins Engine Company (19)	Base Metal Oxides for Adsorber Catalysts	TPUC	Roger D. England	T. Watkins E. Lara-Curzio
2001-059	ORNL SNS (17)	Influence of Recovery on the Recrystallization Textures in Interstial-Free Steel	RSUC	Xun-Li Wang	T. Watkins E. Payzant

HTML Proposal List (continued)

Proposal No.	Organization	Title	Lead Center	Spokesperson	Staff Contacts
2001-060	Tennessee Tech U (3)	Kinetics of Borosilicate Glass Deposition on Silicon	RSUC	Joseph J. Biernacki	C. Rawn J. Bai C. Blue
2001-061	Tennessee Tech U (4)	Phase Changes in Cement Materials Under Cryogenic Conditions	DUC	Joseph J. Biernacki	C. Hubbard C. Rawn J. Bai
2001-062	U of Tenn. (93)	Crystallization Mechanism of Ethylene-Octene Copolymers Under High Pressures	DUC	Paul J. Phillips	C. Rawn
2001-063	Westinghouse Savannah River	Residual Stress in Pinch Welds Processed by Various Means	MAUC	Paul S. Korinko	E. Lara-Curzio T. Watkins
2001-064	U of Alabama-Birmingham (20)	Microstructural Evolution in Non-Equilibrium Sputter Deposited Cu-W Alloys	DUC	Gregg M. Janowski	C. Hubbard
2001-066	Tennessee Tech U (5)	Lacro3-Type Perovskite Coatings for SOFC Interconnect Application	MAUC	Jiahong Zhu	L. Walker C. Rawn A. Payzant
2001-068	Brown U (02)	Nanoindentation Investigations of Hardness Gradients in CVD Diamond	MCAUC	B. W. Sheldon	L. Riester
2001-069	ORNL Chemical Technology Division (18)	Synthesis of Organically Modified Molecular Sieves	TPUC	Sheng Dai	C. Hubbard E. Payzant
2001-070	North Carolina State U (28)	Investigation of Wear and Manufacturing of Tools for Scrap Tire Recycling	MITUC	Albert Shih	S. McSpadden P. Blau C. A. Blue
2001-071	Clemson U (20)	Synthesis and Characterization of Ferroelectric Ceramic Nanoparticles	MAUC	Burtrand I. Lee	L. Allard E. Payzant
2001-072	Georgia Tech (40)	Microstructure of Laser CVD Carbon Deposits	MAUC	W. Jack Lackey	K. More
2001-073	Caterpillar, Inc. (15)	Elemental Segregation and Fracture Toughness in Steels	MAUC	Cynthia Kaichu Hsieh	H. Meyer
2001-074	Tennessee Tech. U (6)	Identification of Aggregates for Tennessee Bituminous Surface Courses	MITUC	L. K. Crouch	S. McSpadden
2001-075	Georgia Tech (41)	Temperature Distribution During the Surface Grinding of TItanium Aluminide	MCAUC	Thomas R. Kurfess	E. Lara-Curzio
2001-076	Synterials, Inc.	Quality Control of Interface Coatings	MAUC	Richard E. Engdahl	L. Allard P. Tortorelli
2001-077	United Technologies Research Center (1)	Effect of Surface, Subsurface, and Volume on the Resonance and Flex Properties of As800 Si_3N_4 Material	MAUC	Greg C. Ojard	M. Ferber H. Lin E. Lara-Curzio

HTML Proposal List (continued)

Proposal No.	Organization	Title	Lead Center	Spokesperson	Staff Contacts
2001-078	U of Kentucky(18)	Mechanical Properties of 3004 Aluminum	MCAUC	Peter P. Gillis	E. Lara-Curzio S. Viswanathan
2001-079	Clemson U (21)	Determination of Laser Damage to Cs Coated Gas Cathodes Used in Night Vision Systems	MAUC	David L. Carroll	H. Meyer
2001-080	II-VI Incorporated	Thermophysical Properties of Materials in Nd-Yag Crystal Growing System	TPUC	Elgin E. Eissler	H. Wang
2001-081	Alcatel Telecommunications	Evaluation of Graphite Degradation Due to Exposure to SiO (G) and O_2 (g) Resulting from the Vaporization of SiO_2 (S) at Elevated Tempeartures	MAUC	John A. Hanigofsky	L. Allard C. Hubbard D. Stinton K. More
2001-082	BWXT, LLC, Technology Development Division	Mechanical Assessment Process Integrity Qualification (MAP-IQ) Development	TPUC	Roland D. Seals	R. Dinwiddie
2001-083	U of Missouri–Columbia (2)	Finite Element Simulation of Residual Stress in A Spot Weld Using Temperature Dependent Physical and Mechanical Properties of the Material	TPUC	Sanjeev K. Khanna	C. Hubbard
2001-084	U of Nebraska (03)	Assembled Magnetic Materials	MAUC	Diandra L. Leslie-Pelecky	H. Meyer A. Pasto
2001-085	Auburn U (1)	Advanced Carbon Material for Thermal Insulation	TPUC	Ralph H. Zee	C. Hubbard H. Wang
2001-086	Smith & Nephew (02)	Frictional Testing of Oxidized Zirconium and Cobalt-Chromium Alloy Against Ultra-High Molecular Weight Polyethylene	MITUC	Daniel A. Heuer	P. Blau
2001-087	MRC	Tab Thin Film Chemical Analysis	MAUC	Paul S. Gilman	H. Meyer, III
2001-088	ORNL Metals And Ceramics Division (19)	Reduction of Thermal Expansion Anisotropy in Quaternary Mo_5Si_3 Intermetallics	DUC	Joachim H. Schneibel	T. Watkins
2001-089	Multi-Phase Services, Inc. (1)	Development of HIgh-Temperature Cr-Base Intermetallic Alloys for Structural Applications	MAUC	Robert H. Tien	L. Allard
2001-090	Clemson U (22)	High-Resolution Electron Microscopy Study on Solubilized Carbon Nanotubes	MAUC	Ya-Ping Sun	L. Allard
2001-091	North Carolina State U (29)	Metallic Glass Machining and Residual Stress Measurement	MITUC	Albert Shih	S. McSpadden C. Liu
2001-092	X-Ray Optical Systems (1)	Transmission of Polarized X-Rays Through Glass Capillary Fibers, Part 2	DUC	Raymond E. Benenson	C. Hubbard J. Bai

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Note: Asterisks indicate HTML staff members.

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Antti, M., and E. Lara-Curzio, "Effect of Notches, Specimen Size, and Fiber Orientation on the Monotonic Tensile Behavior of Continuous Fiber-Reinforced Oxide/Oxide Composites at Ambient and Elevated Temperatures," *Ceram. Eng. Sci. Proc*. **22** (3), 643–50 (2001).

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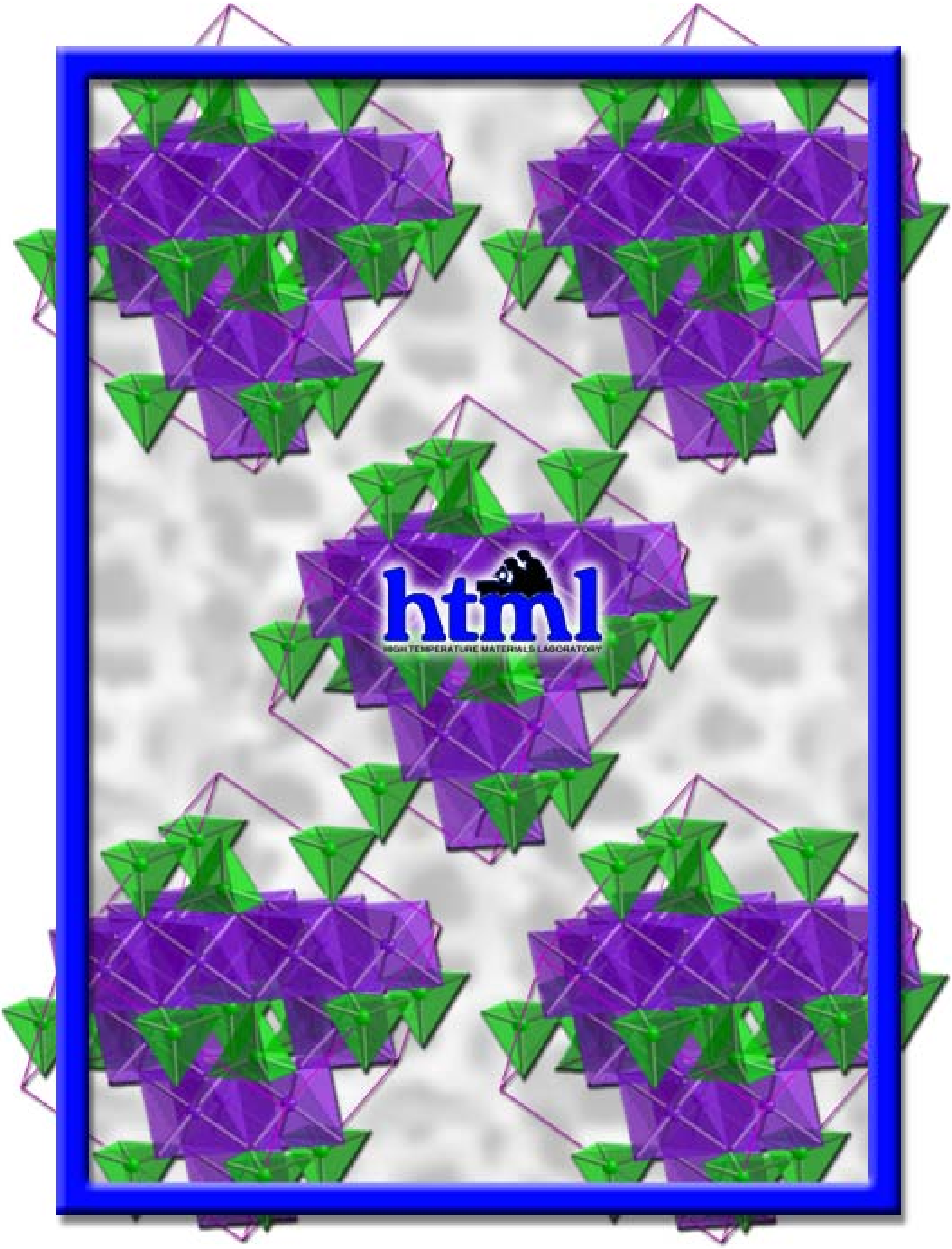
Front cover

Left: Image produced by a scanning electron microscope showing a titanium nitride (TiN) cuboid at the bottom of a pit on the fracture surface of a steel specimen. TiN was added to the steel during fabrication.

Right: A composite of individual auger maps of the same sample. Iron is shown in green, titanium in blue, and sulfur in red. The image shows that the TiN cuboids were responsible for initiating fracture and that sulfur was found at the interface between the iron and TiN.

Back cover

Structure of a $Ni_{1-x}Fe_{2+x}O_4$ spinel in which the nickel atoms are only found in the purple (octahedral) sites and the iron atoms are divided between the green (tetrahedral) and purple sites. The interchange of elements between sites during synthesis must be determined to fully interpret the properties of the spinels.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