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High Temperature Materials Laboratory

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ACRONYMS AND ABBREVIATIONS

ACEM	aberration-corrected electron microscope
AEM	analytical electron microscope
ANSWER	Advanced Neutron Scattering netWork for Education and Research
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
CBCF	carbon-bonded carbon fiber
CRADA	cooperative research and development agreement
DF	dark field
DLC	diamond-like carbon
DOE	U.S. Department of Energy
DSC	differential scanning calorimetry
DUC	Diffraction User Center
EDM	electrodischarge machining
EDS	energy-dispersive spectrometer
EE/RE	DOE Office of Energy Efficiency and Renewable Energy
ELSAM	Ernst Leitz Scanning Acoustic Microprobe
FDA	U.S. Food and Drug Administration
FEG	field-emission gun
FIB	focused ion beam
HFIR	High Flux Isotope Reactor
HTML	High Temperature Materials Laboratory
HVOF	high-velocity oxygen fuel
IC	integrated circuit
ICDD	International Centre for Diffraction Data
IR	infrared
LDRD	Laboratory Directed Research and Development
MAUC	Materials Analysis User Center
MBE	molecular beam epitaxy
MCAUC	Mechanical Characterization and Analysis User Center
MITUC	Machining, Inspection, and Tribology User Center
MRS	Materials Research Society
NDE	nondestructive evaluation
NSLS	National Synchrotron Light Source
NRSF	Neutron Residual Stress Mapping Facility
ODF	orientation distribution function
ORNL	Oak Ridge National Laboratory
OS	DOE Office of Science
RSUC	Residual Stress User Center
RUS	resonant ultrasound spectroscopy
SAN	scanning auger nanoprobe
SEM	scanning electron microscope
SNS	Spallation Neutron Source
SOFC	solid oxide fuel cell
STEM	scanning transmission electron microscope
TBC	thermal barrier coating
TEM	transmission electron microscopy

TMS	The Metallurgical Society, Inc.
TTPUC	Thermography and Thermophysical Properties User Center
USC	ultra-super-critical
USGS	U.S. Geological Survey
UTK	University of Tennessee, Knoxville
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 has been supporting the development of advanced materials since the mid-1980s.

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. The HTML also has a neutron beamline facility at the High Flux Isotope Reactor (HFIR) at ORNL and a synchrotron beamline at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory.

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 machine tools to investigate machining processes as applied to hard materials such as ceramics and special alloys. Special emphasis is given to grinding processes. These dynamometer-equipped machines provide unique capabilities for studying grinding and machining parameters and their roles in controlling the topography and mechanical and wear properties of the resulting surfaces.

Inspection capabilities include a high-frequency scanning acoustic microscope, 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 NSLS 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 NSLS, 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 HFIR at ORNL. This facility allows researchers to quickly measure and map the stress fields inside relatively large solid objects.

Thermography and Thermophysical Properties User Center (TTPUC)

TTPUC 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 online 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. He 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 97 new projects and developing 63 new user agreements. Table 1 presents the breakdown of these statistics.

Figure 1 depicts the continued growth in user agreements and user projects. Note that the total number of HTML proposals has now exceeded 1100. Also, the large number of new agreements bodes well for the future. At the end of this report is a list of proposals to the HTML and a list of agreements between HTML and universities and industries, broken down by state.

Table 1. Statistics of HTML Operations for FY 1999–FY 2002 (showing user proposals and user agreements)

FY	New Proposals				Cumulative Proposals			
	Total	Industrial	Academic	Other	Total	Industrial	Academic	Other
1999	91	39	49	3	906	387	497	22
2000	86	32	48	6	992	419	545	28
2001	92	33	48	11	1084	452	593	39
2002	97	35	56	6	1181	487	649	45
	New Agreements				**Cumulative Agreements**			
	Total	Industrial	Academic	Other	Total	Industrial	Academic	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
2002	63	5	56	2	642	358	261	23

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 the HTML User Forum, which was held in conjunction with the Advisory Committee (now called the Guidance and Evaluation Panel) Meeting in November 2002. The User Forum brought together many previous and current HTML users at the Knoxville Airport Hilton. Technical sessions featured talks on surface modification, coatings, and thin films. A workshop portion of the forum was held, in which HTML staff and users separately discussed the three topics and generated ideas for new techniques and equipment that HTML should acquire to meet user needs in these areas.

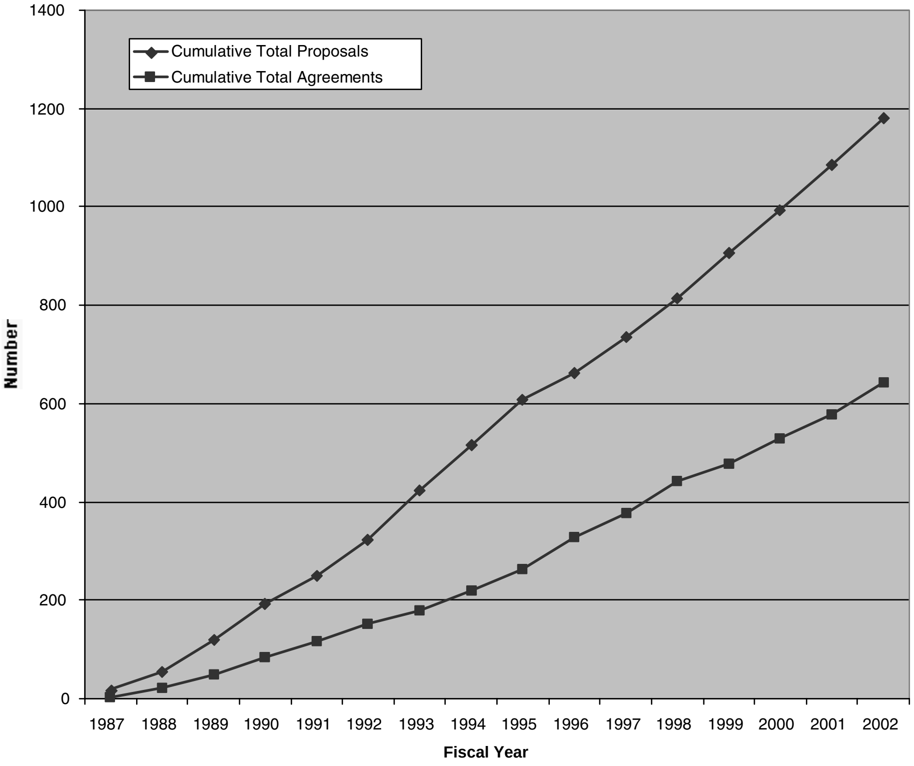


Fig. 1. Plot showing HTML User Program growth through user agreements and user proposals.

1. MATERIALS ANALYSIS USER CENTER (MAUC)

User Center Members
Larry Allard, Leader
Carolyn Wells, Secretary

Doug Blom
Dorothy Coffey
Bernhard Frost
David Joy
Harry Meyer
Karren More
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 HF-2000 FEG analytical electron microscope (AEM)
- Hitachi HD-2000 FEG scanning transmission electron microscope (STEM)
- JEOL 8200 electron microprobe with five wavelength-dispersive X-ray spectrometers
- PHI 680 scanning auger nanoprobe (SAN)
- Hitachi FB-2000 focused ion beam (FIB) micromill
- JEOL 2010F FEG TEM with EDS
- Leica UCT ultramicrotome with cryosectioning capability

MAUC News

Prototype Aberration Corrector for New ACEM Tested in Heidelberg, Germany

In mid-May, Dr. Larry Allard traveled to visit CEOS Co. in Heidelberg, Germany, to test the prototype version of the aberration corrector being designed for the HTML's aberration-corrected electron microscope (ACEM) from JEOL Co. (Fig. 1-1).

The corrector was installed on the electron optical column of a JEOL 2010F field-emission transmission electron microscopy (TEM) that was outfitted with a scanning system to also permit STEM imaging.

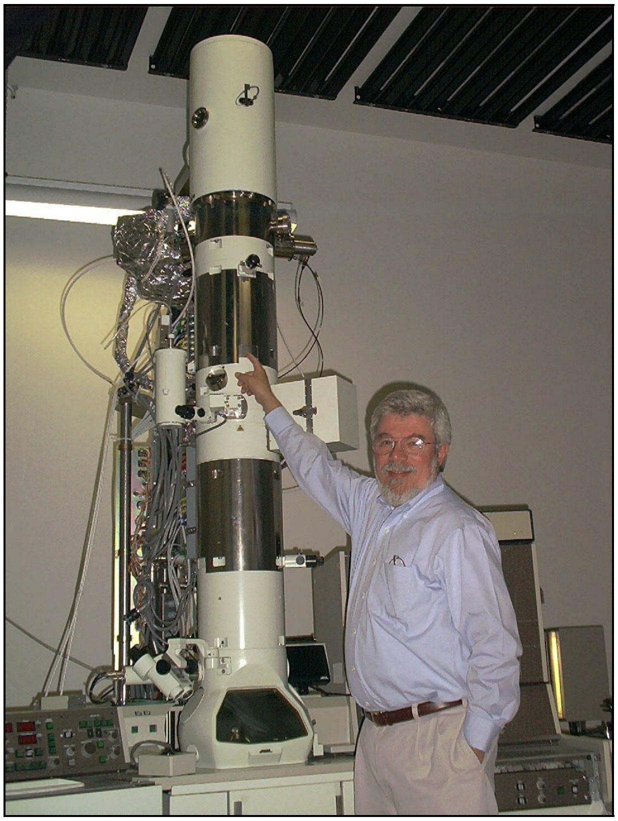


Fig. 1-1. Larry Allard pointing to the corrector element designed for HTML's aberration-corrected electron microscope.

Because the ACEM is designed to provide ultimate resolution at the 0.7Å level in STEM dark-field (DF) mode, the HTML corrector was installed in the illuminating system of the microscope. The instrument also had a corrector for the standard TEM image, installed just below the objective lens.

The software for correction of the TEM image was fully developed and demonstrated successfully. The alpha version of the software for correction of the STEM DF image was still under development, but enough control was available that proof-of-principle of the alignment and correction procedures was successfully demonstrated.

After acceptance by JEOL Co., the commercial version of the corrector will be fabricated and shipped to the factory in Japan for assembly on the base column of the ACEM instrument. To date, the construction schedule is on time.

JEOL 8200 Electron Microprobe Begins Operation

The new JEOL electron microprobe was commissioned for beneficial operation in mid-summer 2002. This instrument is equipped with five wavelength-dispersive spectrometers, as opposed to the old JEOL 733 probe, which was a three-spectometer system. A sophisticated software package coupled with a precision specimen stage, the new machine can be set up for automated, unattended operation and often is run overnight (Fig. 1-2). This extended operation facilitates data collection so that a significantly higher sample throughput is possible. The instrument was funded primarily through DOE Office of Energy Efficiency and Renewable Energy (EE/RE) and Office of Science (OS) programs. HTML funds supported the installation costs and will support future service contracts.

Update on New Advanced Materials Characterization Laboratory

Plans have been made for a new microscopy laboratory specially designed with the extremely quiet environment needed for ORNL's current and future ultrahigh-resolution, aberration-corrected electron microscopes. The original plans had to be significantly revised after bids from three contractors came in more than $2M too high for the available budget. Revised plans for a four-instrument laboratory that can be constructed with the available remaining budget are under development. The projected completion date is December 2003.

Fig. 1-2. Larry Walker operating the new JEOL 8200 electron microprobe.

JEOL 2010F Field-Emission TEM Is Being Installed at HTML

As part of the aberration-corrected electron microscope (ACEM) project, JEOL Co. delivered a new 200kV loaner instrument, the 2010F, to the HTML in early 2002. Unfortunately, the first instrument delivered was severely damaged during shipping, and JEOL had to return the instrument to Japan.

However, a second instrument arrived in excellent shape within 3 months (Fig. 1-3). This machine will provide a high-resolution scanning transmission DF imaging capability. This capability will allow HTML staff to learn STEM imaging techniques and to develop remote operation techniques in preparation for the delivery of the ACEM, expected in early fall 2003.

A best resolution of 1.4Å in DF mode should be achievable, limited only by the effects of the local environment.

Fig. 1-3. New 201F provides high-resolution scanning transmission dark-field imaging capability.

Microsampling Technique Added to Focused Ion Mill

A new technique for site-specific sample selection on the Hitachi focused ion beam (FIB) mill has recently been introduced.

Designed by MAUC consultant Prof. W. C. Bigelow, a special microsampler allows the operator to microsurgically choose a sample in the range of 10 mm square from anywhere on the sample (Fig. 1-4). The operator can extract it in situ for final milling to electron transparency. The technique promises to be useful in most of the user projects that require FIB milling.

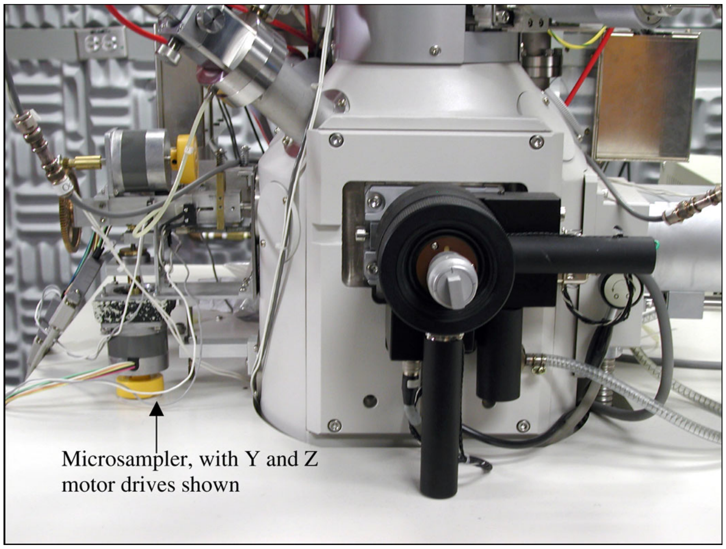


Fig. 1-4. Microsampler provides new technique for projects requiring FIB milling.

Selected Highlights

Failure Methodology for Ferro-Phos Powder Metal Parts—DaimlerChrysler

Industrial Collaborator: Dale Wetzel
HTML: Dr. Harry Meyer

This project was an attempt to investigate and determine the mechanism leading to embrittlement of a powder metal part made from a mixture of iron (Fe) and iron phosphide (Fe_3P) powder. Final parts made from this powder mixture contained approximately 0.45 wt% of phosphorus and that phosphorus was dispersed uniformly within the iron matrix. The “ferro-phos” alloy was chosen specifically for manufacturing these parts as a compromise between cost and materials properties, namely strength and ductility.

On occasion, particular batches of parts from the vendor were too brittle and did not stand up adequately to their use environment. Embrittlement was solved by either adding graphite powder before consolidation, or by performing the consolidation in a hydrogen-rich atmosphere. The goal of this project was to understand the reason for the embrittlement so that these additional processing steps or conditions might be eliminated.

Scanning auger microscopy was used to examine fracture surfaces of “good” and “bad” material. Figure 1-5 shows the secondary electron images of (a) good and (b) bad material from samples fractured in situ. The micrographs identify the types of surface morphologies observed.

Figure 1-6 shows false color images of the combined elemental maps. The areas in yellow result from the overlap of the Fe (red) and P (green), while the areas that show up purple result from the overlap of Fe (red) and O (blue).

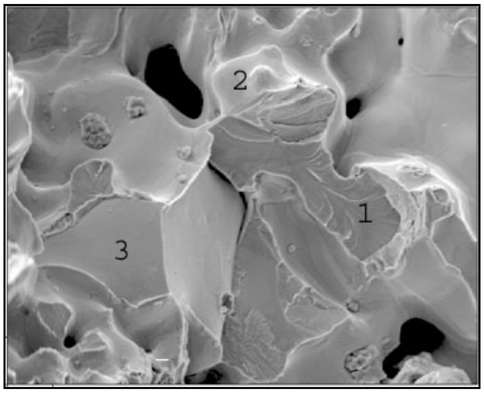


Fig. 1-5 (a). Fracture surface of “good” material: (1) fractured particle, (2) particle surface, (3) grain boundary.

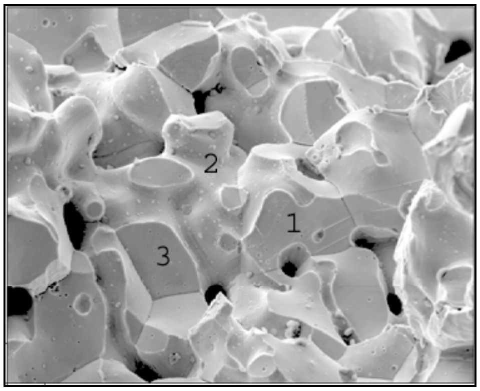


Fig. 1-5 (b). Fracture surface of “bad” material: (1) fractured particle, (2) particle surface, (3) grain boundary.

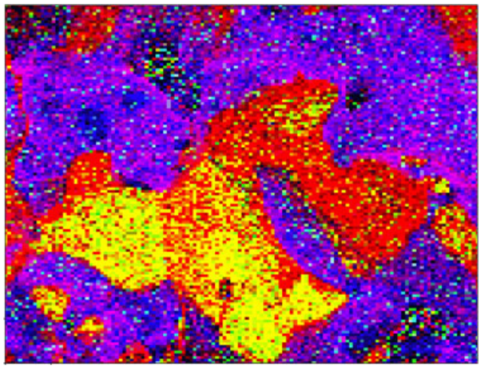


Fig. 1-6 (a). Auger elemental map of “good” material; red areas = Fe; blue areas = O; green areas = P.

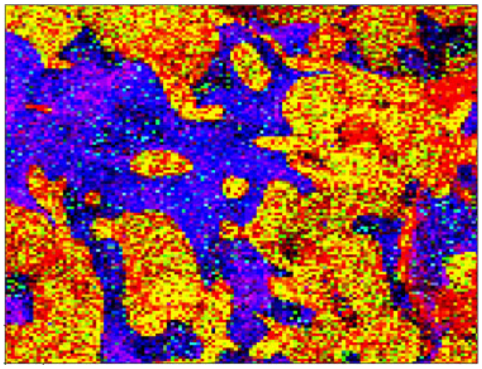


Fig. 1-6 (b). Auger elemental map of “bad” material; red areas = Fe; blue areas = O; green areas = P.

These elemental maps show that P preferentially segregates to some fracture surfaces. This segregation was an unexpected result and is most likely the reason that the material was embrittled. The bad material showed a large fraction of the fracture surface area with segregated P, while the good material had a much lower fraction of segregated P. The added graphite or the hydrogen atmosphere apparently work in some way to reduce the amount of unwanted segregated P.

Grain-Boundary Segregation Studies in Doped Strontium Titanate Capacitors—Honeywell FM&T

Industrial Collaborator: Dan Krueger
HTML: Dr. Douglas Blom

This project characterizes the grain size and grain boundary segregation in Y_2O_3-doped $SrTiO_3$. Doped $SrTiO_3$ is a candidate material for capacitors with both a high-voltage breakdown and high-dielectric constant. Honeywell has developed a processing route to produce good quality capacitors from doped $SrTiO_3$ that are a hundredfold improvement over undoped $SrTiO_3$. However, these capacitors need detailed chemical characterization and measurements of the grain sizes to further improve their materials.

The capabilities of the Hitachi HF-2000 FE-TEM and the Hitachi HD-2000 FE-STEM are ideally suited to making line-profile measurements to quantify the segregation of minor dopants to the grain boundaries in advanced materials. The fine electron probe is scanned in a line across a grain boundary, and the X rays produced are analyzed to measure the composition of the material at the nanometer scale.

The HF-2000 was used for both good quality bright-field images of the grain size and morphology as well as some high-resolution lattice images of the grain boundaries to confirm the lack of second phases in these samples (Fig. 1-7).

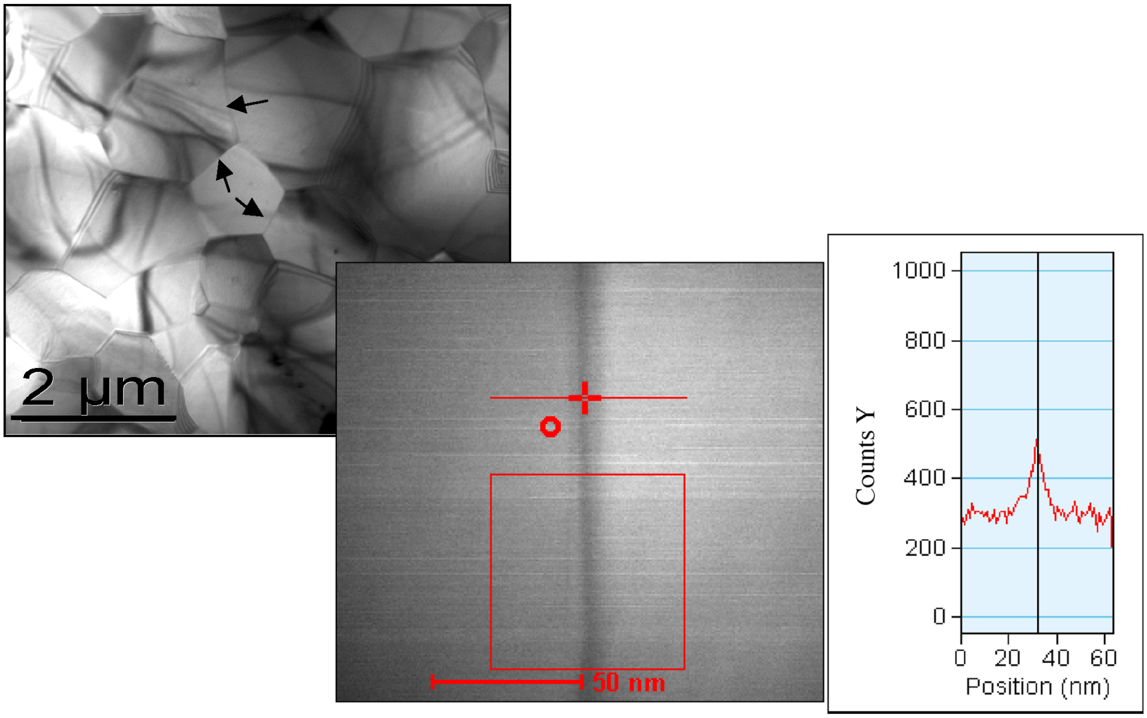


Fig. 1-7. TEM bright-field image of thin foil of Yttria-doped $SrTiO_3$ microstructure. Grain boundaries of the sort arrowed were characterized via EDS analysis on the Hitachi HD-2000 dedicated STEM.

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

User Center Members

Sam McSpadden, Leader
Susan Thomsen, Secretary

Peter Blau
Tyler Jenkins
Lawrence O'Rourke
Jun Qu
Randy Parten
Earl Shelton

MITUC provides basic facilities for investigation of machining processes for high-performance materials, design and fabrication of test specimens in support of other user centers, dimensional characterization of test specimens and other components, and tribology.

Several types of numerically controlled machine tools are available to guest researchers for their projects at MITUC. The machine tools were selected for their similarity to those used in manufacturing facilities throughout the United States. Most equipment is instrumented to permit real-time measurement of key machining 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

Machining

- Chand-Kare grindability test system
- Instrumented ProLIGHT 3000™ tabletop CNC turning center
- Instrumented Benchman® VMC4000 tabletop CNC vertical machining center
- Instrumented Cincinnati Sabre multi-axis grinder
- Instrumented K.O. Lee creep-feed grinder
- Instrumented Nicco creep-feed grinder
- Instrumented Weldon cylindrical grinder

Inspection

- EMD Legend integrated metrology center
- Mahr formtester
- Nikon optical comparator
- Rodenstock Model 600 laser surface profile-measuring system (noncontact)

- Taylor Hobson Talysurf 120 stylus surface-profile-measuring system (contact)
- Kræmer Scientific Instruments SAM2000 scanning acoustic microscope

Tribology

- Cameron-Plint Model TE-77 reciprocating sliding wear tester
- Cameron-Plint TE-53 multimode friction and wear tester
- Compact grindability test system
- CSEM instrumented scratch tester
- High-temperature pin-on-disk system
- Microabrasive wear testing machine
- Microfriction apparatus
- Pin-on-disk friction and wear resting stations
- Reciprocating friction and wear tester
- Repetitive impact testing system
- Sub-scale instrumented brake tester

Selected Highlights

Grinding Wheel Performance Evaluation on the K.O. Lee Creep-Feed Surface Grinder

The K.O. Lee creep-feed surface grinder was used to evaluate grinding wheel performance on three separate user proposals submitted by grinding wheel manufacturers. Two of the projects involved tests to compare the performance of groups of superabrasive cubic-boron-nitride grinding wheels. The third project was aimed at developing an improved conventional, inexpensive, aluminum-oxide grinding wheel for grinding hardened 52100 tool steel. This material is widely used in the automotive industry.

Wheel manufacturers are concerned with providing wheels that are durable, consistent in composition, inexpensive to produce, and capable of grinding large numbers of high-quality parts with little variation in dimensions and surface texture. Perhaps the most important measure of a wheel's performance relative to these goals is its wear characteristics. The ratio of the volume of workpiece material removed to the volumetric wheel wear is known as the grinding ratio or G-ratio.

A very high G-ratio is desirable. This ratio means that the number representing the volume of worn wheel material will be small relative to the number representing the volume of workpiece removed. In laboratory testing, it is desirable to minimize the amount of workpiece material removed so that test times and waste-disposal costs can be kept to a minimum. Thus, it is important to develop accurate measurements of wheel wear to minimize error and ensure repeatability of test results.

Although the wheels were quite different in the three user projects, the same basic test methods were used on all three projects. First, the wheel was mounted on the grinding spindle, trued, balanced with an automatic balancer, and dressed. Truing is the process of selectively removing material from the periphery of the grinding wheel to ensure that it has minimal run-out (i.e., the wheel "runs true" to the centerline of the grinding spindle). A wheel that is not properly trued and balanced will generate parts that have a poor surface finish. Dressing is the process of removing a thin layer of abrasive material and bond material to ensure that the abrasive grains are sharp and exposed to the workpiece.

The primary purpose of the grinding wheel performance evaluation test is to generate measurable wear on the grinding wheel under controlled grinding conditions. A typical machine setup is shown in Fig. 2-1.

The wheel rotates in a clockwise direction at a fixed speed. The wheel is automatically balanced using a microprocessor-controlled balancer. The workpiece is mounted in a vice atop a dynamometer, and the dynamometer is held to the grinder worktable by an electromagnetic chuck. The worktable reciprocates to the left and right.

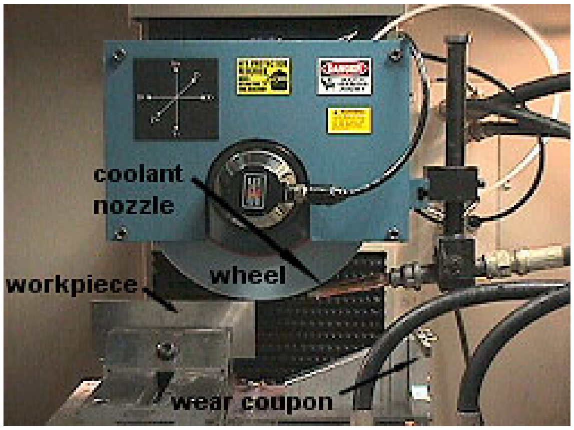


Fig. 2-1. Typical setup for measuring grinding wheel performance on the K.O. Lee creep-feed surface grinder.

Coolant is delivered through the nozzle at the right of the wheel. Coolant velocity is adjusted so that it closely matches the wheel velocity. The wheel is moved downward a small amount just before the worktable moves from left to right and is then raised up slightly while the table returns to its starting position on the left. This movement ensures that grinding is done in one direction only, and this mode of grinding is referred to as “up” grinding. Tangential and normal forces and grinding spindle power consumption are measured during each grinding cycle. A fixed volume of workpiece material is removed during a predetermined number of grinding cycles.

The wheel is then moved under computer control until it is positioned above the wear coupon. Because the wheel is wider than the workpiece, the center portion of the wheel exhibits wear, while the edges do not. The wheel is plunged into the wear coupon, and a mirror image of the profile is imparted to the coupon.

A stylus-type surface profiling instrument, called the Taylor Hobson Talysurf Model 120, generates a wear profile such as the one shown in Fig. 2-2. The profile can be generated with very good accuracy by tracing the wear coupon. The grinding wheel diameter is known, and the area under the wear profile curve is assumed uniform over the entire circumference of the wheel. Therefore, the volume of wheel wear can be easily calculated.

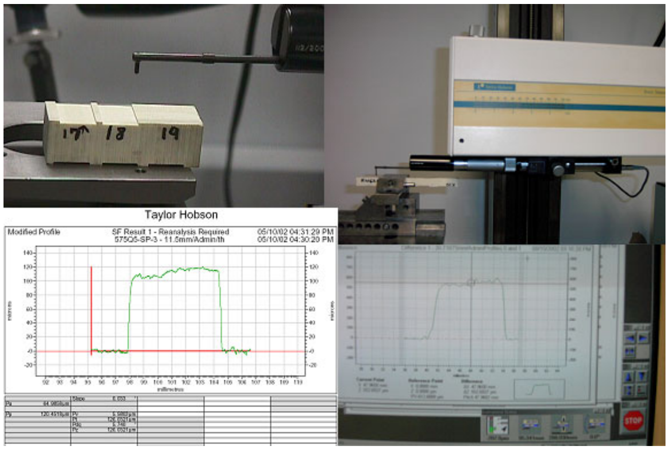


Fig. 2-2. Profile of wheel wear coupon measured using a Talysurf profiling instrument. The volume of grinding wheel wear is calculated from this profile.

Finally, the grinding ratio is computed as the ratio of the volume of workpiece removed to the volume of grinding wheel consumed during the test. This sequence is repeated for each wheel that is being tested and for each set of grinding conditions being evaluated. The wheels can then be ranked according to their wear characteristics.

Figure 2-3 compares the performance of seven different grinding wheels in terms of grinding ratio. A high G-ratio indicates a more cost-effective grinding process in which the wheel wear is low. All seven wheels were operated at a controlled specific material removal rate of Q'=0.5 in.^{3}/min./in. [~5.4 mm^3/s/mm]. Each wheel was manufactured using a slightly different composition of cubic boron nitride abrasive. Wheels identified as "W577-1" and "W580" clearly outperformed the remaining wheels in terms of wear.

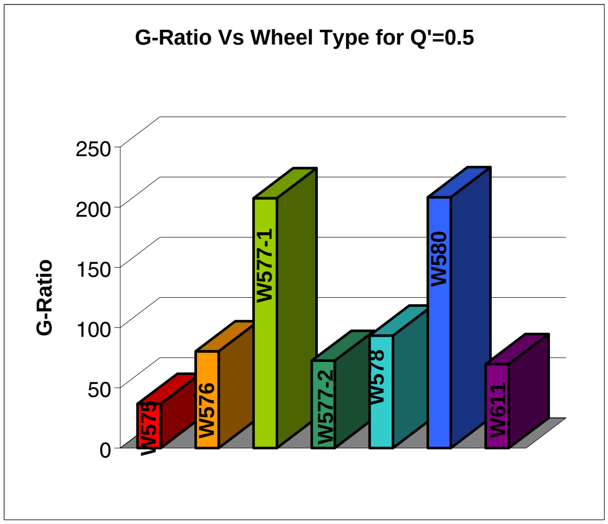


Fig. 2-3. Performance of seven different superabrasive grinding wheels compared based on grinding ratio.

Other measures of wheel performance include specific grinding energy, grinding efficiency, normal and tangential grinding forces, and surface finish. Each of these variables was measured for all seven wheels. The overall wheel performance can be determined quantitatively by assigning a weighted value to each of the above variables and ranking the wheels based on their relative performance for each variable.

The surface roughness of the workpiece is also an important consideration in evaluating wheel performance. Roughness is strongly influenced by the type of wheel used and the grinding conditions chosen. Surface finishes are measured using the same Talysurf instrument used to measure the wear coupon profiles. In addition to being measured for surface roughness, each workpiece is evaluated qualitatively for burrs, evidence of surface regions that have overheated (burning), and repetitive surface imperfections (chatter).

The normal and tangential forces measured during the grinding tests are evaluated to ensure that the wheel performance is consistent throughout the tests and that wheel wear occurs in a linear fashion. The spindle power consumption is measured and recorded during each grinding test. Spindle power can be used to compute the amount of energy required to remove a specific volume of workpiece material. This amount is another important factor in comparing the overall performance of two or more grinding wheels.

Such performance tests for a small group of grinding wheels can last several days or even weeks. Currently no widely accepted standard method exists for measuring wheel wear or determining wheel performance. During the coming year, some of the techniques developed at the HTML for measuring wheel wear will be refined and offered as part of an American Society for Testing and Material- (ASTM-) recommended practice guide for the measurement of wheel wear.

New User Center Capabilities

Scanning Acoustic Microscope Upgraded

The Ernst Leitz scanning acoustic microscope (ELSAM) has been upgraded with a new dual-computer-system and control/imaging software package (Fig. 2-4). Because this microscope is now manufactured by Kræmer Scientific Instruments, GmbH, the microscope will be called the SAM2000 Scanning Acoustic Microscope (SAcM). Scanning acoustic microscopy uses high-frequency acoustic waves to generate visual images of both the surface topography and the underlying structure of materials by detecting reflected echoes, shown schematically in Fig. 2-4. The fully functional SAcM has working frequencies from 100 MHz to 2.0 GHz, offering spatial resolution up to 0.4 µm. This resolution is the highest available for commercial scanning acoustic microscopes in the world. Some specifications are listed in Table 2-1. A higher working frequency provides better resolution, but sacrifices the maximum penetration depth.

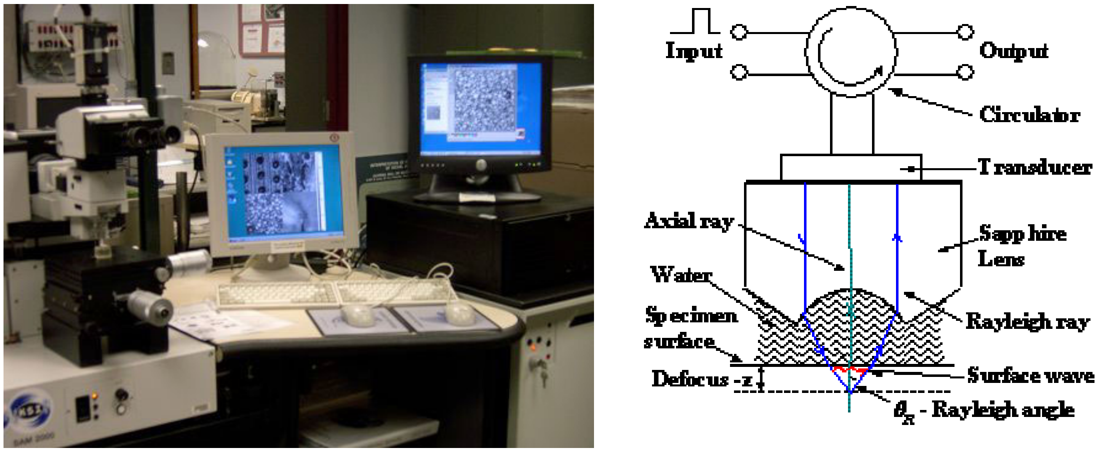


Fig. 2-4. The upgraded Kræmer Scientific Instruments SAM2000 scanning acoustic microscope (l). The principle of operation is shown schematically (r).

Table 2-1. Specifications of KSI SAM2000

Working frequency (Hz)		**100 M**	**200 M**	**400 M**	**1.0 G**	**2.0 G**
Resolution (µm)		8	4	2	0.8	0.4
Max penetration depth* (µm)	Metals	60~100	40~80	30~60	15~25	2~4
	Ceramics	40~80	25~65	20~50	10~20	1~3

*The penetration capability of the acoustic wave highly depends on material properties.

Figure 2-5 shows examples of surface and subsurface images in a nickel alloy specimen coated with nickel-aluminide. By focusing on the surface, Fig. 2-5(a) and the interface, Fig. 2-5(c), the coating thickness was determined to be about 30 mm.

Using SAcM offers two distinct advantages when compared to optical or scanning electron microscopy:

- non-destructive subsurface features detection (lying in the ability of the acoustic waves to penetrate opaque materials) and
- non-destructive mechanical properties visualization and analysis.

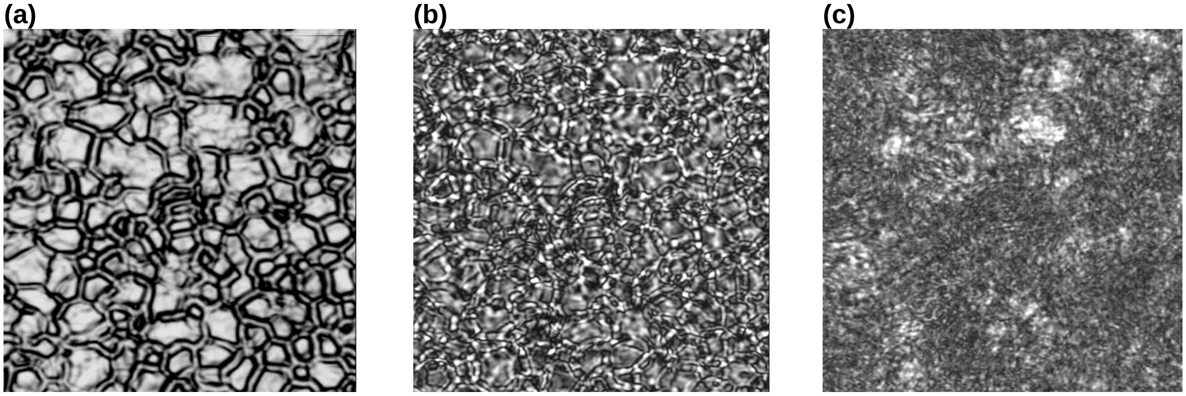


Fig. 2-5. Examples of images generated while measuring the thickness of a NiAl coating on a Ni alloy sample. Working frequency: 400 MHz, coating thickness: 30 mm. (a) Focused on the surface. (b) Focused below the surface. (c) Focused on the interface.

Elaborating further on these advantages, the SAcM can

- generate images and measure subsurface features of both transparent and opaque solid bodies;
- measure the thickness of coatings or thin layers (the minimum measurable thickness can be as small as a few microns depending on different materials);
- evaluate subsurface damage such as cracks caused by machining (both the size and angle of a crack can be measured);
- analyze microelectronic interconnections and packaging;
- generate images and analyze elastic properties;
- visualize residual stress and strain distribution (a special lens is needed for quantitative measurement);
- characterize both isotropic and anisotropic materials; and
- probe biological cells and tissues.

Tabletop Turning and Machining Centers

Machining capabilities at the MITUC have been expanded with the purchase of the Benchman 4000™ vertical machining center and the ProLIGHT 3000™ computer-controlled lathe (Fig. 2-6). The two tabletop machines provide turning, milling, drilling, and tapping capabilities.

These miniature machine tools provide much-needed new capabilities for machining research in areas other than grinding. They have most of the capabilities of full-scale industrial machines, but have the advantages of a very small footprint, low initial cost, ease of operation, and extremely low operating and maintenance costs. The vertical machining center has been instrumented with a Kistler dynamometer that is capable of measuring both force and torque. Similar instrumentation is planned for the turning center.

Fig. 2-6. The Benchman 4000™ CNC vertical machining center (l) and the ProLIGHT 3000™ CNC turning center. Both provide much-needed new machining capabilities.

3. MECHANICAL CHARACTERIZATION AND ANALYSIS USER CENTER (MCAUC)

User Center Members

Edgar Lara-Curzio, Leader
Paula D. Irizarry, Secretary

James G. Hemrick
Ken C. Liu
Ralph L. Martin
Miladin Radovic
Laura Riester
Christopher O. Stevens
Bob Swindeman

The MCAUC specializes in the mechanical characterization of functional and structural materials. The center 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 in controlled environments and at elevated temperatures using standard or customized specimens. Facilities also include equipment for micromechanical testing and instrumented indentation. The MCAUC staff has expertise with a wide variety 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 500kN 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 computerized data acquisition
- Fixtures for uniaxial and biaxial bending (ring-on-ring), Iosipescu shear testing, anti-buckling compression, 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 vacuum or controlled environments
- Rotary bend fatigue machine for testing cylindrical specimens in fully reversed cyclic loading
- Environmental test facility for testing in vacuum (down to 1×10^{-9} torr), inert gas environments or pressurized simulated industrial environments
- Microturbine test facility for assessing the effects of stress and temperature on the durability of materials for microturbine components

- Acoustic emission detectors, resonant ultrasound spectrometer and impulse excitation instrumentation for characterization of mechanical integrity and elastic properties of materials and components
- Electromechanical and micromechanical universal test machines for composite materials and their constituents
- Instrumented indenters for hardness measurements including mechanical properties microprobe (nanoindenter) for measuring contact stiffness, elastic and plastic properties, and fracture resistance of thin films and small volumes of material
- Interfacial test system for evaluation of composite interfacial properties by means of single-fiber push-in
- Work station for stress analysis and life-prediction (ERICA/CERAMIC+CARES)

Creep-Induced Microstructural Changes in Mg-Al-Sr Alloys—Noranda Corporation

Industrial Collaborators: Eric Baril and Emanuelle Landriault
HTML: Ken Liu, Laura Riester, and Edgar Lara-Curzio

As part of ongoing efforts to reduce the weight of vehicles and improve energy efficiency, Noranda is developing creep-resistant die-casting magnesium alloys for drive-train components. To achieve this objective, Noranda is collaborating with HTML researchers to identify the mechanisms that control the rate of creep deformation of these alloys and their relationship with the microstructure of these materials. The analysis of creep tests carried out at the HTML revealed that a new family of Mg-Al-Sr alloys is at least two orders of magnitude more creep resistant than the well- characterized alloy system of Mg-Al-Zn (AZ91).

To understand the role of Sr on the improved creep resistance, Noranda researchers Eric Baril and Emanuelle Landriaut worked with MCAUC researcher Laura Riester to determine the mechanical properties of individual grains of Mg-Al-Sr alloys samples using a nanoindenter (Fig. 3-1). These test specimens had been subjected to creep deformation for various periods of time at temperatures as high as 150°C.

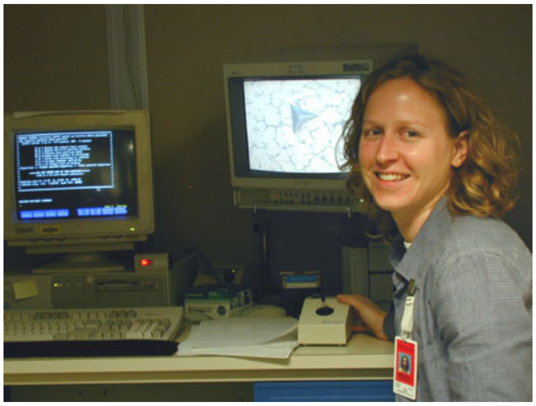


Fig. 3-1. Emanuelle Landriault of Noranda using a nanoindenter to probe the mechanical properties of individual grains of Mg alloys.

Typical grain size in these materials ranged between 5 and 10 microns. Although the results revealed a significant amount of variability, preliminary analyses indicate that the hardness was highest for samples that had failed during creep testing. These results are consistent with the development of dislocations in this material during creep, and will be further analyzed using microstructural information obtained by means of transmission electron microscopy.

Effect of Processing Conditions on the Mechanical Properties of Si_3N_4/SiC Nanocomposites—University of California-Irvine

University Collaborators: Amiya Mukherjee and Matt Gasch
HTML: Ken Liu, Edgar Lara-Curzio, and Laura Riester

Professor Amiya Mukherjee and his group at the University of California-Irvine have been collaborating with MCAUC researchers to study the effect of processing parameters on the creep resistance of Si_3N_4/SiC nanocomposite materials synthesized by electric field-assisted sintering of polymer powder and liquid polymer precursors. The resulting materials consist of a fine-grained matrix of Si_3N_4 (300–500 nm) grains with nanocrystalline (100 nm) SiC particles at the grain boundaries and within Si_3N_4 grains (Fig. 3-2). Some of the most promising results are for materials synthesized in which grain boundaries are partially or completely free of oxide grain boundary phases.

Fig. 3-2. Microstructure of Si_3N_4/SiC nanocomposites.

In addition, graduate student Matt Gasch worked with MCAUC researchers Ken Liu, Edgar Lara-Curzio, and Laura Riester to determine the effect of processing parameters on the elastic modulus, hardness, and fracture resistance of these materials.

Specifically, samples with various amounts of sintering additives (8%, 5%, 3%, 1%, and 0%) were evaluated using a nanoindenter. Significant differences were found in the value of the elastic modulus and hardness among the specimens evaluated.

These differences can be explained by the differences in the microstructure of these materials, which have been characterized by high-resolution electron microscopy.

Comparison of Two Techniques for Determining the Young's Modulus of Materials—Georgia Tech

University Collaborators: Professor Thomas R. Kurfess and Wesley Stone
HTML: Laura Riester

Professor Thomas R. Kurfess and his group at Georgia Tech are developing analytical models to simulate the temperature distribution in workpieces during grinding, specifically when using cubic boron nitride for the machining of titanium aluminide (TiAl) alloys (Fig. 3-3). To support these activities they are collaborating with HTML researchers to determine the temperature dependence of the elastic properties of TiAl using resonant ultrasound spectroscopy (RUS).

Radovic Joins MCAUC Group

Dr. Miladin Radovic joined MCAUC as a postdoctoral researcher on November 2001 to support a DOE Fossil Energy-sponsored project to evaluate the mechanical properties of

Fig. 3.3 Wesley Stone of Georgia Tech evaluating the elastic properties of TiAl.

solid oxide fuel cell (SOFC) materials and components and to develop life and reliability prediction models under the direction of MCAUC researcher Edgar Lara-Curzio. Dr. Radovic received a Ph.D. in materials engineering from Drexel University under the guidance of Professor Michael Barsoum.

Hemrick Joins MCAUC Group

On October 17, 2001, James G. Hemrick presented a seminar entitled "Time-Dependent Deformation Behavior of Cast Alumina Refractories," which was based on his doctoral dissertation work at the University of Missouri-Rolla under the guidance of Professor Bob Moore (Fig. 3-4). The experimental part of his thesis was carried out at the HTML with financial support of DOE's Office of Industrial Technologies. Hemrick joined MCAUC as a postdoctoral researcher in January 2002 to work under the direction of MCAUC researcher Edgar Lara-Curzio.

Fig. 3-4. James Hemrick presenting a seminar at the HTML.

Invention by Lara-Curzio and Coworkers 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 in November 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.

ORNL Researchers Play Role in New Knee Implant—Smith & Nephew

Industrial Collaborator: Marc Long
HTML: Peter Blau and Laura Riester

The most effective surgery for a worn-out knee is total knee replacement. This procedure involves removal of the diseased areas and replacement of the entire joint surface with metal and plastic components. The U.S. Food and Drug Administration (FDA) recently approved a new material developed by Smith & Nephew of Memphis, Tennessee, for knee replacement.

Wear and mechanical testing of this material, prior to FDA approval, was performed at the HTML as part of a user project, by Smith & Nephew's Marc Long and HTML researchers Peter Blau and Laura Riester (Fig. 3-5).

Ridger Life /HEALTH

New 'ceramic' joint for arthritic knees shows great promise

ORNL researchers play role in new implant

Fig. 3-5. ***Oak Ridger*** **article on February 27, 2002, highlighting HTML testing of a new material for knee replacements.**

The new Zr-based alloy, which after an oxidation treatment develops a thin layer between 5 and 10 microns of zirconia, is ideal for this application because of its high wear resistance.

Reducing Ingot Cracking During Aluminum Casting—University of Kentucky

University Collaborators: Peter Gillis and Mohammed Hassan
HTML: Ken Liu, Chris Stevens, and Edgar Lara-Curzio

During aluminum casting, ingot cracking often occurs from a lack of liquid feeding of the mushy zone (liquid-solid region). This cracking occurs especially at the end of solidification when grains start to impinge and finally touch one another, but are still surrounded by a continuous liquid film.

To better understand ingot cracking, MCAUC researchers Ken Liu, Chris Stevens, and Edgar Lara-Curzio are collaborating with University of Kentucky professors Peter Gillis and Mohammed Hassan (Fig. 3-6). Their HTML user project is focused on studying the mechanical behavior of 3004 aluminum alloys in the mushy zone. Using sensitive extensometry, quartz-lamp furnaces with rapid heating capabilities, and acoustic emission detectors, the stress-strain-time response of the material was characterized to generate data in support of viscoplastic modeling efforts. These efforts are expected to lead to a better understanding and elimination of this problem.

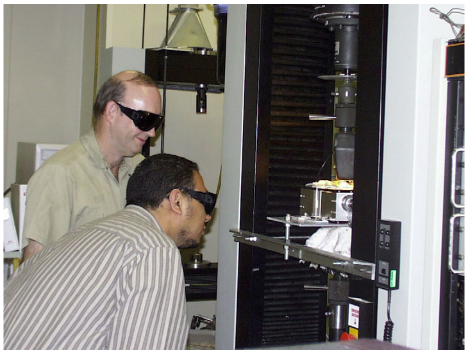


Fig. 3-6. MCAUC's Chris Stevens (l) and Mohammed Hassan studying ingot cracking.

Tensile Strength of Ceramic Foam Ligaments—Texas A&M University

University Collaborators: Ozden Ochoa and Rogelio Verdugo
HTML: Edgar Lara-Curzio

Professor Ozden Ochoa and graduate student Rogelio Verdugo of Texas A&M worked with MCAUC researcher Edgar Lara-Curzio to characterize the mechanical behavior of ceramic foam ligaments (Fig. 3-7). In particular, they focused on the determination of the ligaments' Young's modulus and tensile strength. Ceramic foams have attracted substantial interest because of their potential uses in thermal management and filtration of molten metals and gases.

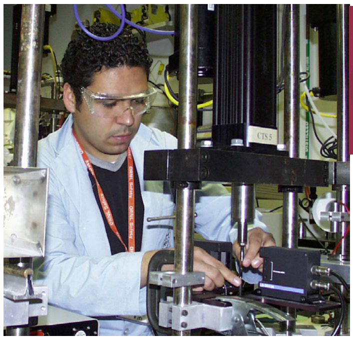


Fig. 3-7. Rogelio Verdugo evaluating ceramic foam ligaments.

As part of this user project, techniques were developed to obtain undamaged single ligaments from carbon and chemically vapor deposited silicon carbide foams with either 20 or 45 pores per inch. The results from this investigation will be used to predict the response of components fabricated with these materials.

To determine the magnitude of the tensile strength of each ligament and its strength-limiting flaw, their fracture surfaces were analyzed by scanning electron microscopy (Fig. 3-8). A sufficiently large number of specimens were evaluated to estimate the parameters of the distribution of tensile strengths. The results from this work were correlated with macroscopic measurements obtained from the diametral compression of bulk foams.

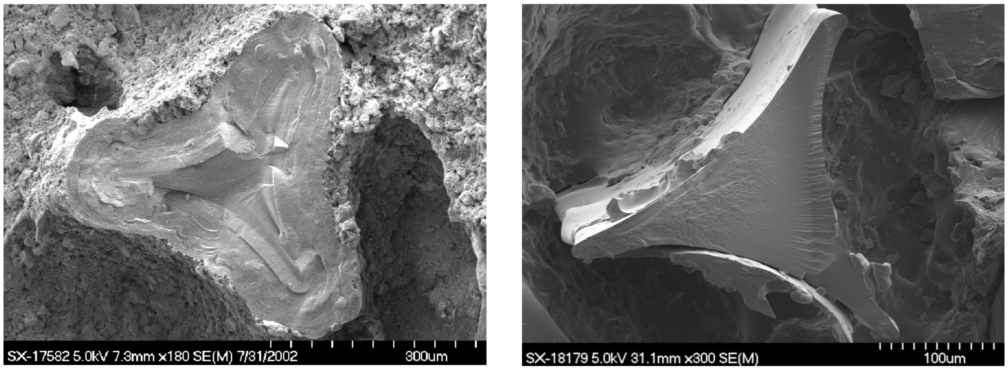


Fig. 3-8. Fracture surfaces of carbon/CVD-SiC and carbon foam ligaments.

Khraisheh Gives "Superplastic" Seminar at the HTML

On February 13, 2002, Dr. Marwan Khraisheh, an assistant professor of mechanical engineering at the University of Kentucky, gave a seminar at the HTML (Fig. 3-9). The seminar was entitled "Mechanics of Multiaxial Deformation of Superplastic Materials: Theory and Experiments."

Fig. 3-9. Marwan Khraisheh presented a seminar about superplastic behavior of materials.

The research interests of Professor Khraisheh include experimental characterization and constitutive modeling of the deformation of advanced materials, superplasticity and superplastic forming, advanced metal forming operations, machining dynamics, and crack initiation under mixed mode loading. During his visit to the HTML, Professor Khraisheh discussed potential research collaborations with MCAUC researchers.

Cracking Processes in Ceramics Reinforced with Carbon Nanotubes—Brown University

University Collaborators: Professor Brian Sheldon and Zhenhai Xia
HTML: Laura Riester

BROWN
UNIVERSITY

Professor Sheldon and postdoctoral fellow Zhenhai Xia are collaborating with MCAUC researcher Laura Riester to investigate the role of carbon nanotubes on the mechanical properties and cracking resistance of porous alumina coatings. These coatings are deposited onto either polycrystalline aluminum or single crystal silicon substrates, and are typically between 1 and 20 microns thick.

These results indicate that the addition of carbon nanotubes results in higher hardness of the coating (Fig. 3-10). It was also found that when indented in a direction parallel to the orientation of the nanotubes coatings, the coating was less tough than unreinforced

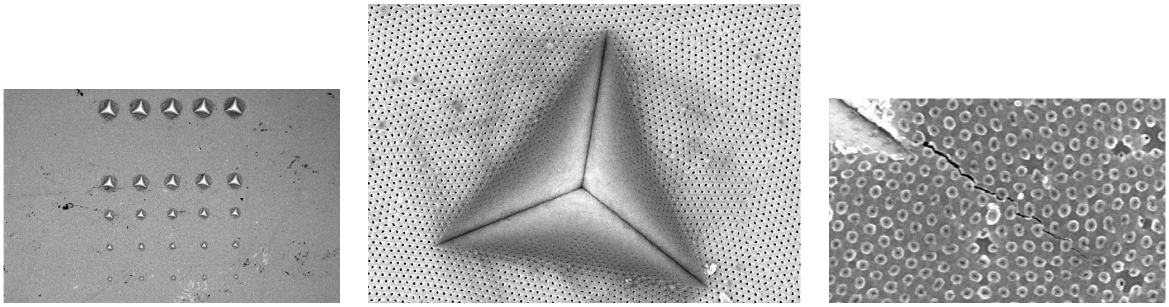

(a) (b) (c)

Fig. 3-10. (a) Arrays of indents in alumina coating. (b) Indent in porous alumina coating without carbon nanotubes. (c) Indent in porous alumina coating with carbon nanotubes. Nanotubes were grown in each pore. Note the cracks emanating from the corners of the indent.

coatings. Significant densification of the porous coatings also occurred as a result of the compressive loads underneath the indenter.

Liu Receives R&D 100 Award

Processes and inventions developed at ORNL garnered another three R&D 100 awards from *R&D Magazine*. ORNL leads the list of national laboratory winners with a total of 112 awards. Among the three 2002 ORNL winning projects was the Spiral Notch Torsion Test, developed by MCAUC researcher Ken Liu in collaboration with Jy-An Wang of ORNL's Nuclear Science and Technology Division. The Spiral Notch Torsion Test is a portable system that tests fracture toughness and strength of materials. In contrast to conventional fracture toughness test methods, it utilizes relatively small test specimens, making it attractive for many applications.

Breder Returns to Crush Ceramic Grains—Saint-Gobain

Industrial Collaborator: Kristin Breder
HTML: Edgar Lara-Curzio

Saint-Gobain Abrasives is a leading manufacturer of abrasive products such as grinding wheels and coated abrasives. Most of the abrasives used by Saint-Gobain Abrasives are based on alumina, alumina/zirconia, and silicon carbide. These abrasives come in a range of sizes and qualities and are incorporated into a range of products with very different bonds. Although fracture strength and fracture behavior are two important properties of abrasive grains, these have not routinely been measured.

Saint-Gobain researcher Kristin Breder worked with MCAUC researcher Edgar Lara-Curzio to determine the fracture strength and fracture behavior of single abrasive grains (Fig. 3-11). Breder, no stranger to the HTML, was part of its research staff between 1993 and 2000.

Using an ORNL-developed testing facility that was specially adapted for the diametral compression of single abrasive grains, Breder carried out, literally, hundreds of tests. Statistical analyses of the fracture loads were obtained and combined with statistical analyses of abrasive grain size distributions to generate strength distribution data (Figs. 3.12 and 3.13). In addition, acoustic emission data and fractography were used to study the

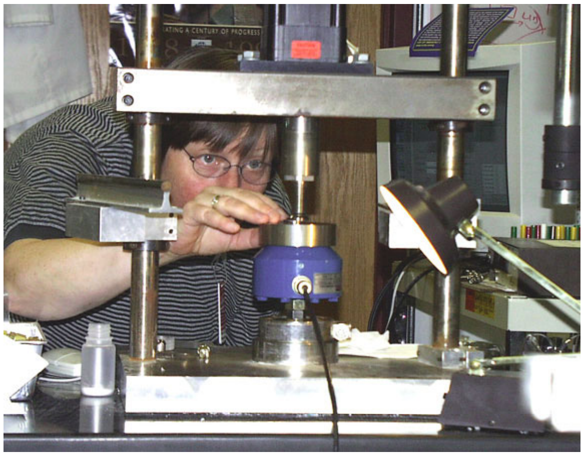


Fig. 3-11. Kristin Breder returns to study properties of abrasive grains.

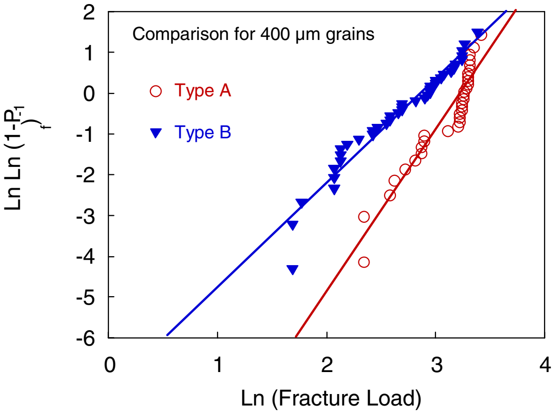


Fig. 3-12. Fracture load distributions for abrasive grains of Type A and Type B.

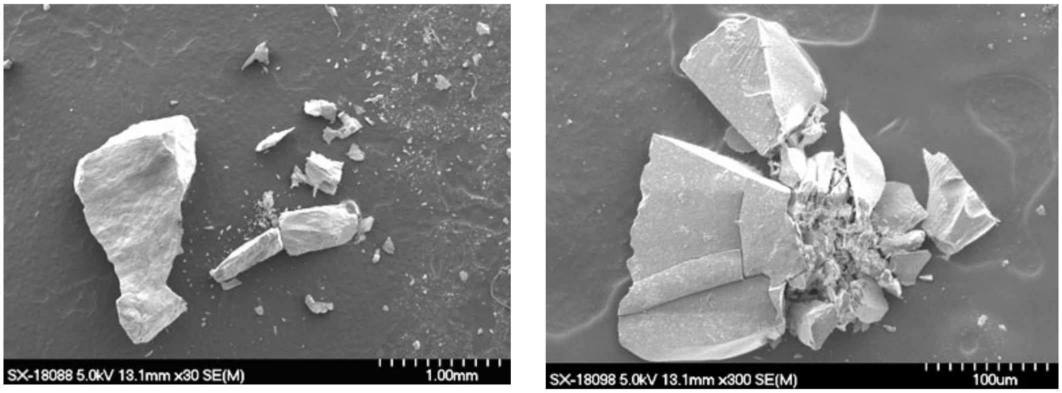


Fig 3-13. Scanning electron micrographs of alumina grains after crush testing.

fracture behavior. The ultimate goal of Breder's user project is to relate the differences in fracture strength and strength distributions of abrasive grains to the differences in grinding behavior, which are already known by Saint-Gobain Abrasives.

Properties of Low-Cost Porous SiC Evaluated—Starfire Systems

Industrial Collaborator: Susan Hayes
HTML: Edgar Lara-Curzio, Laura Riester, and Randy Parten

Wafer carriers and other kiln furniture used in the manufacture of semiconductors must be inert and rigid at temperatures as high as 1400°C. Currently, wafer carriers are made from quartz, SiC-coated graphite, or CVD-SiC. Silicon carbide is the prefered material because of its durability, rigidity, and inertness at high temperatures. Unfortunately, the techniques currently available for fabricating SiC carriers are lengthy and energy-intensive, making wafer carriers very expensive. Therefore, both domestic and foreign manufacturers are actively looking for improved and less expensive methods and materials for making silicon carbide wafer boats.

Starfire Systems Inc. is a small company located in an incubation center at Rensselaer Polytechnic Institute, Troy, New York. They produce “SP-Matrix Polymer,” a polymeric silicon carbide precursor that is an excellent binder for silicon carbide powders.

This precursor, which is based on polycarbosilane, has the unique property of leaving no contaminating elements in the pyrolyzed body while forming a pure silicon carbide matrix. This property is possible because the polycarbosilane polymer contains only C, Si, and H. When the polymer is mixed with SiC powders followed by curing and pyrolysis, a pure, porous SiC part is formed. Currently Starfire Systems Inc. and Chand Associates (Worchester, Massachusetts) are studying the possibility of using Starfire’s polymer for making wafer boats.

To understand the role of porosity and matrix microstructure on the mechanical and physical properties of the final product, Starfire scientist Susan Hayes visited the HTML to work with MCAUC researchers Edgar Lara-Curzio, Laura Riester, and Randy Parten (Fig. 3-14). Flexural tests were carried out to determine the high-temperature strength of materials pyrolyzed at various temperatures. In addition, the thermal shock resistance of these materials was determined at temperatures as high as 1000°C.

Fig. 3-14. Susan Hayes conducting four-point bending tests of porous SiC.

Swindeman Receives Certificate from ASME

MCAUC researcher Bob Swindeman received a certificate from the Board of Governors of the American Society of Mechanical Engineers (ASME). The certificate was “…in testimony of the high regard of his co-workers and the deep appreciation of ASME for his valued services in advancing the engineering profession as a member of the Subcommittee on Materials between 1992 and 2002.” Swindeman is a fellow of ASME.

Constitutive Viscoplastic Behavior of Materials for Automotive Exhaust Manifolds—NW Numerics

Industrial Collaborator: Dr. Ron Foerch
HTML: Ken Liu and Edgar Lara-Curzio

Increasing worldwide competitiveness in the automotive industry has prompted extensive work on the development of models to predict the behavior of automotive engine components and complex assemblies. NW Numerics, a company located in Seattle, Washington, has been in the business of software development and materials characterization since 1996. It has now established a leading position in the modeling of many materials that are relevant to the automotive industry.

As part of a user project, Dr. Ron Foerch of NW Numerics collaborated with MCAUC researchers Ken Liu and Edgar Lara-Curzio to characterize the thermomechanical fatigue behavior and viscoplastic cyclic response of nodular cast iron for automotive exhaust manifolds (Fig. 3-15). Dr. Foerch designed a test matrix with both classical and novel loading and isothermal and temperature variable conditions to solicit simultaneously many synergistic mechanisms of deformation.

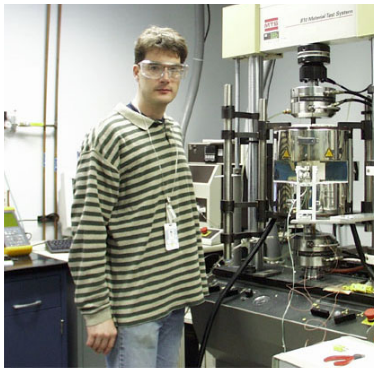


Fig. 3-15. Ron Foerch studies the behavior of materials for automotive exhaust manifolds.

The project also addressed fundamental problems in properly characterizing the behavior of metals at elevated temperatures, namely, the difficulty in separating combined effects of isotropic hardening or softening, kinematic hardening, and viscous overstress at a wide range of stress levels.

The lack of uniformity in the microstructure of the material evaluated will limit the applicability of the experimental results. However, his visit to the HTML was nonetheless productive because he has provided feedback to the material producer (a leading automobile manufacturer) for improving the material. Furthermore, he has used the results obtained to validate the experimental approach. Dr. Foerch expects to return to the HTML to continue the evaluation of improved nodular cast irons.

Data for Numerical Simulation of Spot Welding—University of Missouri-Columbia

University Collaborators: Professor Sanjeev Khanna and Dr. Xin Long
HTML: Ken Liu, Mildain Radovic, James Hemrick, Chris Stevens, and Edgar Lara-Curzio

Professor Sanjeev Khanna and his research group at the University of Missouri-Columbia are developing numerical models to simulate the process of spot welding and predict the distribution of residual stresses in a welded joint. The simulated results are being compared with experimental results obtained using the highly sensitive Moiré interferometry method to study a spot-welded mild steel joint.

Residual stresses play an important role in influencing the fatigue life of spot-welded joints. However, experimental determination of the residual stresses in spot weldments is extremely difficult because of their small size. Numerical simulation can provide important information.

The generation of residual stresses in spot weldments is attributed to the evolution of the microstructure and the consequent changes in the physical and mechanical properties of the materials. Therefore, knowledge of the temperature dependence of the material properties is critical to predict residual stresses accurately.

Professor Khanna and his research associate Dr. Xin Long visited the HTML to work with MCAUC researchers Ken Liu, Miladin Radovic, James Hemrick, Chris Stevens and Edgar Lara-Curzio (Fig. 3-16) to determine the temperature dependence of the elastic constants and stress-strain behavior of A569 steel and two types of aluminum: 6111-T4 and 5754-NG (Fig. 3-17). Tests were carried out at temperatures ranging from 20°C to 1200°C in 200°C increments for the steel and from 20°C to 600°C in 100°C increments for the aluminum alloys.

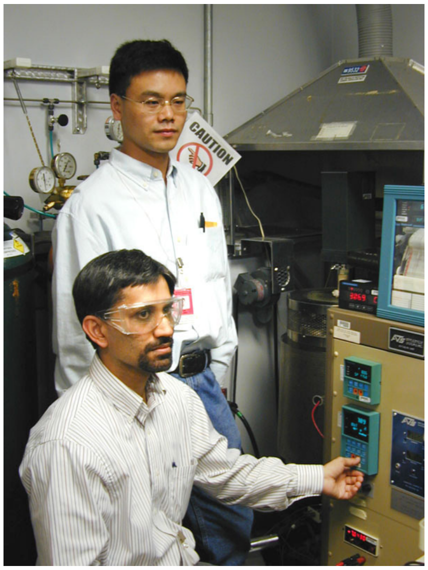


Fig. 3-16. Sanjeev Khanna and Xin Long evaluating the high-temperature elastic properties of materials.

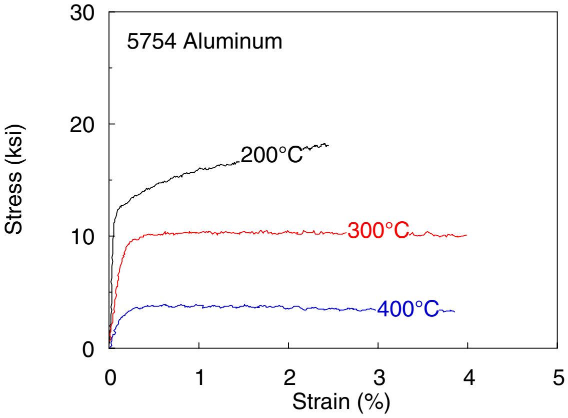


Fig. 3-17. Effect of temperature on the tensile behavior of 5754 aluminum.

Phase Transformations in Metallic Glasses—North Carolina State University

University Collaborators: Professor Albert Shih and Mustafa Bakkal
HTML: Laura Riester and Edgar Lara-Curzio

Unlike pure metals and metallic alloys, metallic glasses have no regular crystalline structure. This lack of long-range order or microstructure often results in high strength and low damping. This result is one reason why zirconium-based metallic glasses are being used to manufacture golf club heads. The high strength-to-weight ratio, excellent wear properties, good forming and shaping qualities, and unusual magnetic properties of metallic glasses also make them attractive for many other applications. Metallic glasses can achieve their glassy, disordered state by using high cooling rates and by alloying metals with dramatically different atomic sizes and chemical characteristics.

North Carolina State University Professor Albert Shih and graduate student Mustafa Bakkal visited the HTML to assess the machinability of metallic glasses. Because of their low thermal conductivity and large thermal expansion, metallic glasses undergo differential cooling during processing. Therefore large thermal gradients can be generated, leading to the buildup of residual stresses. Such stresses influence the mechanical behavior of metallic glasses and may affect their manufacturability. To better understand the changes that take place in these materials during machining, the hardness and elastic modulus of machining chips were evaluated by nanoindentation. The average of the results of more than 100 nanoindents in chips obtained at two different cutting speeds are summarized in Fig. 3-18 and Table 3-1. Additional characterization studies conducted in other HTML user centers suggest that the increase in chip hardness with machining speed results from crystallization and the formation of zirconia.

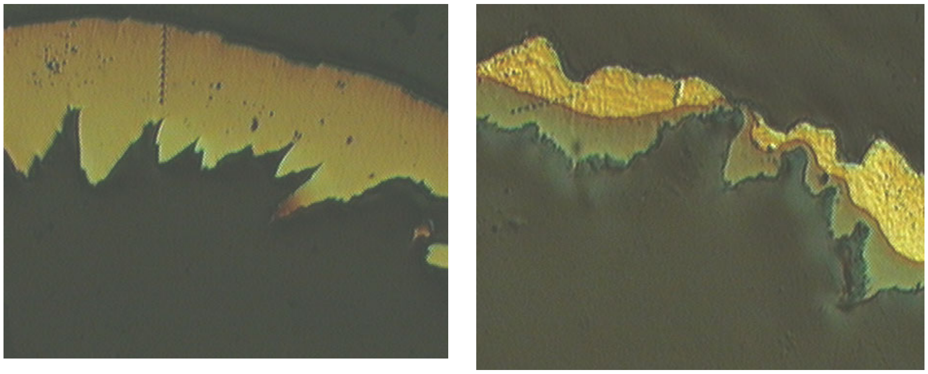


Fig. 3-18. Nanoindents on the polished cross section of metallic glass chips.

Table 3-1. Average nanoindentation results

Cutting speed (m/s)	Average hardness (GPa)	Average elastic modulus (GPa)
0.38	6.05	93.13
1.54	9.40	140.5

Lara-Curzio Receives ASTM's Advanced Ceramics Award

ASTM INTERNATIONAL
Committee C28 on
Advanced Ceramics
Advanced Ceramics Award
presented to
EDGAR LARA-CURZIO
In recognition of your Outstanding Service and Active Participation in the Development of Standards on Advanced Ceramics in the Field Advanced Ceramics
Michael G. Jenkins
Chairman
April 2002

At the 2002 spring meeting of committee C28 of ASTM, MCAUC researcher Edgar Lara-Curzio received the 2002 Advanced Ceramics Award from ASTM Committee C28 on "Advanced Ceramics." The award was in recognition of his "… outstanding service and participation in the development of Standards for Advanced Ceramics."

Honeywell Evaluates Alumina Tubing for Fuel Systems—Honeywell

Industrial Collaborator: Rick Rateick
HTML: Edgar Lara-Curzio

Honeywell

The inside diameters of alumina tubes are being considered for use as wear surfaces in an aerospace fuel system component. Because adequate mechanical property data are not available for probabilistic design using these tubes, Honeywell engineer Rick Rateick visited the HTML to work with MCAUC researcher Edgar Lara-Curzio to generate design data from the evaluation of alumina tubes (Fig. 3-19).

Because the properties of ceramic materials are highly dependent on processing, including machining, the test specimens were prepared in the same way that an actual component would be manufactured. Alumina O-ring test specimens were evaluated under diametral compression to probe

Fig. 3-19. Rick Rateick evaluating alumina tubes.

surface flaws that control the hoop strength while alumina tubular specimens were evaluated in tension to probe both surface and volumetric flaws.

Test specimens were obtained from three different lots and were evaluated at ambient conditions. A total of 30 O-rings and a total of 15 tubes from each of the three lots were evaluated. The test results are currently being analyzed and will be complemented with optical and scanning electron fractography to determine the flaw origin, as required by probabilistic design analyses.

Evaluation of Low-Cost Diamond-Like Coatings—University of North Texas

University Collaborators: Professor Teresa Golden, C. Pingsutiwong, and Q. Wang
HTML: Laura Riester and Michael Lance

Professor Teresa Golden and her colleagues at the University of North Texas developed an electrochemical process to synthesize diamond-like carbon (DLC) from a solution of acetylene in liquid ammonia at temperatures below –40°C. C. Pingsutiwong and Q. Wang worked with MCAUC researchers Laura Riester and Michael Lance to assess the influence of substrate (e.g., crystallographic orientation, surface energy, lattice match) on the deposition of DLC films (Fig. 3-20). DLC is amorphous carbon exhibiting a broad range of properties, from graphite-like to diamond-like. It possesses unique physical and chemical properties such as high hardness, high thermal conductivity, high optical transparency in the infrared (IR) region, high electrical insulation, low coefficients of friction, and high chemical inertness and corrosion resistance.

Fig. 3-20. C. Pingsutiwong and Q. Wang evaluated low-cost diamond-like carbon coatings.

Films deposited on different substrates such as Ni, Mo, Cu, Co, Fe, stainless steel, and brass were investigated (Fig. 3-21). Raman spectroscopy was used to determine the different carbon compositions of the films. Nanoindentation was used to measure the hardness and to establish the reliability of the coatings.

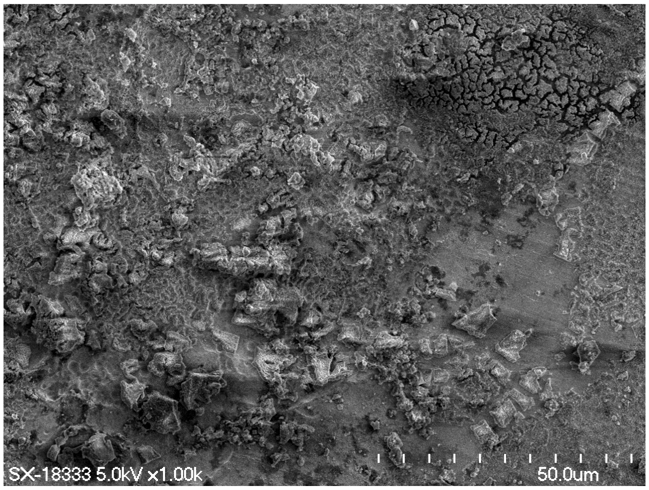


Fig. 3-21. Diamond-like coatings synthesized in an electrochemical process.

DLC films deposited on nickel from acetylene in liquid ammonia under galvanostatic mode produced higher Raman intensities than those from films deposited by potentiostatic mode. Adding sodium acetylide in liquid ammonia improved the Raman intensity. When using only sodium acetylide as an electrolyte, the films become more inhomogeneously deposited, and poor Raman intensity was obtained.

The Raman results also revealed that the DLCs were successfully deposited onto Mo, Co, brass, and stainless steel. The films on copper substrate were mostly graphitic. To better interpret the nanoindentation results, the hardness and stiffness of the various substrates were determined before indentation of the DLC coatings.

4. DIFFRACTION USER CENTER (DUC)

User Center Members
Andrew Payzant, Leader
Geneva Worley, Secretary

Jianming Bai
Claudia Rawn
Scott Speakman
Tom Watkins

The DUC uses variable-temperature X-ray, synchrotron, and neutron diffraction methods to characterize crystalline phases and 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.

The DUC supports academic, industrial, and U.S. Department of Energy (DOE) laboratory users' diffraction needs. The diffraction facilities are extensively used by qualified staff members in the Metals and Ceramics Division who are conducting a wide variety of ceramic and alloy research and development efforts sponsored by DOE and other 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 2002 include the following.

- A total of 25 new user proposals were prepared in FY 2002.
- More than 20 research papers were co-authored by DUC staff in 2002.
- S. A. Speakman joined DUC as an ORNL postdoctoral research associate.
- C. J. Rawn gave an invited presentation at the 2002 TMS Annual Meeting.
- C. J. Rawn was appointed to a joint ORNL-UT faculty position.
- C. J. Rawn was elected to Vice-Chair of the Oak Ridge Chapter of ASM International.
- E. A. Payzant gave an invited presentation at the 2002 TMS Annual Meeting.
- E. A. Payzant gave an invited presentation at a Gordon Research Conference.
- C. R. Hubbard was elected to the ICDD Board of Directors.
- C.R. Hubbard received the McMurdie Award at the 2002 Denver X-Ray Conference.

DUC staff members were principal investigators on two Laboratory Director's 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 lay a foundation for cooperative future use of the upgraded instruments at the High Flux Isotope Reactor and the revolutionary Spallation Neutron Source.

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 (low temperature, controlled environment)
- Synchrotron X-ray Beamline X-14A (low and high temperature)

Selected Highlights

New Capillary Furnace Installed at X14A Beamline

HTML: C. Rawn and J. Bai

A new high-temperature furnace was built at Ames Laboratory for the DUC synchrotron beamline at the National Synchrotron Light Source (NSLS). It was installed in April 2002 (Fig. 4-1).

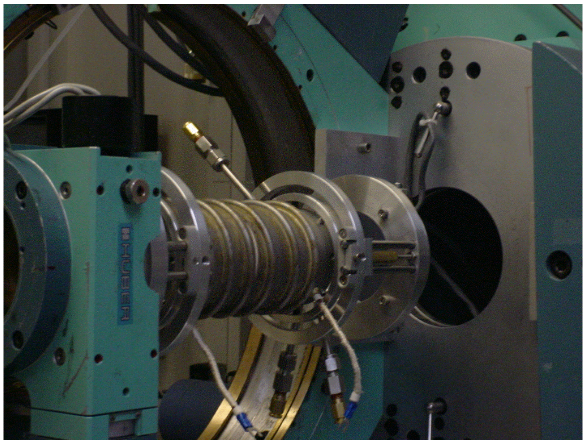


Fig. 4-1. New capillary furnace mounted on the X14A goniometer.

This furnace will greatly extend the capabilities of the user center to conduct in situ high-temperature X-ray diffraction studies on small quantities of material with high accuracy and precision.

Solid Oxide Fuel Cell Materials: $LaCrO_3$ Perovskites for SOFC Interconnects—Tennessee Technological University

University Collaborators: J. Zhu and Z. Li
HTML: A. Payzant

The application of chromia-forming ferritic steels for solid oxide fuel cell (SOFC) interconnects must address a number of problems. These problems include Cr migration from the interconnect onto the cathode side and insufficient protectiveness of the thermally grown chromia oxides, which lead to rapid degradation of the cell performance. A thin layer of doped lanthanum chromite on ferritic steel may act as a protective coating to mitigate the Cr volatility problems and facilitate the use of metallic interconnect in SOFCs.

In this project, the $LaCrO_3$ thin film was successfully synthesized on a ferritic stainless steel substrate by two novel approaches: reactive formation and sol-gel processing. Sr dopants were also successfully introduced into $LaCrO_3$ in an attempt to reduce the electrical resistance of the coating. X-ray diffraction and scanning electron microscopy were used to analyze the coating microstructure, surface morphology, and phases. The electrical resistance of the sol-gel processed samples before and after thermal and cyclic oxidation at 800°C in both air and reducing environment was determined from 600 to 900°C. It was found that the $(La,Sr)CrO_3$ coating was effective in reducing the degradation in electrical conduction of the interconnect materials.

Solid Oxide Fuel Cell Materials: Stability of Nanocrystalline Ceria-Zirconia Composites—New Mexico Institute of Technology

Industrial Collaborator: J. Dong
HTML: S. Speakman and A. Payzant

Ceramic composites based on Sm-doped CeO_2 (SDC) and Y-doped ZrO_2 (YSZ) are potential SOFC electrolytes.

The in situ high-temperature X-ray diffraction instruments in the DUC were used to study particle growth and solid phase reactions between SDC and YSZ factors.

These two factors, which affect stability, were studied as a function of temperature up to 1400°C (see Fig. 4-2).

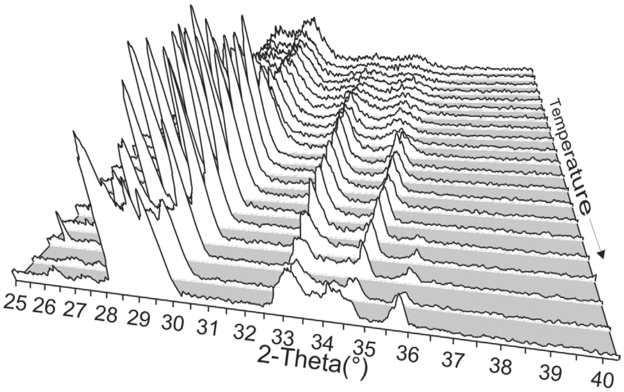


Fig. 4-2. XRD patterns of the SDC/YSZ nanoscale particle mixture collected at temperatures from 21 to 1400°C. The sharpening of peaks is indicative of particle growth, and the formation of new peaks is indicative of a solid phase reaction.

Superconducting Materials: In Situ Observation of Eutectoid Reaction in Nb_3Al—The Ohio State University

University Collaborators: F. Buta, R. Snyder, and E. Collings
HTML: S. Speakman

Florin Buta, a graduate student from Ohio State University, used the in situ high-temperature X-ray diffractometers to study the eutectoidal reaction between A15 Nb_3Al and sigma-type Nb_2Al. This reaction is significant in the processing of superconducting wire based on Nb_3Al. The temperatures required (in excess of 1900°C) necessitated the use of refractory metal heating elements in an oxygen-free atmosphere. Satisfactory temperatures were achieved in a 10^{-6} torr vacuum. However, rapid vaporization of Al initially prevented the collection of equilibrium data. Use of a gettered argon gas atmosphere, with $PO_2 < 10$ ppm, slowed the Al vaporization and allowed the eutectoidal reaction to be observed.

Hydrogen Storage Materials: In Situ Study of Zr_2Fe Hydride Formation—The University of Nevada at Reno

University Collaborators: D. Chandra and M. Coleman
HTML: A. Payzant

An in situ study was completed of Zr_2Fe (SAES St198), which is used for the gettering of hydrogen and tritium. HTML's unique controlled atmosphere high-temperature X-ray diffractometer enabled comparative studies under nitrogen, hydrogen, and inert atmospheres. Presentations were made at the TMS2002 and the MRS Spring Meeting.

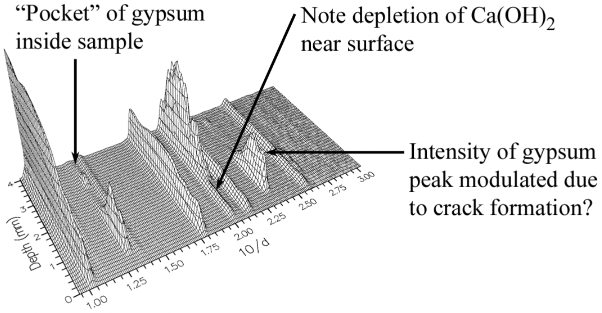
Highway Construction Materials: Studies on Portland Cement—Georgia Institute of Technology

University Collaborators: A. Wilkinson and A. Jupe
HTML: J. Bai

The exposure of structures made from Portland cement-based concrete to sulfate-containing ground waters or wastes leads to damage. Ultimately, over extended periods of time, it leads to failure. As part of a chemical and microstructural investigation of this degradation, Energy Dispersive X-ray Diffraction (EDXRD) in a spatially resolved mode at the Advanced Photon Source was used to construct depth profiles of the phase composition in cement paste samples that had been exposed to sulfate under varying conditions.

Spatially resolved EDXRD is very powerful. Good spatial resolution can be achieved without in any way damaging the original specimen. The high-energy X rays used for EDXRD will penetrate many millimeters inside the cement specimen. However, EDXRD provides powder diffraction patterns with poor reciprocal space resolution, and this resolution can make phase identification difficult.

Monchromatic synchrotron powder diffraction data were collected using HTML beam line X14A at the NSLS to complement the EDXRD data. Very small specimens of sulfate-exposed cement paste were obtained from different depths below the surface of a cement paste rod by a destructive procedure. These specimens were used to obtain a clear picture of the phases present as a function of depth (Fig. 4-3).


“Pocket” of gypsum inside sample

Note depletion of $Ca(OH)_2$ near surface

Intensity of gypsum peak modulated due to crack formation?

Fig. 4-3. EDXRD data as a function of depth below the specimen surface for a type V Portland cement paste cylinder exposed to 33,800 ppm Na_2SO_4 solution for 52 weeks. The development and destruction of various phases as a function of depth below the specimen surface can be readily observed.

Solar Cell Materials: Effects of Temperature, Pressure, and Precursor Morphology on the Growth Kinetics of $CuInSe_2$ Films—University of Florida

University Collaborators: S. Kim, K. Kim, and T. Anderson
HTML: A. Payzant

Suku Kim and Kyoung Kim, graduate students at the University of Florida, used the in situ high-temperature X-ray diffractometer to study the growth of CuInSe2 from precursor films grown by molecular beam epitaxy (MBE). The rapid X-ray data acquisition rates made possible through the use of a position-sensitive detector enables observation of the evolution from the precursor phases to the final product. This study showed that the reaction paths and reaction rates are strongly influenced by the morphology of the precursor film, with intermediate phases found in some precursor morphologies but not in others (Fig. 4-4). A manuscript is already in review, describing the activation energies and rate kinetics for each precursor system in order to assist in optimization of the thin film processing.

Gas Storage Materials: Low-Temperature Studies on Gas Clathrate Hydrates—The Colorado School of Mines

Industrial Collaborators: D. Sloan and M. Eaton
HTML: C. Rawn

Natural gas hydrates are widespread in the seafloor sediments of continental margins and in arctic permafrost. The U.S. Geological Survey (USGS) has estimated that the energy content of the methane contained in natural methane hydrate deposits exceeds that of all known fossil-fuel reserves.

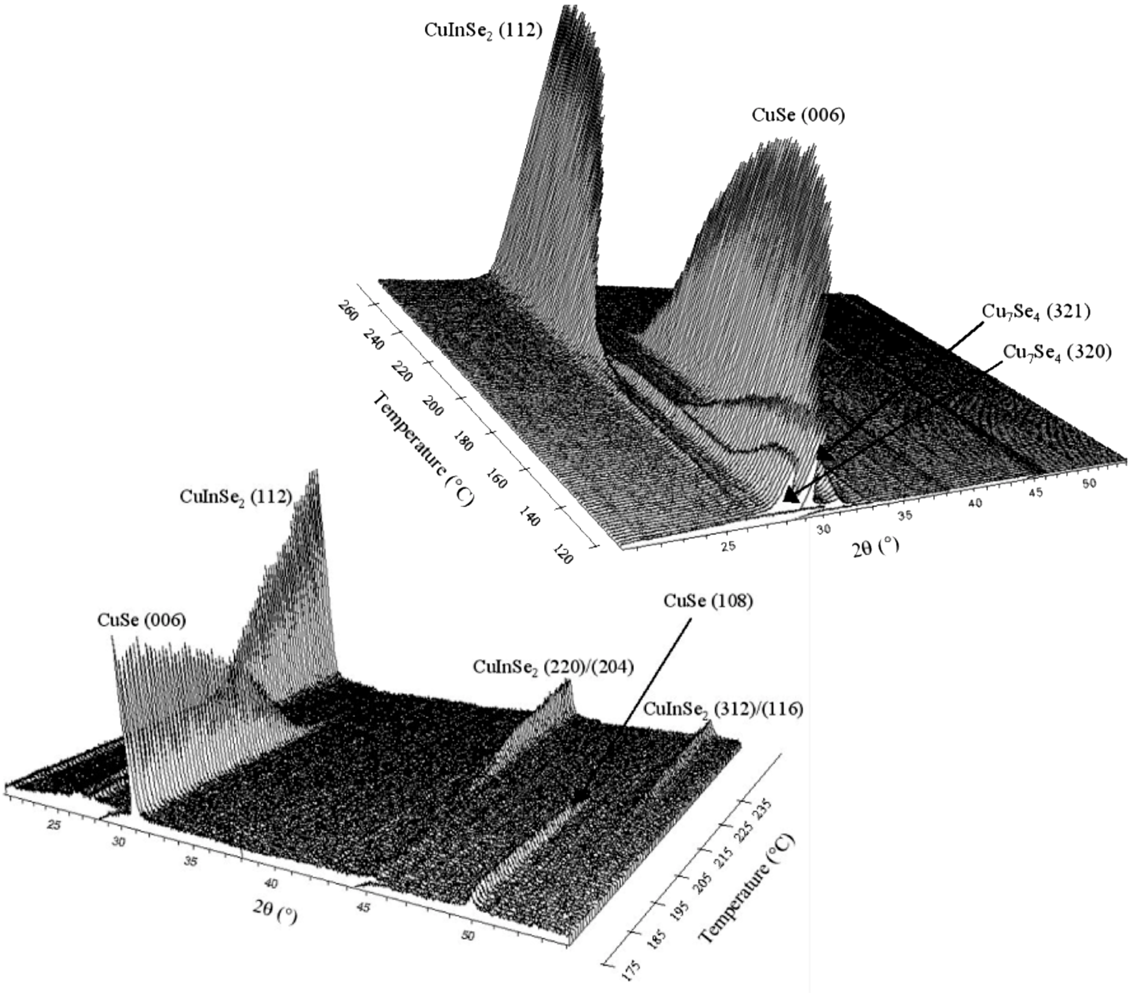


Fig. 4-4. The precursor in the upper right trace began with a layer of amorphous InSe and a top layer of elemental Cu and Se. Upon heating Cu_7Se_4 was formed, then CuSe, and finally $CuInSe_2$. In the lower left trace it began with five bilayers of alternating crystalline CuSe and amorphous InSe. In this case, the $CuInSe_2$ phase formed directly from the precursors.

Natural gas hydrates also play a major role in the global carbon cycle and are of environmental interest as a source of greenhouse gases that could contribute significantly to global warming. Carbon dioxide hydrate is also of interest in this connection, having been proposed as a form in which industry-generated carbon dioxide could be sequestered in the deep ocean. Dr. Claudia Rawn has ongoing research in the area of gas clathrate hydrates, originally funded through an ORNL internal LDRD proposal and currently funded by the DOE Office of Fossil Energy.

Michael Eaton, a graduate student in Dr. Dendy Sloan's hydrate research group at the Colorado School of Mines, visited the HTML to gain proficiency in X-ray data collection and analysis on gas clathrate hydrate samples (Fig. 4-5). A propane clathrate hydrate sample was provided by the USGS in Menlo Park, California.

Fig. 4-5. Michael Eaton, visiting from the Colorado School of Mines.

X-ray powder diffraction data were collected as a function of temperature using the low-temperature X-ray diffractometer at the HTML. The sample was loaded onto a cold sample stage and was kept below roughly 100 K in flowing He during loading, to avoid decomposition and frosting. Data were collected up to the decomposition temperature.

Diffraction User Center Research Featured at TMS Annual Meeting

The TMS Annual Meeting was held in Seattle in February 2002. Andrew Payzant organized and chaired a session on fuel cells and membranes, and Claudia Rawn organized and chaired a session on gas clathrate hydrates for a symposium titled “Fundamentals of Advanced Materials for Energy Conversion.”

Many of the invited and contributed papers at the TMS meeting featured the HTML User Program. Papers included those by Dr. Junhang Dong (New Mexico Tech), Dr. Michael Hu (ORNL), Dr. Dhanesh Chandra (University of Nevada-Reno), Dr. Joachim Schneibel (ORNL), and Dr. John Zhu (Tennessee Tech).

Industrial Applications of XRD Session Organized

Dr. Camden Hubbard co-organized a full-day session on industrial applications of XRD at the 2002 Denver X-Ray Conference. The conference was held in Steamboat Springs, Colorado, July 29–August 2. Invited presentations included real-time XRD for process control for galvanizing steel for auto bodies (a presenter from Germany), the use of XRD in the heavy duty diesel industry (by Roger England, Cummins Engine Co.), and texture measurement for characterizing aluminum for container manufacture (by Hasso Weiland, Alcoa Technical Center). Contributed presentations covered a wide range of industrial applications of XRD and neutron diffraction. These presentations included prototype high-temperature superconductor wire manufacturing (by Robert Snyder, Ohio State University), pulp and paper industry boiler tube stress analysis, and gasifier refractory degradation studies (by Camden Hubbard, ORNL).

Hubbard Receives the McMurdie Award

The McMurdie Award was presented by the International Centre for Diffraction Data (ICDD) to HTML’s Camden Hubbard at the Denver X-Ray Conference’s annual meeting in August (Fig. 4-6).

Dr. Hubbard was cited “… for his seminal contributions to the computer-aided evaluation of X-ray powder diffraction patterns and editing of the Powder Diffraction File, and decades of contributions to enhance the accuracy of powder methods of X-ray crystallography.”

Fig. 4-6. Robert Synder (r), past chair of ICDD, presenting the McMurdie Award to Camden Hubbard.

5. RESIDUAL STRESS USER CENTER (RSUC)

User Center Members

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

Jianming Bai
O. Burl Cavin
Hahn Choo
Gerard M. Ludtka
E. Andrew Payzant
Roberta Peascoe
Stephen Spooner
Thomas R. Watkins

HTML user projects and DOE programs are increasingly concerned with the life prediction and failure analysis of engineering structures and how to improve the life of structures 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 RSUC was established to meet this need and to provide a facility for research into controlling residual stresses through modifications in the forming, surface treating, and finishing processes; changes in the design; or stress-relief procedures.

RSUC provides three principal measurement capabilities (see RSUC Instruments) 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
- PANalytical X'Pert Pro X-ray diffractometer with high-temperature stages
- X-ray large-specimen stress analyzer
- Neutron Residual Stress Mapping Facility (NRSF), including remote access

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

HFIR is one of the two highest flux steady state sources of neutrons in the world. The NRSF and the neutron powder diffraction instrument leverage on the operating, maintenance, and upgrade programs of DOE–Energy Research. Similarly, the RSUC activities at the X14A beamline at the National Synchrotron Light Source (NSLS) benefit greatly from the DOE-ER sponsorship of NSLS. RSUC also gratefully acknowledges the consignment of a high-temperature X-ray diffraction system by the PANalytical (formerly Phillips Analytical) and Anton Paar.

New and Enhanced Instruments

X-Ray Diffraction Large-Specimen Stress Mapping

Automation of the Technology for Energy Corporation's TEC X-ray diffraction large-specimen stress analyzer made considerable progress during the year. User projects in stress mapping by X-ray diffraction will begin in early FY 2003. The automation and instrumentation involved construction of a gantry system (Fig. 5-1) and addition of two sets of translation and rotation positioning systems.

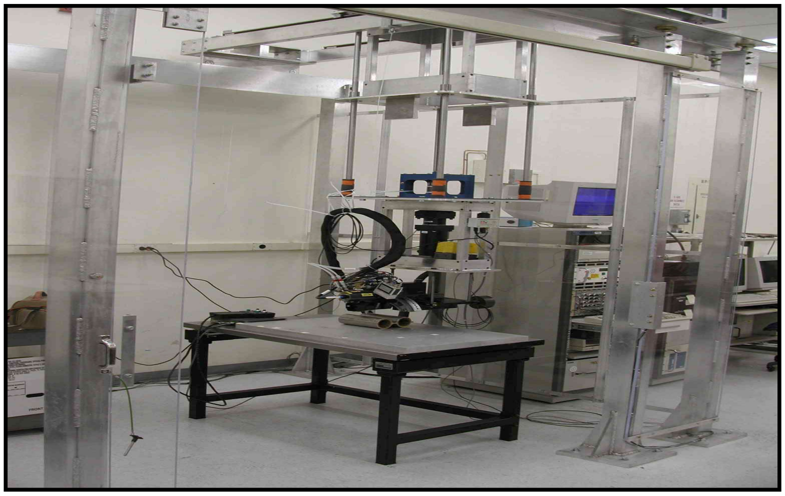


Fig. 5.1. Automated X-ray diffraction large-specimen stress analyzer and gantry system.

One positioning system is for research-sized specimens; the other is for large parts the size of an engine block. The computer automation of the system is based on the same software that will run the NRSF system and monochromator. The use of common software for these two stress-mapping facilities will provide users a common graphical user interface and hence a faster learning curve.

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) and other industry- sponsored projects.

Neutron Residual Stress Mapping Facility

The HFIR began a major upgrade in July 2000 that involved the replacements of the beryllium reflector and four beam tubes. The HFIR upgrades have been completed, and operation of the reactor began in winter 2002. Throughout the remainder of 2002, the DOE-ER–funded upgrades to facilities and instruments have continued.

With DOE-ER sponsorship, the HB-2 beam tunnel (~50 tons of shielding) and 20-ton crane were designed, fabricated, and installed. The beam tunnel and guide installation will provide thermal neutron beams to four experiments including NRSF, the wide area powder diffractometer, and the high-resolution powder diffractometer. The upgrades at beam tube HB-2 are particularly relevant to NRSF. The dedicated instrument position for NRSF will greatly enhance the beam time and eliminate time lost to periodic setup and alignment. Periodic adjustments were required previously because of the shared use of the 25-year-old former instrument.

Efforts planned for FY 2003 include installation of instrument shutters, construction of the instrument monochromator plugs, and installation of instrument cranes. Following completion of these, the new NRSF system will be installed and tested in summer 2003. Upgrades to NRSF include a new monochromator system, multiple detectors, two high-resolution goniometers with expanded capacity for large and small specimens, and enhanced automation and remote operation capabilities. When completed, NRSF will provide an approximately tenfold improvement in measurement capability compared to the shared instrument that was available to NRSF before the upgrades began.

Engineering Materials Science Beam Line at SNS Receives Major Funding

The Canadian Foundation for Innovation has approved a proposal submitted by Canadian scientists for construction funding for the Engineering Materials Science Beam Line, VULCAN, at ORNL's Spallation Neutron Source (SNS). This proposal represents approximately 75% of the funding needed for this beam line and instrument. SNS, currently under construction, is scheduled for completion in 2006. It is currently DOE's largest science project and is intended to provide the world's best pulsed neutron source for neutron scattering.

VULCAN, one of ten approved instrument proposals, aims to make major advances in scientific understanding in materials. The research will include processing, forming, joining, heat treating, and performance by providing a compound instrument (strain mapping and simultaneous small angle scattering). It will be complimentary to HTML's upgraded neutron residual stress facility at HFIR.

Several RSUC staff members are members of the instrument advisory team that developed the scientific justification for this instrument. Additional funding is still needed to fully construct VULCAN and to achieve the extensive materials research goals defined by U.S. academic, industrial, and government laboratory scientists and engineers. Many of the recommended studies for VULCAN will have exceptional value for EE/RE programs.

Related Activities Beneficial to RSUC

Neutron Laue Strain Mapping LDRD Funding Awarded

The through thickness mapping of residual strains for determination of residual stresses has proved valuable to both academic and industrial users via the HTML User Program's NRSF at HFIR. However, when large grained materials (e.g., from a casting) are encountered, the method can fail because of too few grains contributing to the diffraction pattern. The Laue method is an alternative that permits measurement of orientation and strain in individual grains. Together with ORNL Solid State Division colleagues, a neutron Laue strain mapping method was proposed, based on a highly successful X-ray microbeam method. The project was funded by ORNL's LDRD program for FY 2002 and 2003. Researchers will conduct demonstration experiments and establish the scientific case for a subsequent proposal for development at ORNL (either at HFIR or at SNS).

Zhili Feng of Engineering Mechanics Corporation of Columbus Presented Keynote Lecture at the Oak Ridge ASM International Educational Symposium

Dr. Zhili Feng of Engineering Mechanics Corporation of Columbus presented the keynote lecture at the Oak Ridge Chapter of ASM International's 2002 Educational Symposium on the application of neutron scattering in materials science and engineering. His lecture highlighted the value of experimentally measured residual stresses. His presentation included numerous examples of neutron residual stress mapping studies conducted with RSUC facilities involving weldments.

At Engineering Mechanics Corporation, this data has been extensively used to develop finite-element method models for welding processes and for post-weld heat treatment. Welding is a critical enabling manufacturing technology and developing improved processes and the ability to predict properties and performance will have a major impact. While models have been successfully developed for a number of cases, Feng also pointed out cases, such as friction stir welding and post-weld heat treatment, in which considerable progress in weld models is still needed. Feng concluded with his vision of goals for next-generation stress-mapping facilities. Many of these goals will be achieved with the upgrade of the NRSF at HFIR; others will await the construction of VULCAN at the SNS.

NSF Proposal for International Materials Institute Program Submitted

A proposal was submitted to the International Materials Institute Program of the National Science Foundation for funding to establish an international consortium to be called the Advanced Neutron Scattering netWork for Education and Research (ANSWER). The consortium would support collaborative research and education in neutron scattering-based materials research. Proposed activities include research programs focusing on in situ, real-time characterization of mechanical behavior of advanced engineering materials as well as exchange programs, international workshops and symposia, and neutron schools. The proposal was developed by Professor Peter Liaw and Hahn Choo at the University of Tennessee and Drs. Camden Hubbard, HTML, and Xun-Li Wang, SNS, both of ORNL. A total of $3.6M was requested for the 5-year program. Currently the proposal is under review. More than 50 participants from universities, industries, and national laboratories supported the proposal. Supporters represented eight different countries, with most of the support coming from participants in North America. If funded, HTML's RSUC would be one of the key research groups and facilities used by U.S.-based academic and industrial researchers and would also serve as a focus for workshops and symposia.

User from University of Tennessee Successfully Defends Thesis

Puja Kadolkar successfully defended her M.S. thesis on June 27, 2002. Her thesis focused on the characterization of laser-bonded TiC and Si particles to an aluminum substrate. The characterization was completed partially in the RSUC. This relatively new coating application method has garnered some interest for wear applications, particularly for lightweight aluminum. The residual stress, microstructure, and strength were determined and related to the laser-bonding process parameters, particularly the table speed at optimum laser power. It was found that the quality of the coating degraded with increasing table speed. Research staff member Dr. Thomas Watkins assisted with the research at HTML and served on Kadolkar's thesis committee. Following graduation from UT this summer, Puja Kadolkar will start an internship at ORNL.

Selected Highlights

RSUC continues to address critical industrial and academic problems, typically using a combination of RSUC and other HTML facilities. Interest 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. These activities demonstrate a growing use of a combination of the RSUC instruments to use a comprehensive measurement or mapping of stress to enhance life prediction, understand failure, guide improvement in manufacturing processes, and address fundamental science.

Characterized White Layers Formed in Hard Turning of 52100 Steel (Two Projects)—Georgia Tech

University Collaborators: A. Ramesh and S. Melkote
HTML: T. R. Watkins

A hard turning is a finish machining process that has significant economic and environmental advantages over a more typical finishing method such as grinding. However, some undesirable surface artifacts known as “white layers” can form during hard turning. The scope of this project is to determine the nature of the formation of the white layer in 52100 steel. The project will help to gain a physical understanding of the process and improve in hard turning modeling efforts. This information would allow manufacturers to choose machining parameters that would not result in white layer formation.

Researchers from Georgia Tech hypothesized that white layers form during hard turning predominantly because of mechanical loading conditions such as low cutting speeds. The white layers consist of refined grains of the base material because of plastic deformation. Layers formed under predominantly thermal loading, such as high cutting speeds, consist of retransformed or “untempered” grains. At RSUC, when machining with high cutting speeds, the workpiece temperatures are high enough to cause austenitization of surface and near-surface layers of the work material followed by a rapid quench to form an untempered martensitic microstructure.

The X-ray diffraction results showed that retained austenite content was higher in the samples that experienced relatively higher surface temperatures either because of high cutting speeds or electrodischarge machining (EDM). Because cutting forces and hence mechanical deformation were approximately the same, the differences can be explained by the austenite phase transformation.

A stress-assisted retained austenite transformation reduced the retained austenite by 14% for the slowly machined sample relative to the unmachined. The reduction in retained austenite was less in the faster machined sample (only 8%) because the surface temperatures were higher. The higher temperatures likely caused more re-austenization because of an incomplete quench, which resulted in more retained austenite. The EDM sample has the most retained austenite as the surface was melted (>1535°C), and the subsequent quench did not get near the finish temperature [Fig. 5-2 (a)]. The measured surface axial and hoop residual stresses are shown in Fig. 5-2 (b). The residual stress gradients as a function of depth are shown in Fig. 5-3.

The initial surface residual stresses in the unmachined sample were isotropic and compressive. Slow-speed machining anisotropically increased the compressive residual

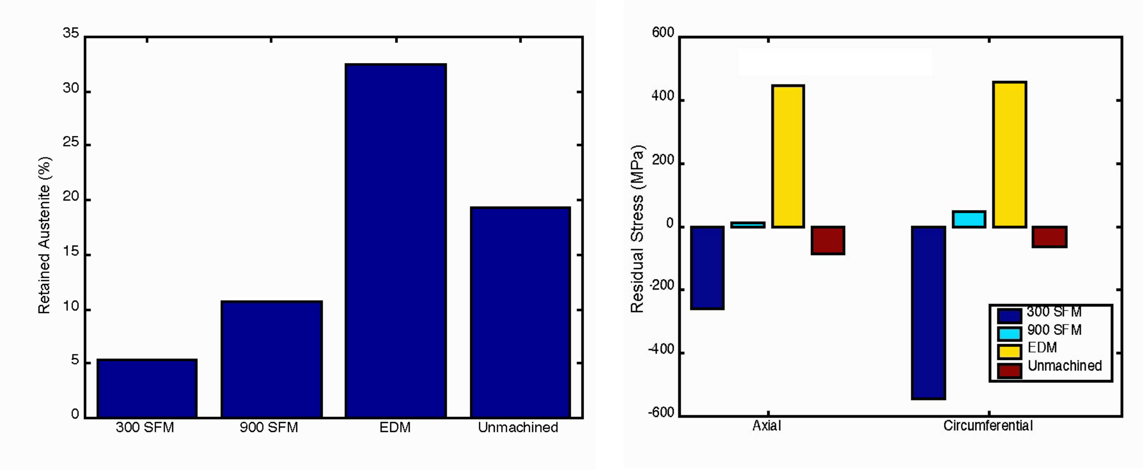


Fig. 5-2. (a) The percentage of retained austenite per machining condition. (b) The residual stresses per machining condition for axial and circumferential directions.

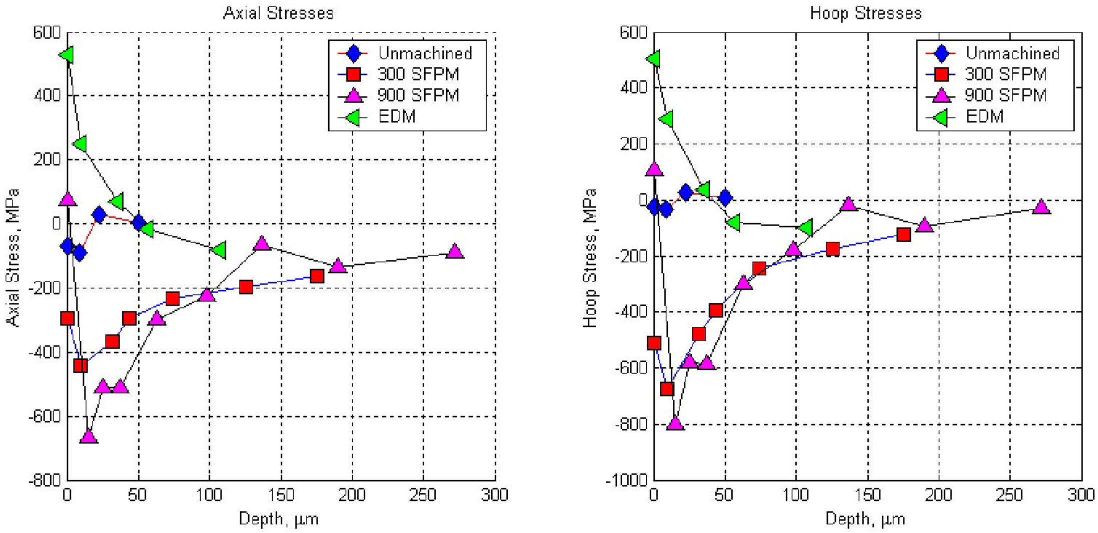


Fig. 5-3. Axial (l) and hoop (r) through thickness residual stresses for hard-turned 52100 steel.

stress. This residual stress was the superposition of the stresses because of the plastic deformation of mechanical contact plus the compressive elastic strain/plastic deformation of the retained austenite-to-martensite transformation. High-speed machining anisotropically increased the tensile residual stress. This residual stress is likely the superposition of the tensile plastic deformation of mechanical contact plus the compressive plastic deformation because of the constrained thermal expansion of the frictionally heated surfaces plus the compressive elastic strain/plastic deformation of the retained austenite-to-martensite transformation. Under these conditions, the surface heating effect was of greater magnitude, resulting in tensile residual stresses.

In the case of the EDM sample, the surface residual stresses were isotropic and tensile. This result is indicative of the large thermal heating on the workpiece surface. This residual stress is likely the superposition of the relatively large compressive plastic deformation because of the constrained thermal expansion of the heated surfaces from EDM plus the smaller compressive elastic strain/plastic deformation of the retained austenite transformation.

Studies of Recrystallization and Deformation Texture at RSUC—ORNL SNS

Laboratory Collaborators: Y. Wang and Xun-Li Wang
HTML T. R. Watkins

The study on texture evolution during recrystallization is one of the most important topics in materials science and engineering. Textures play an important role in many industrial processes, such as deep-drawing steel used for the automobile industry and silicon steel used for electron transformation and thin films in the electronic industry. A complete understanding on the mechanisms to control the recrystallization texture is essential for improving the performance of those materials.

A researcher from ORNL's SNS visted the RSUC to obtain quantitative information on textures of those growing grains that can be obtained by combining the technique of synchrotron radiation with laboratory X-ray diffraction. Commercial Ti-stabilized IF steel was cold-rolled from 3.7 to 1.2 mm thick. Some samples were subsequently annealed at 550°C for 2.5 h, which promoted partial recrystallization or growth of new grains from the highly deformed matrix. Texture measurements on partially crystallized samples using high-energy synchrotron radiation gave mainly the "remained" deformation texture components (i.e., eliminating those recrystallized, or growing, grains with large grain size) [Fig. 5-4 (b)]. Laboratory X-ray texture measurements on partially crystallized samples gave the average texture (remained deformation component plus the growth grains) [Fig. 5-4 (a)]. By subtracting the retained deformation regions [Fig. 5-4 (b) from Fig. 5-4 (a)], the texture for the recrystallized regions is obtained
[Fig. 5-4 (c)].

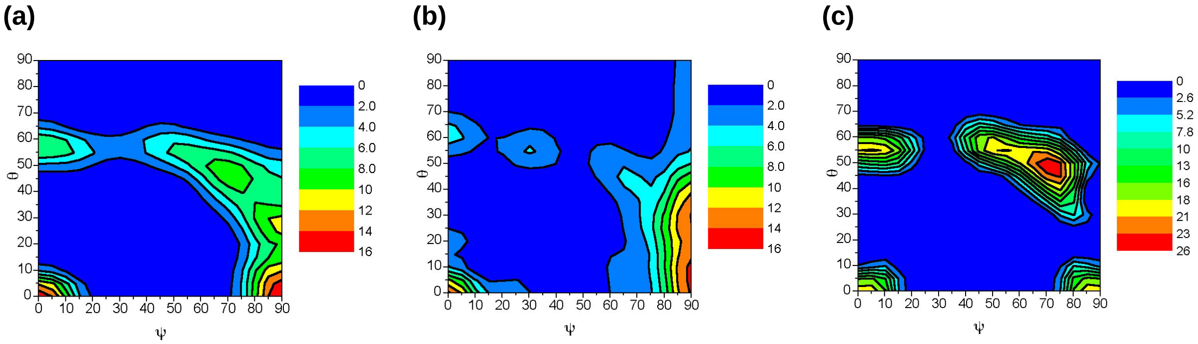


Fig. 5-4. ODF (φ = 45° section) of (a) a partially recrystallized sample (measured by laboratory X-ray diffraction), (b) an ODF-retained deformation matrix after partial recrystallization, and (c) new texture components because of recrystallization, calculated by subtracting (b) from (a).

The texture in the recrystallized regions consists of mainly the {111}<112> component, which developed by consuming the primary -fiber in the cold-rolled sample. In addition, there is another component near {100}<110>. However, since the laboratory X-ray diffraction only measures texture near the sample surface (~µm), it is unclear whether the component near {100}<110> is representative of the recrystallized region in the bulk. Measurements by neutron diffraction, which provide a measure of the bulk, should help to clarify this feature.

RSUC Assists with Metallic Glass Characterization—North Carolina State University

University Collaborators: M. Bakkal and A. Shih
HTML: T. R. Watkins

A researcher from North Carolina State University (NCSU) visited RSUC to study the effect of machining of zirconium-based metallic glass material (Zr-17.9Cu-14.6Ni-5Ti-10Al-at%) with emphasis on the analysis of machining chips, the machined surface, and tool wear. Because of the low thermal conductivity of the Zr-based metallic glass material, the heat is retained in the chip, making it red-hot for an extended period of time. For this reason, the turning of metallic glass is different from machining other structural metallic materials.

Turning of standard 1/4-in. diameter metallic glass bars was performed at NCSU with a 0.02-in. depth of cut; 0.002-in./rev feed rate; and 75-, 150-, and 300-ft/min surface speeds. X-ray diffraction was used to characterize the chips and the three machined surfaces resulting from the three different surface speeds. The characterization would identify possible devitrification or increased crystallization of the material.

According to preliminary results, there is no evidence of any additional crystallization on the cutting surface and chips for the 75-ft/min cutting speed. For the 150-ft/min cutting speed, evidence of crystallization on the chips was observed but not on the turned surface. For the 300-ft/min cutting speed, crystalline phases were observed on both the turned surface and chips. Thus, increasing cutting speed causes greater heat accumulation on the surface and chips and, as a consequence of that heat accumulation, a greater tendency for crystallization.

HTML Assists in Study of Residual Stresses in Tungsten Carbide Coatings—University of Florida at Gainesville

University Collaborators: D. P. Butt and D. Parker
HTML: O. B. Cavin and T. R. Watkins

A researcher from the University of Florida visted the RSUC to characterize the residual stress state of high-velocity oxygen fuel (HVOF) thermal-sprayed coatings of tungsten carbide cobalt and the effects on the fracture behavior of high-strength steel alloys (Fig. 5-5). The coatings are being evaluated as a suitable alternative to conventional hexavalent chrome plating for aircraft landing gear and hydraulic component sliding wear applications.

Correlation of the effects of residual stress both in the coating and substrate on the fatigue crack initiation conditions for various stress levels should allow for prediction of the conditions in which fatigue cracks will occur. The results indicate a large increase (~50×) in compressive stress with polishing after spraying. Following fatigue testing, the compressive residual stresses decrease with increasing fatigue load.

Analysis continues at the University of Florida to relate these results to fatigue crack origination. Ultimately, optimization of coating spray parameters is sought to maximize the performance of the coatings for specific applications. It is interesting to note that additional laboratory testing, as well as flight testing, has shown that the HVOF coatings currently

Fig. 5-5. Don Parker, University of Florida-Gainesville, mounting a fatigue specimen on the PTS rotating anode system.

outperform the chrome-plated components in wear applications by more that 2:1 in service life (based on seal life and component wear).

Studies of Residual Stresses in Thermal Barrier Coatings—PANalytical

Industrial Collaborator: J. Ellis
HTML: T. R. Watkins and O. B. Cavin

A researcher from PANalytical (formerly Philips Analytical) visted the RSUC to determine the relationship of failure mechanisms to the residual stress state in thermal barrier coating (TBC) systems at room and elevated temperatures (Fig. 5-6). Knowledge of residual stress as a function of temperature is critical for understanding TBC failure, which is typically because of a thermally grown oxide scale.

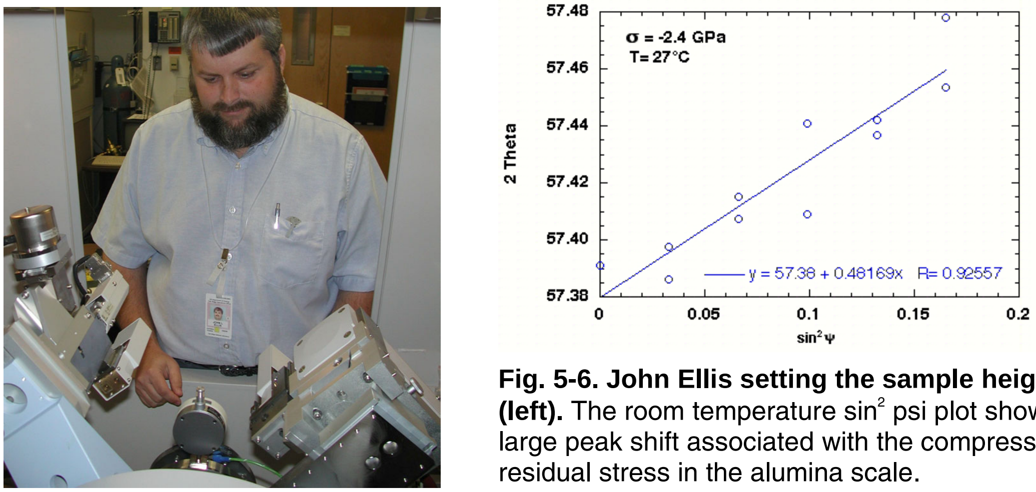


Fig. 5-6. John Ellis setting the sample height (left). The room temperature $\sin^2$ psi plot shows a large peak shift associated with the compressive residual stress in the alumina scale.

The new PANalytical X'Pert Pro MPD system with the Anton Paar HTK 16 hot stage stationed at the HTML provided a unique opportunity to study this problem. The theta-theta geometry was coupled with the parallel beam optics required for high-temperature stress work. This combination allows conventional and widely accepted $\sin^2$psi methods to be applied rather than being constrained to the fixed incident method (due to gravity) at the synchrotron.

A bare, commercially available "PtAl" bond coat on a Rene N5 substrate was oxidized at 1200°C for about 48 hours on the high-temperature stage. As was seen previously in a synchrotron study, effectively no residual stress was discerned at elevated temperature in the alumina scale. A large –2.4 GPa residual stress was observed at room temperature.

6. THERMOGRAPHY AND THERMOPHYSICAL PROPERTIES USER CENTER (TTPUC)

User Center Members

Ralph B. Dinwiddie, Leader
Geneva N. Worley, Secretary

Wallace D. Porter
Hsin Wang

The TTPUC is dedicated to measuring thermophysical properties as a function of temperature and correlating these properties with the processing, microstructure, and performance of materials. Specifically, TTPUC staff work with users to determine thermophysical properties such as thermal diffusivity, thermal conductivity, specific heat, and thermal expansion. They also work with users 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.

The TTPUC continues to develop capabilities in the field of infrared (IR) imaging and sensing using focal plane array IR cameras and fast, single-point IR 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.

TTPUC Instruments

- 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 analyzer

Selected Highlights

Motorola Energy System Group Studied Thermal Transients in IC Power Transistor Arrays at TTPUC—Motorola

Industrial Collaborators: H. Maleki, John Oglesbee, and Chris Thongsouk
HTML: Hsin Wang and R. B. Dinwiddie

The Motorola Energy System Group worked with TTPUC staff to capture heat pulses in integrated circuits (IC) power transistor arrays. An infrared microscope was used to study the surface temperature profiles of power transistor arrays in IC during operation. Each transistor array was set to conduct current for 20–50 microseconds. The integration time of the IR camera was adjusted to stay between 2 and 10 microseconds.

A thorough study of the camera’s timing characteristics allowed its precise synchronization to transient thermal events in the transistor arrays. Progressively adding incremental delay times to the synchronization pulses allowed the complete characterization of the thermal transients as a function of time and location.

The IR microscope timing characteristics were determined by imaging an incandescent lamp filament during pulsed operation. The transistor arrays heated up to about 165°C. Figure 6-1 shows an example of heat pulses in a lamp filament.

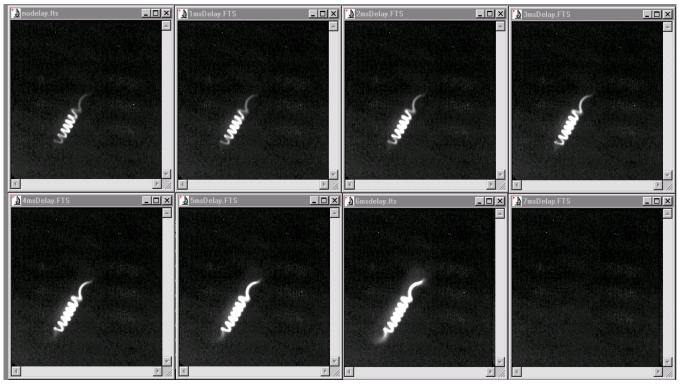


Fig. 6-1. Microscopic IR images of lamp filament with 0 ms (top left) to 7 ms (bottom right) delay time. The IR camera integration delay was determined to be 7 ms.

University of Missouri-Columbia Conducted Research at TTPUC on Alloys Used in Automobile Spot Welding Process—University of Missouri-Columbia

University Collaborators: Sanjeev K. Khanna and X. Long
HTML: Hsin Wang

The University of Missouri-Columbia is conducting a modeling effort on spot welding of aluminum alloys (Al5754 and Al6111) and steel (A569) used in the automobile industry. Thermophysical data on alloys used in the spot welding process are not available in the literature.

Thermal diffusivity measurements as a function of temperature were performed on the laser flash unit up to 500°C for aluminum alloys and 1000°C for A569 steel. Specific heat of the material was measured by the differential scanning calorimetry (DSC). The thermal conductivity, heat capacity, and diffusivity measurements are being used in the modeling of the spot welding process.

II-VI Inc. Studied Thermal Transport Properties of Materials Used in Crystal Growth Process—II-VI Inc.

Industrial Collaborator: Xiaoming Li
HTML: Hsin Wang

A researcher from II-VI Inc. worked with TTPUC staff on materials involved in the crystal growth process (Fig. 6-2). During the first visit, the thermal transport properties of iridium metal (crucible), alumina and zirconia insulations, quartz crystal, and ND-YAG crystals were tested using the laser flash unit and the hot disk thermal constants analyzer.

Thermal diffusivity of these materials was measured up to 1400°C in the laser flash unit. These data are used in the modeling of the crystal growth process at II-VI Inc. A better understanding of the temperature distribution and heat transfer during crystal growth is key to improving product quality and reliability.

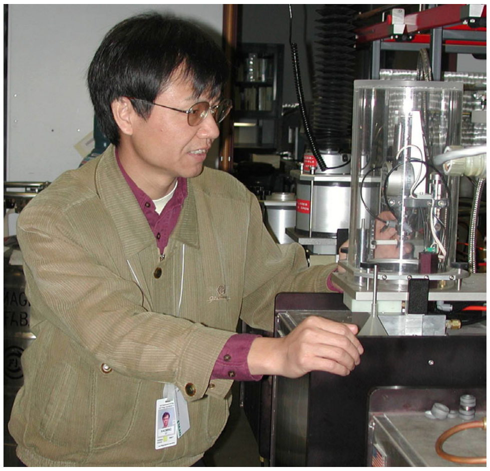


Fig. 6-2. Xiaoming Li, a researcher from II-VI Inc.

Auburn University Studied Thermal Conductivity of Carbon Aerogels at TTPUC—Auburn University

University Collaborators: Ralph Zee and Kashyap Yellai
HTML: Hsin Wang and Wally Porter

Researchers from Auburn University studied the thermal conductivity of carbon aerogels. This material will be used as high-temperature thermal insulations for space applications. The thermal diffusivity of carbon aerogels was obtained using the laser flash method. The thermal diffusivity values were compared with carbon-bonded carbon fiber (CBCF) material developed at ORNL. Specific heat of the carbon aerogels was measured by the DSC system up to 1400°C. The effect of a vacuum and radiative heating were also studied during the laser flash measurements.

Multimodal Gross Rail Defect Detector—Intellutran

Industrial Collaborators: Daniel W. McDonald (PI), Gary A. Armstrong, and Paul B. Burn
HTML: Ralph B. Dinwiddie

Intellutran, which produces measurement systems for the transportation industry company, is studying the use of ultrasensitive IR imaging. The imaging will be used as part of a multimodal detection system for finding large transverse fissures in railroad rail heads at inspection speeds of 12 m/s. Transverse defects cause 29% of train derailments in the United States and cost the industry millions of dollars a year.

For this project, two rails with “man-made” defects were prepared and studied. The first rail had a series of blind holes drilled upward from the bottom of the railhead and terminating at various depths below the top surface (2, 5, 10, 15, 20, and 25 mm). The second rail was prepared by the MITUC with three notches, each starting at the bottom of the railhead and continuing upward to various depths below the top surface (10, 20, and 25 mm). By studying the derivative of the IR signal under thermal gradient conditions, the notches that are 10 and

20 mm below the surface are revealed (Fig. 6-3). Uniform heating of the top surface reveals the blind holes that are 2 and 5 mm below the surface (Fig. 6-4).

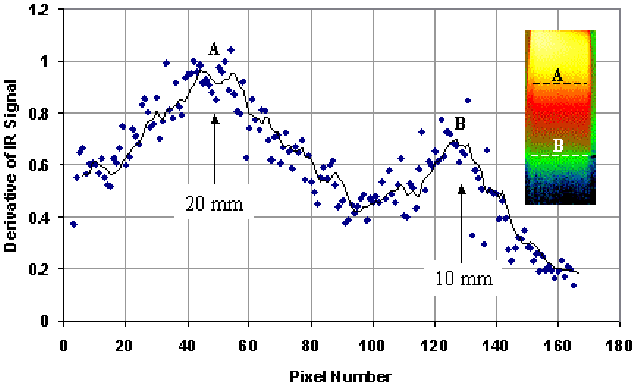


Fig. 6-3. Studying the derivative of the IR signal under thermal gradient conditions reveals the notches that are 10 and 20 mm below the surface.

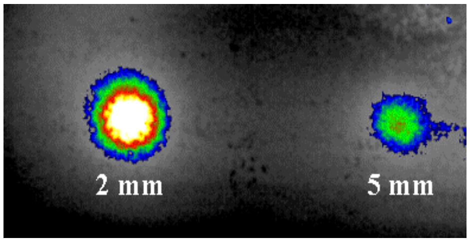


Fig. 6-4. Uniform heating of the top surface reveals the blind holes that are 2 and 5 mm below the surface.

University of Tennessee Studied Plastic Zones During Fatigue Crack Growth Using High Resolution Infrared Imaging—University of Tennessee, Knoxville

University Collaborators: Bing Yang and P. K. Liaw
HTML: H. Wang

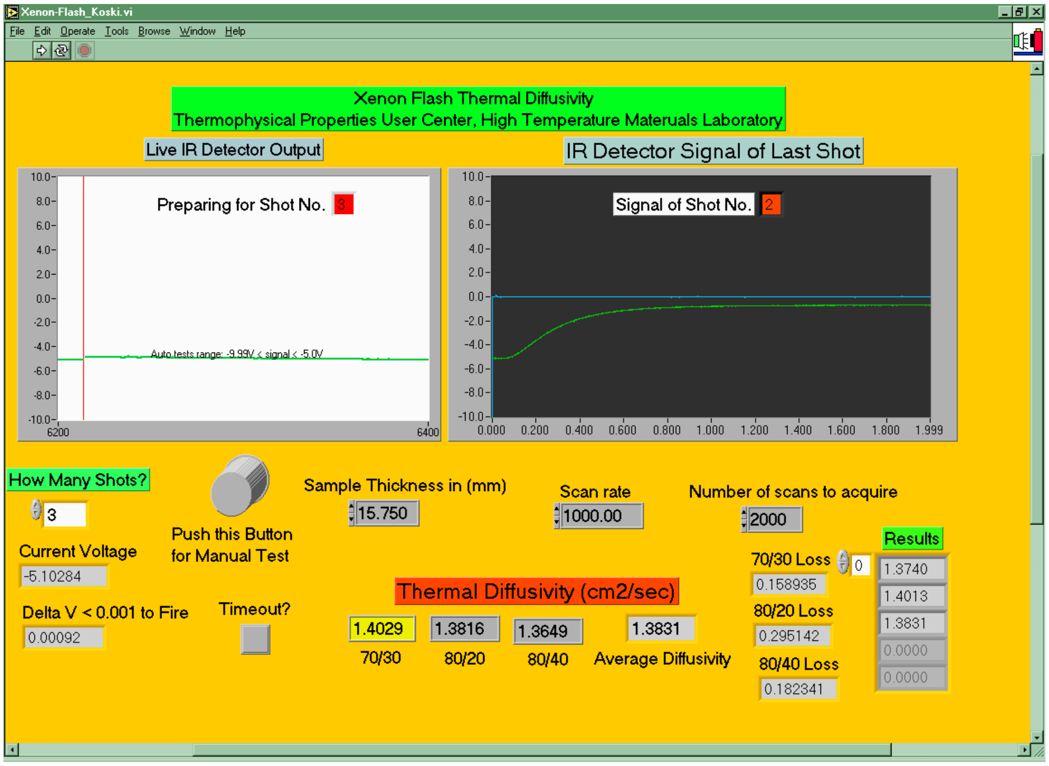
The new IR imaging system at the University of Tennessee Materials Science and Engineering Department was used to study plastic zones during fatigue crack growth. The IR microscope and close-up lenses from TTPUC were used to achieve better spatial resolution.

Ultimet superalloy was tested using notched samples. Each sample was precracked with a length of 0.45”. Fatigue test conditions were f = 10 Hz and R = 0.1. To visualize the plastic zone near the crack tip, high-speed imaging (150 Hz at 640 × 320 pixels and 500 Hz at 128 × 128 pixels) were used to record temperature change within each fatigue cycle. With the spatial resolution of 15 microns per pixel, the plastic zone was observed. Further image processing is being conducted at UTK and TTPUC to eliminate the effect of heat conduction.

TTPUC Upgraded its Xenon Flash Diffusivity System

HTML: H. Wang and R. B. Dinwiddie

The xenon flash room temperature thermal diffusivity system was developed by R. B. Dinwiddie. Very reliable, the system measures thermal diffusivities of a variety of specimens. A recent upgrade was supported by the Metal and Ceramics Division at ORNL. The computer system was upgraded from Windows 3.1 to Windows 2000, and the controlling software was upgraded from QuickBasic to LabView 6.0i. The system can now operate at much higher speeds and process thermal diffusivity data instantly. The system can be controlled from a laptop computer. It is also portable and can be used for field tests (see Fig. 6-5).



Xenon Flash Thermal Diffusivity
Thermophysical Properties User Center, High Temperature Materuals Laboratory

Preparing for Shot No. 3

Signal of Shot No. 2

How Many Shots? 3

Current Voltage -5.10284

Delta V < 0.001 to Fire 0.00092

Sample Thickness in (mm) 15.750

Scan rate 1000.00

Number of scans to acquire 2000

Thermal Diffusivity (cm2/sec)

70/30	80/20	80/40	Average Diffusivity
1.4029	1.3816	1.3649	1.3831

70/30 Loss 0.158935

80/20 Loss 0.295142

80/40 Loss 0.182341

Results 0
1.3740
1.4013
1.3831
0.0000
0.0000

Fig. 6-5. User interface of the new LabView-based xenon flash diffusivity system.

STANDARD NONPROPRIETARY USER AGREEMENTS

The next several pages comprise a listing of universities and companies that have entered into standard nonproprietary user agreements with the HTML. The list includes 338 companies, and 20 government and other facilities, and 166 universities from across the United States.

U.S. INDUSTRY—338

Alabama
Citation Corporation (Birmingham)
Foseco Morval (Bessemer)
Monarch Tile, Inc. (Florence)
United Defense LP (Anniston)

Arizona
Advanced Ceramics Research (Tucson)
Materials Focus, Inc. (Tuscon)
Motorola (Tempe)
RASTRA of the Americas (Litchfield Park)

California
Allied Signal Inc. Ceramic Components (Torrance)
Alzeta Corporation (Santa Clara)
Amercom, Inc. (Chatsworth)
CERCOM, Inc. (Vista)
Ensci, Inc. (Pismo Beach)
FMC Corporation (Santa Clara)
IBM Almaden Research Center (San José)
Lockheed Martin Skunk Works (Palmdale)
M. J. Schiff & Associates, Inc. (Upland)
Membrane Technology Research (Menlo Park)
Northrop Corporation (Pico Rivers)
Nuclear and Aerospace Materials Corporation (Poway)
Rohr, Inc. (Chula Vista)
Solar Turbines, Inc. (San Diego)
SRI International (Menlo Park)
Sullivan Mining Corporation (San Diego)
Sunstrand Corporation (San Diego)
Ultramet (Pacoima)
X-Ray Instrumentation Associates (Mountain View)

Colorado
Coors Ceramics Company (Golden)
Golden Technologies Company (Golden)
Material Physics Research (Highlands Ranch)
Johns Manville (Littleton)
NA Technologies (Golden)
Protecto Wrap Company (Denver)
Quantum Peripherals (Louisville)
Schuller International, Inc. (Littleton)
TDA Research, Inc. (Wheat Ridge)

Connecticut
ABB C-E Services, Inc. (Windsor)
Plasma Coatings, Inc. (Waterbury)
Praxair, Inc. (Danbury)
Steven Winter Associates, Inc. (Norwalk)
Torrington Company (Torrington)
United Technologies Pratt and Whitney (East Hartford)

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

Florida
American Boats Crushing Company Inc. (Boca Raton)
Constellation Technology Corporation (Largo)
Florida Solar Energy Center (Cape Canaveral)
Metamic LLC (Lakeland)
Pratt and Whitney (West Palm Beach)
Siemens Westinghouse Power Corporation (Orlando)

Georgia
Advanced Engineered Materials, LLC (Woodstock)
Amercord, Inc. (Lumber City)
Ceradyne, Inc. (Scottdale)
Component Composite Coatings International LLC (Roswell)
Ionic Atlanta, Inc. (Atlanta)
Microcoating Technologies (Chamblee)
Motorola Energy Systems Group (Lawrenceville
RCF Seals (Vidalia)
Rolls Royce, Inc. (Atlanta)
Thermal Ceramics (Augusta)

Illinois

A. Finkl & Sons (Chicago)
Adtech Nepth Inc. (Oak Park)
Alloy Engineering and Casting Company (Champagne)
Applied Thin Films, Inc. (Evanston)
Belcan Corporation (Peoria)
Caterpillar, Inc., Technology Center (Peoria)
Deere & Company (Moline)
Insulating Concrete Form Association (Glenview)
Metal Construction Association (Glenview)
Scot Forge (Spring Grove)
Wagner Castings Company (Decatur)

Indiana

Allison Engine Company (Indianapolis)
Cummins Engine Company, Inc. (Columbia)
Dana Corporation (Richmond)
Firestone Building Products Co. (Carmel)
GM Corporation/Delco Remy Division (Anderson)
Haynes International (Kokomo)

Kentucky

ARCO Aluminum, Inc. (Louisville)
CC Metals and Alloys, Inc. (Calvert)
Florida Tile Industries (Lawrenceburg)
Logan Aluminum (Russellville)
Machining Research Inc. (Florence)
Secat, Inc. (Lexington)
Stoody Company (Bowling Green)

Louisiana

Lockheed Martin Michould Space Systems (New Orleans)

Maine

Busek Co. Inc. (Natick)
Surmet Corporation (Burlington)
Uniform Metal Technologies, LLC (Watertown)

Maryland

Hilcoit Corporation (Upper Marlboro)
Krispin Technologies, Inc. (Rockville)
RCMA (Rockville)
Refractory Composites, Inc. (Glen Burnie)
W. R. Grace and Co-Conn (Columbia)

Massachusetts

American Superconductor Corporation (Watertown)
Brigham and Women's Hospital (Boston)
Ceramics Process Systems Corporation (Milford)
Chand Kare Technical Ceramics (Worcester)
Chand Metallurgical (Worcester)
Dynamet Technology (Burlington)
Foster Miller, Inc. (Waltham)
GTE Laboratories, Inc. (Waltham)
Hydrogen Microplasmatron Technologies, LLC (Cambridge)
JPS Elastomerics Corporation (Holyoke)
Norton/TRW Ceramics (Northboro)
Osram Sylvania/University of Massachusetts at Lowell (Lowell)
Philips Analytical Inc. (Natick)
Refractory Testing Associates (Chestnut Hill)
Rohm and Haas (Woburn)
Saint-Gobain Norton (Northboro)
Sarnafil, Inc. (Canton)

Michigan

Bosch Braking Systems (Farmington Hills)
Chrysler Corporation (Highland Park)
Delphi Corporation (Brighton)
Detroit Diesel Corporation (Detroit)
Dow Chemical Company (Midland)
Dow Corning Corporation (Midland)
Duro-Last Roofing, Inc. (Saginaw)
Eaton Corporation (Southfield)
Energy Conversion Devices, Inc. (Troy)
Ford Motor Company (Ann Arbor)
GM Powertrain Group (Pontiac)
GM R&D Center (Warren)
Hoskins Manufacturing Company (Hamburg)
Howmet (Whitewall)
Inductoheat (Madison Heights)
Modern Alloying Technologies, LLC (West Bloomfield)
Parker Abex NWL (Kalamazoo)
Tenneco Automotive (Glass Lake)
Thixomat (Ann Arbor)

Valenite, Inc.(Troy)
Visteon Corporation (Allen Park)
W. H. Porter, Inc. (Holland)

Minnesota

3M (St. Paul)
FMC Naval Systems Division (Minneapolis)
H. B. Fuller Company (Vadnais Heights)
Phygen, Inc. (Minneapolis)
Seagate Technology (Minneapolis)

Mississippi

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

Missouri

Deloro Stellite Group Ltd. (St. Louis)
McDonnell Douglas Aerospace (St. Louis)
MTI-Emory Ford (St. Louis)
SB&TD Business Systems (Lancaster)

Montana

Anaconda Foundry Fabrication Co., Inc. (Anaconda)
Columbia Falls Aluminum Company (Columbia Falls)

New Hampshire

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

New Jersey

ARDE, Inc. (Norwood)
Ceramic Magnetics, Inc. (Fairfield)
Certech, Inc. (Wood Ridge)
Engelhard Corporation (Edison)
INRAD, Inc. (Northvale)
International Paper (Princeton)
Lucent Technologies (Murray Hill)
Material Technologies, Inc. (Shrewsbury)
Nanopowder Enterprises, Inc. (Piscataway)
NEC Research Institute, Inc. (Princeton)
Phone-Poulenc, Inc. (Cranbury)
Stryker Howmedica Osteonics (Rutherford)

New Mexico

Eberline Instruments (Santa Fe)
Environmental Technology & Education Center (Albuquerque)
TPL Inc. (Albuquerque)

New York

Advanced Refractory Technical, Inc. (Buffalo)
AKZO Nobel Chemicals, Inc. (Dobbs Ferry)
Applied Nano Metrics Inc. (Stormville)
Carborundum Company (Niagara Falls)
CDH Energy Corporation (Cazenovia)
CMP Industries, Inc. (Albany)
Corning Inc. (Corning)
Eastman Kodak Company (Rochester)
General Electric Company (Schenectady)
Gray-Syracuse Inc. (Chittenanog)
Monofrax, Inc. (Falconer)
MRC, Division of Praxair Surface Technologies (Orangeburg)
ReMaxCo Technologies, Inc. (Kenmore)
Starfire Systems, Inc. (Watervliet)
Sulzer Metco, Inc. (Westbury)
T. J. Watson Research Center (Yorktown Heights)
U. K. Software Services, Inc. (Grand Island)
X-Ray Optical Systems, Inc. (Albany)

North Carolina

Advanced Energy (Raleigh)
ALLVAC (Monroe)
Cree Research, Inc. (Durham)
Magnequench Technical Center (Research Triangle Park)
MicroMet Technology, Inc. (Matthews)
Porvair Fuel Cell Technology (Hendersonville)
Selee Corporation (Hendersonville)
Syngenta Agribusiness Biotechnology Research (Research Triangle Park)
Teledyne Allvac (Monroe)

Ohio

Advanced Ceramics Corporation (Lakewood)
Bicron-NE (Solon)
Cincinnati Machine Company (Cincinnati)

Commonwealth Aluminum (Uhrichsville)
Deformation Control Technology, Inc. (Cleveland)
Doehler-Jarvis Technologies, Inc. (Toledo)
Engineering Mechanics Corporation of Columbus (Columbus)
Equistar Technology Center (Cincinnati)
General Electric Aircraft Engines (Cincinnati)
Goodyear Tire and Rubber Company (Akron)
IAP Research, Inc. (Dayton)
Illinois Tool Works (Troy)
Lincoln Electric (Cleveland)
LTV Steel Company (Independence)
MascoTech Forming Technologies (Minerva)
Mead Research (Chillicothe)
Metal Building Manufacturers Association (Cleveland)
Milacron, Inc. (Cincinnati)
Owens Corning Technical Center (Granville)
Park-Ohio Transportation Group (Cleveland)
PCC Airfoils, Inc. (Beachwood)
Procter & Gamble Company (Cincinnati)
Republic Technologies International (Lorain)
Rhenium Alloys, Inc. (Elyria)
Sandusky International (Sandusky)
The Timken Company (Canton)
Tosoh SMD, Inc. (Grove City)
Universal Energy Systems, Inc. (Dayton)
Western Environmental Corporation (Franklin)

Pennsylvania

Advanced Technology Materials, Inc. (University Park)
AHT, Inc. (Chicora)
Alcoa Technical Center (Alcoa Center)
Alcoa, Inc. (Alcoa Center)
Alvord-Polk, Inc. (Millersburg)
Ametek Specialty Metals (Eighty Four)
Bethlehem Steel Corporation (Bethlehem)
Calgon Corporation (Pittsburgh)
Carlisle Syntec, Inc. (Carlisle)
Carpenter Technology Corporation (Reading)
Certainteed Corporation (Valley Forge)
Concurrent Technologies, Inc. (Johnstown)
Congoleum (Newton)
Duraloy Technologies, Inc. (Scottdale)
IBACOS (Pittsburgh)
II-VI Inc. (Saxonburg)
J & L Specialty Steel (Pittsburgh)
Kennametal, Inc. (Latrobe)
Leroy A. Landers (Philadelphia)
Materials Resources International (North Wales)
National Forge Company (Irvine)
PPG Industries, Inc. (Pittsburgh)
Thermacore, Inc. (Lancaster)
West Homestead Engineering and Machine Company (Homestead)
Westinghouse Electric Corporation (Pittsburgh)

Rhode Island

Quadrax Corporation (Portsmouth)

South Carolina

Spirax Sarco, Inc. (Blythewood)

Tennessee

Activated Metals & Chemicals (Sevierville)
American Magnetics, Inc. (Oak Ridge)
American Matrix, Inc. (Knoxville)
Atlantic Research Corporation (Knoxville)
Browne Technology, Inc. (Nashville)
BTR Sealing Systems (Rockford)
Camel Manufacturing Company (Pioneer)
Carroll Kenneth Johnson (Oak Ridge)
Cavin Consulting Services (Knoxville)
Ceramaspeed, Inc. (Maryville)
Church and Dwight Company, Inc. (Knoxville)
Computational Mechanics Corporation (Knoxville)
CTI, Inc. (Knoxville)
Delta M Corporation (Oak Ridge)
DG Trim Products (Alcoa)
Eagle Racing (Loudon)
Eastman Chemical Company (Kingsport)
EG&G ORTEC (Oak Ridge)
EmeraChem (Knoxville)

Forged Performance Products (Oak Ridge)
Goal Line Company (Knoxville)
H. R. DeSelm (Knoxville)
HRD, Great Lakes Research Div. (Elizabethton)
IMTech Company (Knoxville)
Intellutran (Knoxville)
IntraSpec, Inc. (Oak Ridge)
J. A. Martin (Knoxville)
Jeffrey Chain Corporation (Morristown)
MATTEC, LLC (Knoxville)
Metal-Tech of Tennessee, LLC (Newport)
Microbial Insight, Inc. (Knoxville)
MINCO Acquisition Company (Midway)
MMPact, Inc. (Oak Ridge)
Multi Phase Services, Inc. (Knoxville)
Nano Instruments, Inc. (Knoxville)
Noranda Magnesium, Inc. (Franklin)
Oxyrase, Inc. (Knoxville)
Precious Metals Corporation (Sevierville)
Pyrotek, Inc. (Trenton)
Ronald K. McConathy (Kingston)
SCI-TEC, Inc. (Knoxville)
SENES Oak Ridge, Inc. (Oak Ridge)
Smelter Service Corporation (Mt. Pleasant)
Smith and Nephew (Memphis)
Technology for Energy Corporation (Knoxville)
Tennessee Center for Research and Development (Knoxville)
Textron Specialty Materials-Div. Avco Corporation (Nashville)
Third Millennium Technologies, Inc. (Knoxville)
TTE Diecasting (Oak Ridge)
VAMISTOR Corporation (Sevierville)
Walford Technologies (Oak Ridge)
William Thomas Pope (Clinton)

Texas

Agriboard Industries (Electra)
CarboMedics, Inc. (Austin)
Dallas Optical Systems, Inc. (Rockwell)
Exxon Research & Engineering Company (Annadale)
F. W. Gartner Thermal Spray Company (Houston)
Ludlum Measurement Inc. (Sweetwater)
Mogas Industries (Houston)
Poco Graphite Inc. (Decatur)
Robert Hageman (Austin)
Smith International, Inc. (Houston)
Stone & Webster Engineering Corp. (Houston)
Tycom Corporation (Austin)

Utah

LOTEC, Inc. (West Valley City)
Mantic Corporation (Salt Lake City)

Virginia

B&W Nuclear Technologies (Lynchburg)
Babcock & Wilcox/McDermott Technology, Inc. (Lynchburg)
E. R. Johnson Associates, Inc. (Fairfax)
Energy Recovery, Inc. (Virginia Beach)
HY-Tech Research Corporation (Radford)
Materials Modification, Inc. (Fairfax)
Materials Technologies of Virginia (Blacksburg)
Metalspray North America LLC (Richmond)
National Electronics Manufacturing Initiative, Inc. (Herndon)
Philip Morris Inc. (Richmond)
RAND Corporation (Arlington)
Reynolds Metal Company (Richmond)
Soil and Land Use Technologies (McLean)
Synterials, Inc. (Herndon)

Washington

Chiroscience R&D Inc./Darwin Molecular (Bothell)
Galvalume Sheet Producers of North America (Kalama)
Kyocera Industrial Ceramics Corp. (Vancouver)
Northwest Numerics and Modeling, Inc. (Seattle)
Weyerhaeuser Company (Tacoma)

West Virginia

Huntington Alloys/Special Metals Div. (Huntington)
INCO Alloys, Inc. (Huntington)
Weirton Steel Corporation (Weirton)

Wisconsin

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

GOVERNMENT AND OTHER FACILITIES—20

Alabama

NASA/Marshall Space Flight Center (Huntsville)

California

NASA-Jet Propulsion Laboratory (Pasadena)
Space and Naval Warfare Systems Center (San Diego)

Colorado

U.S. Bureau of Mines Albany Research Center (Golden)

Florida

National High Magnetic Field Laboratory (Tallahassee)

Illinois

National Coil Coating Association (Chicago)

Maryland

NAHB Research Center (Upper Marlboro)
National Institute of Standards and Technology (Gaithersburg)
U.S. Food and Drug Administration (Rockville)

Massachusetts

Single Ply Roofing Institute (Needham)

Ohio

NASA Glenn Research Center (Cleveland)

Virginia

Army Research Laboratory (Fort Belvoir)
NASA Langley Research Center (Hampton)
United States Army Research Laboratory (Fort Belvoir)

Washington, D.C.

American Iron and Steel Institute
Appliance Industry/Government CFC Replacement Consortium Inc.
Federal Highway Administration, U.S. Department of Transportation
National Highway Traffic Safety Administration
Society of the Plastic Industry/Spray Polyurethane Foam Division
Structural Insulated Panel Association

UNIVERSITIES—166

Alabama

Alabama A&M University (Normal)
Auburn University (Auburn)
Southern Research Institute (Birmingham)
Tuskegee University (Tuskegee)
University of Alabama (Huntsville)
University of Alabama (Tuscaloosa)

Arizona

University of Arizona (Tucson)
Arizona State University (Tempe)

Arkansas

University of Arkansas (Fayetteville)

California

California Institute of Technology/Jet Propulsion Laboratory (Pasadena)
California State University (Los Angeles)
Naval Postgraduate School (Monterey)
Stanford University (Stanford)
University of California (Berkeley)
University of California (Davis)
University of California (Irvine)
University of California (Los Angeles)

University of California (La Jolla)
University of California (Santa Barbara)
University of California (Santa Cruz)
University of Southern California (Los Angeles)

Colorado

Colorado School of Mines (Golden)
University of Colorado (Boulder)
University of Denver (Denver)

Connecticut

University of Connecticut (Storrs)
Yale University (New Haven)

Delaware

University of Delaware (Newark)

Florida

Florida A&M University (Tallahassee)
Florida Atlantic University (Boca Raton)
Florida International University (Miami)
Florida State University (Tallahassee)
University of Central Florida (Orlando)
University of Florida (Gainesville)

Georgia

Georgia Institute of Technology (Atlanta)
Institute of Paper Science and Technology (Atlanta)

Hawaii

University of Hawaii (Honolulu)

Illinois

Illinois Institute of Technology (Chicago)
Northwestern University (Evanston)
Southern Illinois University (Carbondale)
University of Illinois (Urbana)

Indiana

Indiana University (Indianapolis)
Purdue University (West Lafayette)
Purdue University Calumet (Hammond)
University of Notre Dame (Notre Dame)

Iowa

Iowa State University (Ames)

Kansas

Kansas State University (Manhattan)
Wichita State University (Wichita)

Kentucky

Berea College (Berea)
Eastern Kentucky University (Richmond)
University of Kentucky (Lexington)
University of Louisville (Louisville)
Western Kentucky University (Bowling Green)

Louisiana

Louisiana State University (Baton Rouge)
Southern University (Baton Rouge)
University of New Orleans (New Orleans)

Maine

University of Maine (Orono)

Maryland

Johns Hopkins University (Baltimore)
U.S. Naval Academy (Annapolis)
University of Maryland (College Park)

Massachusetts

Clark University (Worcester)
Harvard University (Cambridge)
Massachusetts Institute of Technology (Cambridge)
Mount Holyoke College (South Hadley)
Northeastern University (Boston)
Tufts University (Medford)
University of Massachusetts (Amherst)

Michigan

Michigan State University (East Lansing)
Michigan Technological University (Houghton)
University of Michigan (Ann Arbor)
Wayne State University (Detroit)
Western Michigan University (Kalamazoo)

Minnesota

University of Minnesota (Minneapolis)

Mississippi

Mississippi College (Clinton)
Mississippi State University (Starkville)
The University of Mississippi (Oxford)

Missouri

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

Montana

University of Montana (Missoula)

Nebraska

University of Nebraska (Lincoln)

Nevada

DRI Institute (Reno)
University of Nevada (Reno)

New Hampshire

Dartmouth College (Hanover)

New Jersey

New Jersey Institute of Technology (Newark)
Princeton University (Princeton)
Rutgers University (Piscataway)
Stevens Institute of Technology (Hoboken)

New Mexico

New Mexico Institute of Mining and Technology (Socorro)
New Mexico State University (Las Cruces)
University of New Mexico (Albuquerque)

New York

Clarkson University (Potsdam)
Cornell University (Ithaca)
New York State College of Ceramics at Alfred University (Alfred)
Polytechnic University (Brooklyn)
Rensselaer Polytechnic Institute (Troy)
Rochester Institute of Technology (Rochester)
State University of New York at Stony Brook (Stony Brook)
University of Rochester (Rochester)

North Carolina

Appalachian State University (Boone)
Duke University (Durham)
North Carolina Agricultural and Technical State University (Greensboro)
North Carolina State University (Chapel Hill)
University of North Carolina (Chapel Hill)
University of North Carolina (Charlotte)

North Dakota

University of North Dakota (Grand Forks)

Ohio

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

Oklahoma

Oklahoma State University (Stillwater)
University of Oklahoma (Norman)

Oregon

Oregon Graduate Institute of Science and Technology (Portland)
Oregon State University (Corvallis)
Portland State University (Portland)

Pennsylvania

Carnegie Mellon University (Pittsburgh)
Drexel University (Philadelphia)
Lehigh University (Bethlehem)
Pennsylvania State University (Landenberg)
University of Pennsylvania (Philadelphia)
University of Pittsburgh (Pittsburgh)

Rhode Island

Brown University (Providence)

South Carolina

Clemson University (Clemson)
Furman University (Greenville)
University of South Carolina (Columbia)

South Dakota

South Dakota State University (Brookings)

Tennessee

East Tennessee State University (Johnson City)
Fisk University (Nashville)
Maryville College (Maryville)
Tennessee State University (Nashville)
Tennessee Technological University (Cookeville)
University of Memphis (Memphis)
University of Tennessee (Knoxville)
Vanderbilt University (Nashville)

Texas

Rice University (Houston)
Southwest Research Institute (San Antonio)
Texas A&M University (College Station)
The University of Texas at Austin (Austin)
University of Houston (Houston)
University of North Texas (Denton)
University of Texas (Arlington)
University of Texas (Austin)
University of Texas (El Paso)
University of Texas-Pan American (Edinburg)

Utah

University of Utah (Salt Lake City)

Virginia

Institute for Defense Analyses (Alexandria)
Norfolk State University (Norfolk)
University of Virginia (Charlottesville)
Virginia Polytechnic Institute and State University (Blacksburg)

Washington

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

Washington, D.C.

Georgetown University Medical Center
Howard University
The George Washington University

West Virginia

West Virginia University (Morgantown)

Wisconsin

Marquette University (Milwaukee)
University of Wisconsin (Madison)

HTML PROPOSAL LIST

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Proposal No.	Organization	Lead Center	Proposal Title	Spokesperson	Staff Contact
2002-002	U of Tennessee Space Inst. (87)	MAUC	Thermophysical Property Analysis Proposed Research	Mary Helen McCay	Larry Walker
2002-003	U of Alabama–Birmingham (21)	TTPUC	Thermoelastic Behavior of Wc/Co Composites	Professor Krishan K. Chawla	Cam Hubbard
2002-004	U of Florida (7)	DUC	Investigation and Control of Cuinse2 Growth Kinetics and Reaction Paths	Dr. Timothy James Anderson	Cam Hubbard
2002-005	North Carolina A&T State U (2)	MAUC	Kinetic Characterization of the Temperature Programmed Synthesis of Nanostructured Molybdenum Nitride from Molybdenum Oxide Percursors	Professor Kenneth L. Roberts	Larry Allard, Cam Hubbard
2002-006	Motorola Energy System Group (3)	TTPUC	Infrared Thermal Imaging of Integrated Circuit Field Effect Power Transistors	Hossein Maleki	Hsin Wang, Ralph Dinwiddie
2002-007	NW Numerics and Modeling, Inc.	MCAUC	Characterization of Viscoplastic Si-Mo Cast Iron Behavior and Constitutive Model Calibration	Dr. Ronald A. Foerch	Edgar Lara-Curzio
2002-008	Georgia Tech (42)	MITUC	Tribological Testing of a New Biomaterial for Articular Cartilage Replacement	Dr. David N. Ku	Ron Ott
2002-009	American Boarts Crushing Company Inc. (3) and Saint-Gobain Abrasives (14)	MITUC	An Evaluation of the Effects of Grain-Size Variation on CBN Grinding Wheel Performance	Mr. Christian Winkel	Sam McSpadden
2002-010	IAP Research Inc. (1)	MCAUC	Mechanical Evaluation of NdFeB Permanent Magnetic Materials	Dr. Bhanu Chelluri	Edgar Lara-Curzio
2002-012	U of Alabama (29)	RSUC	Microstructural Analysis of Alloys Tested in Molten Zn-Al Alloy	Dr. Ramana G. Reddy	Larry Allard. Andrew Payzant
2002-013	Honeywell FM&T (1)	MAUC	Microstructural Characterization of Core-Shell Structure of Doped Strontium Titanate and Strontium Zirconate	Mr. Daniel S. Krueger	Larry Allard, Cam Hubbard

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Proposal No.	Organization	Lead Center	Proposal Title	Spokesperson	Staff Contact
2002-014	United Technologies Research Center (2)	MCAUC	Cohesive/Adhesive Strength of Ebc Coatings on Sic Ceramics at Elevated Temperature	Dr. Shanti V. Nair	Edgar Lara-Curzio
2002-015	United Technologies Research Center (3)	MCAUC	Creep and Stress Rupture Behavior of Behavior of SiC Containing an Environmental Barrier Coating (EBC)	Dr. Shanti V. Nair	Edgar Lara-Curzio
2002-017	Vanderbilt U (15)	MITUC	Characterization of Ablation Craters in Polyacrylamide Gels and Single Crystals	Dr. Richard F. Haglund	Sam McSpadden
2002-018	U of Tennessee (88)	MAUC	Two-Dimensional Dopant Profiling of Shadow Junctions By Off-Axis Electron Holography	Professor David C. Joy	Larry Allard
2002-019	ORNL, Chem. Sci. Div. (20)	MAUC	Confined Reactor Synthesis of Nanoparticles	Dr. Adam J. Rondinone	Larry Allard, Ian Anderson
2002-020	Kansas State U (5)	MAUC	Sublimation Crystal Growth of Aluminum Nitride	Professor James H. Edgar	Harry Myer
2002-021	Hemlock Semiconductor Corp. (1)	RSUC	Residual Stresses in Polycrystalline Si Boules	Dr. Jonathon J. Host	Thomas Watkins, Andrew Payzant
2002-023	U of Wisconsin (5)	DUC	Measurement of High-Temperature Lattice Parameters of Rare Earth Metal Disilicides	Professor Y. Austin Chang	Claudia Rawn
2002-024	New Mexico Tech (8)	DUC	Development of New Types of Solid Oxide Fuel Cell Materials	Dr. Junhang Dong	Andrew Payzant, Larry Allard
2002-025	Ford Motor Company (14)	TTPUC	Non-Destructive Testing of Ultrasonic Welding of Aluminum Components Using IR Thermography	Dr. Elizabeth Hetrick	Hsin Wang, Ralph Dinwiddie
2002-026	U of Tennessee Space Institute (94)	TTPUC	Study of Temperature Distribution in Ceramic Coatings Attained During Laser Assisted Surface Treatment of Metal Substrates	Professor Narendra B. Dahotre	Hsin Wang, Tom Watkins

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Proposal No.	Organization	Lead Center	Proposal Title	Spokesperson	Staff Contact
2002-027	U of Missouri–Columbia (3)	MCAUC	High Temperature Mechanical Testing for Finite Element Prediction of Residual Stress in a Spot Weld Using Temperature Dependent Mechanical Properties of the Materials	Dr. Sanjeev K. Khanna	Edgar Lara-Curzio
2002-028	Stoody Company (1)	MCAUC	Creep Rupture Behavior of Modified 9Cr-1Mo Flux Cored Arc Weldments	Dr. Ravi Menon	Bob Swindeman, Edgar Lara-Curzio
2002-029	Clemson U (23)	MAUC	High-Resolution Electron Microscopy Study on Semiconductor and Metal Nanoparticles	Professor Ya-Ping Sun	Larry Allard
2002-030	NASA Langley Research Center (9)	MAUC	Synthesis, Characterization and Application of Single-Walled Carbon Nanotubes and Nanocomposites	Dr. Catharine C. Fay	Larry Allard, Michael Lance
2002-031	ORNL, Metals and Ceramics Division (22)	DUC	Reduction of Thermal Anisotropy in Ternary Ti_5Si_3 Intermetallics	Joachim H. Schneibel	Thomas Watkins
2002-032	U of Florida (7)	RSUC	Fracture and Fatigue Characterization of High Strength Steel Alloys Coated with Tungsten Carbide Cobalt Coatings	Darryl P. Butt	Tom Watkins
2002-033	North Carolina State U (30)	MAUC	Novel Functionally Gradient Nanostructured Diamond and Diamond-Like Films	Professor Jagdish Narayan	Larry Allard, Edgar Lara-Curzio, Peter Blau
2002-034	Brown U (3)	MCAUC	Nanoindentation of Carbon-Nanotube Reinforced Composite Coatings	Professor Brian W. Sheldon	Laura Riester
2002-035	Milacron Marketing Co. (5)	MITUC	Productivity Improvement with Chemically Treated Grinding Wheels	Mr. Greg J. Foltz	Sam McSpadden
2002-036	Texas A&M U (5)		Ceramic Foam Ligament Characterization	Dr. Ozden O. Ochoa	Edgar Lara-Curzio

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Proposal No.	Organization	Lead Center	Proposal Title	Spokesperson	Staff Contact
2002-037	American Boarts Crushing (5) and Milacron Inc. (6)	MITUC	An Increased Understanding of How the Fracture Mechanism, Morphology and Friability of Cubic Boron Nitride Along with Wheel Fabrication Conditions Influence Material Removal Properties in Vitrified Grinding Wheels	Mr. Christian Winkel	Sam McSpadden, P. Blau
2002-038	DaimlerChrysler Corporation (1)	MAUC	Failure Methodology for Ferro Phos Powder Metal (Dcx Spec Ms8127a)	Mr. Dale C. Wetzel	Larry Allard
2002-039	U of Tennessee (95)	TTPUC	Microscopic IR Imaging of Fatigue Cracks	Professor Peter K. Liaw	Hsin Wang, Edgar Lara-Curzio
2002-040	Visteon Corporation (2)	DUC	Liquidus Temperatures Measurements Using High-Temperature X-Ray Diffraction	Dr. Edward N. Boulos	Andrew Payzant
2002-041	Saint-Gobain Abrasives	MCAUC	Strength Testing of Ceramic Abrasive Grains	Dr. Kristin Breder	Edgar Lara-Curzio, Laura Riester, Matt Ferber
2002-042	StarFire Systems Inc. (1)	TTPUC	Thermal and Mechanical Properties of Internally Porous Silicon Carbide	Dr. Walter J. Sherwood	Ralph Dinwiddie, Edgar Lara-Curzio, Wally Porter
2002-043	C-3 International (1)	MAUC	Characterization of C-3 Surface Treated Tools Steels	Mr. Mark A. Deininger	L. Allard, E. Lara-Curzio, C. Hubbard, P. Blau, S. McSpadden
2002-044	U of Tennessee (96)	DUC	Structural and Magnetic Characterization of Polymeric Inorganic-Organic Hybrid Materials	Dr. John F. Turner	Claudia Rawn
2002-045	Intellutran (1)	TTPUC	Multimodal Gross Rail Defect Detector	Daniel W. McDonald	Ralph Dinwiddie

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Proposal No.	Organization	Lead Center	Proposal Title	Spokesperson	Staff Contact
2002-046	Remaxco Technologies, Inc. (6)	MAUC	Development of a Commercial Process for Production of Silicon Carbide Fibrils	Richard D. Nixdorf	Larry Allard, E. Lara-Curzio, A. Payzant
2002-047	Mississippi State U (3)	TTPUC	The Degradation of Composite Systems During Cryogenic Operation	Dr. Judy Schneider	Ralph Dinwiddie, Wally Porter
2002-048	Sci-Tec, Inc. (1)	MAUC	Nano-Gold Tagged DNA for Medical Diagnostica and Drug Discovery	Dr. Val V. Golovlev	Larry Allard
2002-049	U of Tennessee (99))	RSUC	Measurement of Residual Stresses and Texture in Thin Films	Professor George M. Pharr	Thomas Watkins
2002-050	U of North Texas (2)	MCAUC	Study of Diamond-Like Carbon Films (Dlc) Deposited by Electrochemical Techniques	Professor Teresa D. Golden	Laura Riester, Michael Lance
2002-051	NASA–Marshall Space Flight Center (2)	MCAUC	Radiation Effects on the Mechanical Properties of Polycrystalline Diamond and Polycrystalline Silicon Thin Films	Robert L. Newton	Michael Lance
2002-052	Delphi (3)	MITUC	Sub-Scale Testing Method for Automotive Brake Lining Friction and Wear Characterization	Martin J. Reder	Peter Blau
2002-053	Georgia Institute of Technology (43)	MCAUC	Stress Measurement and Reliability Assessment of Next-Generation Microelectronic Systems	Dr. Markondeya Raj Pulugurtha	Michael Lance
2002-054	U of Tennessee (98)	MITUC	The Relationship Between Near-Surface Morphology and Near-Surface Mechanical Properties in Medical Grade Ultra High Molecular Weight Polyethylene Following UV Irradiation	Dr. Roberto Benson	Sam McSpadden
2002-055	Fisk (2)	DUC	Phase Stability in the Mg-B System at Elevated Temperatures	Arnold Burger	Claudia Rawn
2002-056	ORNL, Solid State Division (23)	DUC	Ab Initio Structure Solution of a Lanthanum Ruthenate and a Lanthanum Iridate Using Powder Diffraction Methods	Dr. Peter G. Khalifah	Claudia Rawn

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Proposal No.	Organization	Lead Center	Proposal Title	Spokesperson	Staff Contact
2002-057	U of Arkansas (1)	RSUC	Material Properties for Industrial Hard Coatings	Professor Deepak G. Bhat	Camden Hubbard
2002-058	Tufts U (3)	MAUC	Proposal to Evaluate Electron Tomography as a Method for Studying Interfaces in Reversible Electrochemical Cells	Professor Ronald B. Goldner	Larry Allard
2002-059	Ohio State U (8)	DUC	Investigations of the Nb-Al Phase Equilibrium at High Temperatures	Professor Robert L. Snyder	Andrew Payzant
2002-060	Corning Incorporated (6)	DUC	Determination of Crystal Orientation of a (100) Silicon Wafer and a (111) Calcium Fluoride Laser Prism	Dr. Harrie Stevens	Andrew Payzant
2002-061	Purdue U (3)	TTPUC	Role of Dopants in Stabilizing the Microstructure of Air Plasma-Sprayed Thermal Barrier Coatings (TBCs)	Professor Rodney Trice	Hsin Wang
2002-062	ORNL, Metals And Ceramics (23)	TTPUC	Characterization of Fe Based Bulk Metallic Glasses (BMGs)	Dr. C. T. Liu	Wally Porter
2002-063	Georgia Institute of Technology (44)	DUC	Sulfate Attack on Portland Cement: Constant Wavelength Synchrotron XRD in Support of Depth Profile Data Obtained by EDXRD	Professor Angus P. Wilkinson	Andrew Payzant
2002-064	North Carolina State U (31)	MITUC	Machining of Metallic Glass	Dr. Albert J. Shih	Sam McSpadden, C. T. Liu
2002-065	Tennessee Technological U (7)	DUC	Characterization of $MnCr_20_4$ Spinel Phase for SOFC Application	Professor Jiahong Zhu	E. A. Payzant, Larry Walker
2002-066	Michigan State U (3)	TTPUC	Flash Diffusivity Measurement of Two-Layer Material	Dr. Robert L. McMasters	Ralph Dinwiddie
2002-067	University of North Carolina–Charlotte (1)	MITUC	Single Point Turning of Silicon Nitride	Dr. John A. Patten	Sam McSpadden
2002-068	U of Central Florida (3)	TTPUC	Thermal Conductivity of Polymer-Derived Ceramics	Professor Linan An	Hsin Wang

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Proposal No.	Organization	Lead Center	Proposal Title	Spokesperson	Staff Contact
2002-070	Ford Motor Company (15)	MAUC	Microstructural Changes in Nox Trap Materials Under Lean and Rich Conditions at High Temperature	Dr. Chaitanya K. Narula	Larry Allard
2002-071	Georgia Institute of Technology(45)	DUC	Phase Transitions and Thermal Expansions in Low Thermal Expansion Compositions (M+)x[Zr1-x(M3+)x]P207 and (M3+)0.5(M5+)0.5P207	Professor Angus P. Wilkinson	Andrew Payzant
2002-072	U of Tennessee (100)	MAUC	Electron Beam Stimulated Desorption Studies for Nano-Scale Processing Applications	Professor Philip D. Rack	Harry Meyer
2002-073	Pennsylvania State U (29)	MAUC	Study of the Feasibility of the Use of Platinum Loaded Nanoporous Carbon as Electrodes in Proton Exchange Membrane Fuel Cells	Professor Henry C. Foley	Larry Allard
2002-074	Georgia Tech (46)	RSUC	Plasticity Induced Damage in Reciprocating Grinding of Polycrystalline Gamma Titanium Aluminide	Dr. Steven Danyluk	Tom Watkins
2002-075	North Carolina State U (32)	RSUC	Laser Physical Vapor Deposition of Boron Carbide Coatings	Dr. Jag Kasichainula	Tom Watkins
2002-077	U of Arkansas (2)	MCAUC	Investigation of Ultra-Low-Load Nanoindentation in GaAs (100)	Dr. Ajay P. Malshe	Laura, Riester, Edgar Lara-Curzio, Larry Allard
2002-078	North Carolina State University (33)	TTPUC	Thermal Conductivity of Thermoelectric Thin Films	Dr. Jag Kasichainula	Ralph Dinwiddie, Hsin Wang
2002-079	U of Tennessee (101)	MCAUC	Evaluation of Mechanical Properties of Laser Induced Rapidly Solidified Fe-Oxides/A319 Al Composite Coatings	Professor Narendra B. Dahotre	Laura Riester, Peter Blau
2002-080	Colorado School of Mines(1)	DUC	Low Temperature Powder Diffraction Studies of Clathrate Hydrates	Professor E. Dendy Sloan	Claudia Rawn

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Proposal No.	Organization	Lead Center	Proposal Title	Spokesperson	Staff Contact
2002-082	Vanderbilt U (15)	MCAUC	Graphitic Carbon Nanofiber/Organic Polymer Composites as Structural Materials Having Superior Mechanical Properties	Professor Luoyu Xu	Edgar Lara-Curzio
2002-083	Rice U (6)	MAUC	Fib Processing of Spun Fibers for the Continued Growth of SWNTs	Professor Richard E. Smalley	Larry Allard
2002-084	Honeywell Electronics	RSUC	HTXRD Analysis of Thermal Expansion and Residual Stress in Semiconductor Barrier Layers	Dr. Eal H. Lee,	Cam Hubbard, Scott Speakman
2002-085	U of Pennsylvania (1)	TTPUC	Thermal Conductivity of Ceramic Filled Polymer Composites	Professor I-Wei Chen	Hsin Wang
2002-086	MIT Lincoln Laboratory (1)	TTPUC	Thermal Conductivity Measurements for Several Laser Materials in the 80-320 K Temperature Range	Dr. Roshan L. Aggarwal	Ralph Dinwiddie
2002-087	Detroit Diesel Corporation (5)	MITUC	Tribological Evaluation of Materials for High Temperature Unlubricated Diesel Engine Applications	Dr. Nabil S. Hakim	Peter Blau
2002-088	Dow Corning (13)	MAUC	Study of Adhesion Related Interface Layers in Multilayer Film Structures	Dr. Wei Chen	Larry Allard
2002-089	U of Arkansas (3)	RSUC	Investigation of the Residual Stresses in PVD and CVD Hard Coatings on Cutting Tools at Room and Elevated Temperatures	Professor Deepak G. Bhat	Andrew Payzant
2002-090	U of Tennessee(102)	RSUC	Evolution of Microstructure and Lattice Strain During Aging and Primary Creep Deformation of Titanium Aluminides	Professor Hahn Choo	Cam Hubbard, Andrew Payzant
2002-091	U of Tennessee (103)	RSUC	Measurement of the Plastic Damage Zone Around Crack Tip Using Neutron Diffraction	Professor Peter K. Liaw	Cam Hubbard
2002-093	Caterpillar (16)	MCAUC	Life Prediction of Silicon Nitride Valves for Diesel and Natural Gas Engines	Dr. Lou Balmer Millar	Edgar Lara-Curzio
2002-094	North Carolina State U (34)	RSUC	Mapping of Residual Stresses in Welded Joints	Dr. Tasnim Hassan	Cam Hubbard

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Proposal No.	Organization	Lead Center	Proposal Title	Spokesperson	Staff Contact
2002-095	U of Oklahoma (1)	MAUC	Characterization of Single-Walled Carbon Nanotubes Produced by Catalytic Disproportionation of Co: Structure, Dispersion and State of Aggregation	Professor Daniel E. Resasco	Larry Allard
2002-096	U of Arkansas (4)	MAUC	An Investigation of Interdiffusion Between A Cutting Tool and Selected Workpiece Materials by Electron Probe Microanalysis	Professor Deepak G. Bhat	Larry Walker
2002-097	U of Michigan (10)	RSUC	Transformations in Ni-Based Refractory Alloy Glasses (Rag)	Dr. John C. Bilello	Cam Hubbard

PUBLICATIONS AND PRESENTATIONS, FY 2002

Note: Asterisks indicate HTML staff members.

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