ORNL/TM-2007/095

HIGH TEMPERATURE MATERIALS LABORATORY USER PROGRAM

19th ANNUAL REPORT
OCTOBER 1, 2005 – SEPTEMBER 30, 2006

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July 2007

Prepared by
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managed by
UT-BATTELLE, LLC
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-00OR22725

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ACRONYMS AND ABBREVIATIONS

ACEM	aberration-corrected electron microscope
DOE	U.S. Department of Energy
DUC	Diffraction User Center
EERE	DOE Office of Energy Efficiency and Renewable Energy
FCVT	FreedomCAR and Vehicle Technologies Office, EERE
FIB	focused ion beam
FWMUC	Friction, Wear, and Machinability User Center
GM	General Motors
HFIR	High Flux Isotope Reactor
HTML	High Temperature Materials Laboratory
IR	infrared
MAUC	Materials Analysis User Center
MCAUC	Mechanical Characterization and Analysis User Center
MEMS	microelectromechanical systems
MMC	metal matrix composite
NIST	National Institute of Standards and Technology
NRSF2	Neutron Residual Stress Mapping Facility
NSLS	National Synchrotron Light Source
OFCVT	Office of FreedomCAR and Vehicle Technologies
ORNL	Oak Ridge National Laboratory
QSS	quasi-steady state
RSUC	Residual Stress User Center
SEM	scanning electron microscope
SOFC	solid-oxide fuel cell
STEM	scanning transmission electron microscope
TBC	thermal barrier coating
TEM	transmission electron microscopy
TMS	The Minerals, Metals & Materials Society
TTPUC	Thermography and Thermophysical Properties User Center
UCF	University of Central Florida
UT	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 (HTML) is a national user facility designed to solve materials problems that limit the efficiency and reliability of advanced energy conversation systems. Sponsored by the U.S. Department of Energy (DOE) Office of Transportation Technologies in the Office of Energy Efficiency and Renewable Energy (EERE), the HTML provides researchers from industry, academia, and government with access to a skilled staff and numerous sophisticated, often unique devices for materials characterization. Located at Oak Ridge National Laboratory (ORNL), the 37,511-ft^2 building houses six "user centers" — clusters of specialized equipment designed for specific types of properties measurements. The HTML also manages a neutron beamline facility at the High Flux Isotope Reactor (HFIR) at ORNL and a synchrotron beamline at Brookhaven National Laboratory's National Synchrotron Light Source (NSLS).

The High Temperature Materials Laboratory has been supporting the development of advanced materials since the mid-1980s.

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

The Program

Within the HTML are programs that 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. Nonproprietary research is provided free of charge if the user publishes the information produced; projects typically last from a few days to a couple of weeks. The major provison is that the results must be submitted for publication within six months after completion of the research.

For proprietary research, which involves a fee, the user and HTML staff estimate the amount of HTML staff time required to complete the work. The user agrees to pay for the time at an hourly rate specified by DOE before research begins, and the user owns the research data.

Work is performed for other federal agencies via direct funding or through cooperative research and development agreements, which typically consist of a cost-sharing arrangement between the HTML and the outside organization but can also include 100 percent funds-in work. The HTML can also characterize materials for another organization on a noncompetitive, full-cost-recovery basis under a "work for others" agreement.

Most projects involve materials primarily related to transportation technologies. Materials characterized at the HTML include ceramics; metal-, polymer-, and ceramic-matrix composites; lightweight materials such as aluminum and magnesium alloys; steels; and electronic materials.

The User Centers

Materials Analysis User Center (MAUC)

Contact: Dr. Larry Allard (865-574-4981) allardlfjr@ornl.gov

The MAUC provides a research staff of technical experts for characterizing the structure and chemistry of advanced materials. Research specialties include characterization of nanophase materials, nanoparticulates, and multilayer surface films; structural ceramics; and electron holography. Electron microscopy and surface chemical analysis are employed to determine surface chemistry and microstructure down 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.

Friction, Wear, and Machinability User Center (FWMUC)

Contact: Dr. Peter Blau (865-574-5377) blaupj@ornl.gov

The mission of the FWMUC is to enable the selection, development, and use of advanced materials and surface treatments by characterizing their frictional behavior, durability under wear-causing conditions, and response to machining processes, especially grinding. The FWMUC has testing capabilities that support research in a series of complementary subject areas: friction, lubrication, and wear (tribology); dimensional metrology; and machining. Testing machines include equipment for instrumented scratch testing and abrasion by belts. Dimensional measurement and surface examination capabilities include a high-frequency scanning acoustic microscope, a coordinate measuring machine, and instruments for measuring surface texture and topography. Rounding out the suite of capabilities, FWMUC offers a high-precision diamond-turning machine, a unique grindability tester, and several instrumented machine tools.

Mechanical Characterization and Analysis User Center (MCAUC)

Contact: Dr. Edgar Lara-Curzio (865-574-1749) laracurzioe@ornl.gov

The MCAUC staff has expertise in the mechanical characterization of a wide variety of functional and structural materials, including metals, ceramics, polymers, and composites. The MCAUC manages numerous mechanical test frames with uniaxial and multiaxial capabilities to conduct tests in tension, compression, flexure, torsion, shear, and internal pressurization in controlled environments and at elevated temperatures using standard or customized specimens. The MCAUC staff also has expertise in analytical modeling, failure, finite-element stress, and life-prediction analyses of materials and structures.

Diffraction User Center (DUC)

Contact: Dr. Andrew Payzant (865-574-6538) payzanta@ornl.gov

The DUC uses variable-temperature laboratory, synchrotron X-ray, and neutron diffraction techniques to characterize metals, ceramics, and polymers. The 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 a vacuum and up to 1500°C in air. DUC users have access to the National Synchrotron Light Source (NSLS) at Brookhaven (NY) National Laboratory (see RSUC section below).

Residual Stress User Center (RSUC)

Contact: Dr. Camden Hubbard (865-574-4472) hubbardcr@ornl.gov

RSUC equipment and staff are able to characterize stresses in materials ranging from thin films to large industrial components. To assist in studying important materials design properties such as fatigue life, fracture strength, onset of yield, and microcracking, the RSUC has two principal areas of expertise: X-ray diffraction (XRD) 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 parallel beam. The neutron residual stress facility, located at ORNL's High Flux Isotope Reactor (HFIR), includes a special neutron spectrometer for rapid data collection and computer capabilities for data analysis. The spectrometer instrumentation allows researchers to quickly measure and map the stress fields inside relatively large solid objects.

Users can also access the Brookhaven (NY) National Synchrotron Light Source (NSLS) through the RSUC. The beamline that the HTML maintains at Brookhaven National Laboratory has structure and residual stress analysis capability.

Thermography and Thermophysical Properties User Center (TTPUC)

Contact: Dr. Ralph Dinwiddie (865-574-7599) dinwiddierb@ornl.gov

TTPUC researchers study thermal stability, expansion, and thermal conductivity of materials to 1400°C. A laser flash instrument measures thermal diffusivity at temperatures up to 1900°C. The center also possesses a large collection of high-speed, high-sensitivity infrared (IR) cameras for capturing thermal events digitally, allowing on-line or post-operation measurement of temperatures during rapid transient events. Thermography capability is portable and may be used for off-site user projects.

HTML DIRECTOR'S REPORT

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

The HTML User Program continued its work with industrial, academic, and governmental users this year, accepting 84 new projects and developing 19 new user agreements. The table below presents the breakdown of these statistics.

The plot on the following page depicts the continued growth in user agreements and user projects. The total number of HTML proposals has now exceeded 1,450. The number of new HTML user agreements bodes well for the future. A list of the 19 new agreements executed during FY 2006 between the HTML and universities or private-sector companies is on page 53.

Table 1. Statistics of HTML operations for FY 2003–FY 2006 (showing user proposals and user agreements)

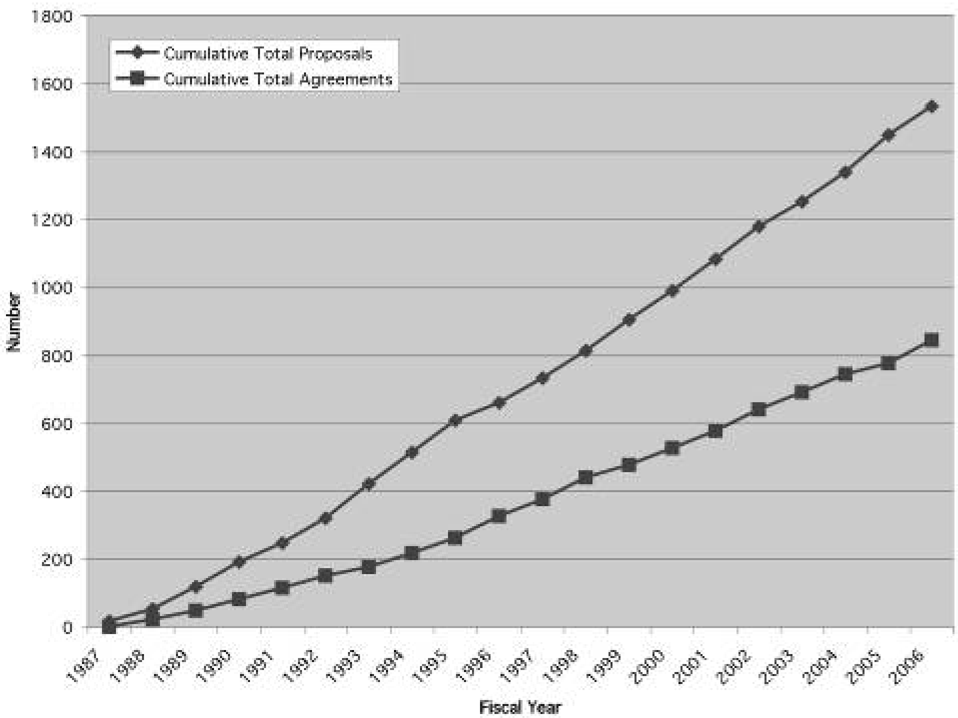
FY	New proposals				Cumulative proposals			
	Total	**Industrial**	**Academic**	**Other**	***Total***	**Industrial**	**Academic**	**Other**
2003	72	27	44	1	1253	514	693	46
2004	87	28	54	5	1340	542	747	51
2005	110	56	52	2	1450	598	799	53
2006	84	38	45	1	1534	636	844	54
	New agreements				**Cumulative agreements**			
	Total	**Industrial**	**Academic**	**Other**	***Total***	**Industrial**	**Academic**	**Other**
2003	50	44	4	2	692	402	265	25
2004	53	47	5	1	745	449	270	26
2005	33	27	6	0	778	476	276	26
2006	19	18	1	0	846	519	286	41

Program highlights this year included several outstanding user projects (some of which are highlighted in later sections), the annual meeting of the HTML Programs Guidance and Evaluation Panel, and continued progress in equipment and facilities development. For instance, this year we intensified our thrust into the realm of thermoelectric materials characterization, mirroring the renewed emphasis at DOE for using these materials to produce electric power from waste heat. The HTML acquired instrumentation to measure bulk materials' thermal conductivity, electrical resistivity, and Seebeck coefficient as a function of temperature. Since many thermoelectric devices consist of thin-film generators, we initiated the purchase of a device to provide similar data for thin-film thermoelectrics.

Also, after years of requests from potential customers, we located and refurbished a total hemispherical emittance measurement instrument and have made it available to users. Other instruments were also added or upgraded.

The Aberration-Corrected Electron Microscope (ACEM) proved to be a highly capable instrument, collecting data on and photographs of many materials at the atomic-resolution level (see Chap. 1, "Materials Analysis User Center"). It has passed nearly all of its qualification tests, and we anticipate final acceptance early next fiscal year.

Final funding ($2M) was received this year for HTML's participation in construction and operation of the VULCAN beamline at the Spallation Neutron Source (SNS). The SNS, an extremely high-flux pulsed-neutron source, is in the final stages of construction at ORNL, and the VULCAN beamline is to be the "materials" diffractometer. These funds will provide for development of an off-line specimen alignment system, which will allow users to have their experiment completely set up at an off-line location, so when their project time begins they can put the specimen on the beam and begin taking data instantly. The off-line alignment capability will save much precious beamline time. These funds will also be used to acquire extra detectors to further increase VULCAN's coverage and speed and to support staff for VULCAN operations.



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

1. MATERIALS ANALYSIS USER CENTER

User Center Members

Larry Allard, Leader
Carolyn Wells, Administrative Secretary
Doug Blom
Dorothy Coffey
Jane Howe
Harry Meyer
Ted Nolan
Larry Walker

The Materials Analysis User Center (MAUC) has facilities and a staff of technical experts for characterizing the structure and chemistry of advanced materials. Emphasis is placed on using MAUC resources to relate microstructure to materials performance. The MAUC is a suite of laboratories with the latest-generation electron microscopes and surface analysis instruments, all available to visiting researchers. Research specialties include characterizing nanophase materials such as catalysts, fullerenes (carbon nanotubes), and nanoparticulates; investigating structural ceramics; electron holography (for example, for dopant profiling in semiconductors); and characterizing multilayer surface films. The JEOL aberration-corrected electron microscope allows imaging of single atoms and crystal structures at the sub-angstrom resolution.

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)
- 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 with microsampling capability
- JEOL 2200FS-AC aberration-corrected scanning transmission electron microscope (STEM)/transmission electron microscope (TEM)
- Hitachi 3400 variable-pressure SEM with EDS
- Leica UCT ultramicrotome with cryosectioning capability

Selected Highlights

Metal Atom Arrangement on Catalyst Supports

Industry Collaborator: Steven Bradley
HTML Staff: Douglas Blom and Larry Allard

HTML staff members Dr. Douglas Blom and Dr. Larry Allard worked with Dr. Steven Bradley of UOP LLC to study the mechanism of Pt catalyst cluster formation on Al_2O_3 and SiO_2 supports. It is well known that ultradispersed Pt is not highly catalytically active and that only when the Pt cluster size reaches 1 nm does the catalyst exhibit high turn-over frequency. The mechanism by which Pt atoms congregate to form the active clusters is not understood, nor is the morphology of an active cluster well characterized. The atomic-resolution imaging capabilities of the ACEM allow for the study of the fresh catalyst with dispersions of single Pt atoms and small Pt clusters (Fig. 1-1 next page). Coupled with the ex situ reactor capabilities, for the very first time, it will be possible to study the formation of the catalytically active Pt species. Using software written by Dr. Blom, a series of images can be automatically recorded and saved, then properly registered to form a “movie” sequence. The two frames from the “movie” shown in Fig. 1-1 are taken from a fresh sample of Pt on SiO_2. The images show the formation of a larger Pt cluster from two smaller clusters (atomic groups on upper left) during the STEM imaging.

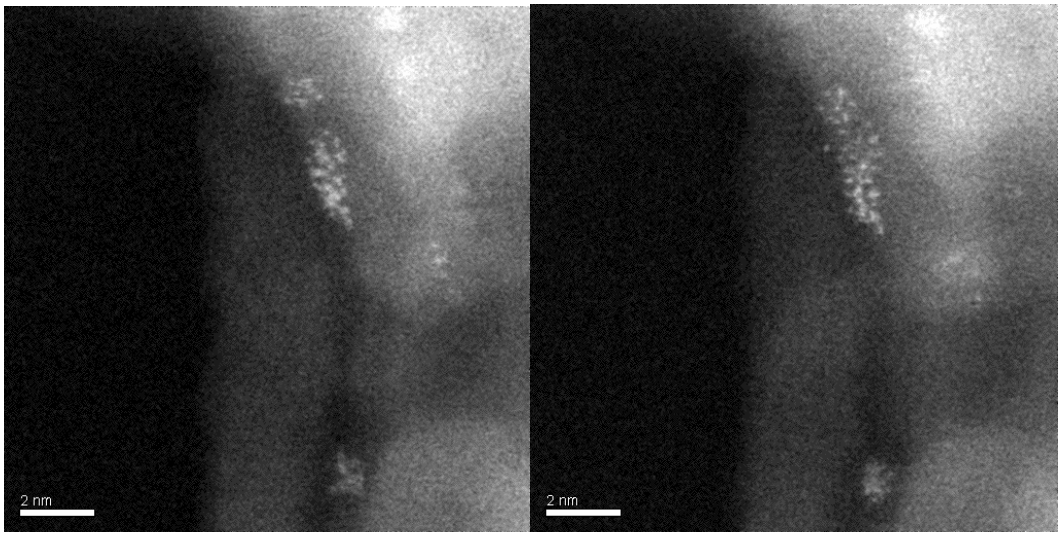


Fig. 1-1. Pt “rafts” on SiO_2 support material. Left image is several seconds before right image. Note two atom clusters joining (upper left) to form larger cluster on right.

The bright objects in Fig. 1-1 are individual Pt atoms. The cluster, which has a “raft-like” morphology, is only 1 to 2 atoms thick, although about 1 nm in lateral dimension. The behavior of the rafts under the influence of the electron beam may mimic elevated-temperature exposures; parallel work with HTML’s ex situ reactor system should shed more light on these mechanisms.

Application of Scanning Auger Microanalysis to Steel Casting Failure

Industry Collaborator: Harry Walton
HTML Staff: Harry Meyer

Mr. Harry Walton, a consultant to the steel and bearing industries, worked with the HTML’s Dr. Harry Meyer to study the cause of embrittlement of large cast-steel structures used in a hammer mill for automobile crushing and recycling. Newly cast hammers appeared to be extremely brittle and were failing during initial testing immediately after fabrication. In the Auger lab, a small piece of a tested sample, approximately 2 × 2 mm, was fractured in air and inserted directly into the analysis chamber within 2 min of fracturing. Two distinct types of surfaces were observed in secondary electron images of the surface (see Fig. 1-2a and 1-2c next page).

Both regions show a line of different but similarly sized spots: those in (a) are protrusions; those in (b) are pits. Elemental analysis revealed that the nominal surface of region (a) was primarily Fe (from the steel) with C and O present due to fracturing in air.

The protrusions showed evidence of Al, N, and S. Similar analysis of region (c) showed that the nominal surface was covered by a film rich in Al and N, probably AlN. The pits showed traces of S but were mainly Fe (see Fig. 1-2c and d). Argon ion sputter profiling confirmed that the C and O of region (a) were adsorbed species due to brief air exposure following fracture. Sputter profiling on region (c) showed that the AlN film was approximately 35 nm thick. The conclusion was that embrittlement was caused by AlN forming at the grain boundaries during casting.

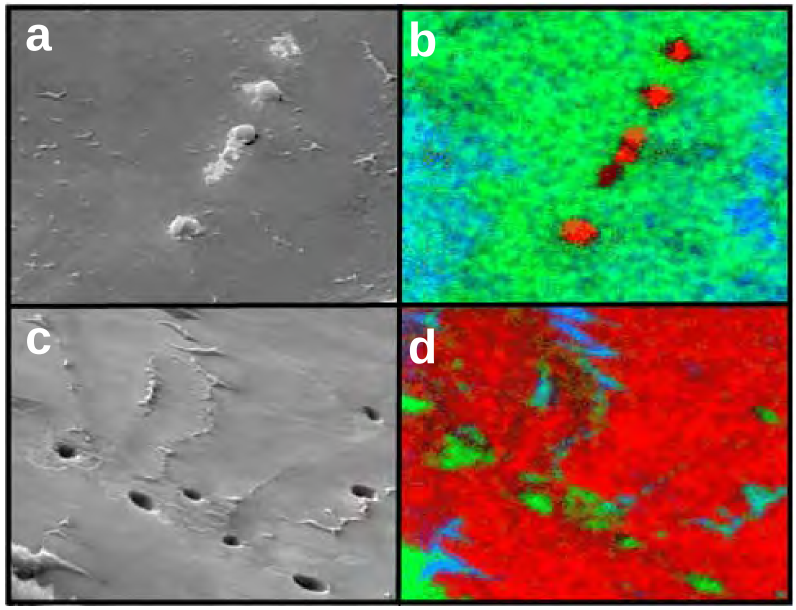


Fig. 1-2. SE micrographs and Auger elemental maps of the regions (a) and (c), with (b) and (d) the corresponding elemental maps. The top color map shows a combination of Fe (green), C (blue), and S (red), while the bottom map shows a combination of Fe, C, and N.

Remote Transmission Electron Microscopy Study Helps Small Business in Ohio

Industry Collaborator: Max Lake
HTML Staff: Jane Howe

HTML staff member Dr. Jane Howe carried out a remote TEM research session, working with Mr. Max Lake and his associates from a Cedarville, Ohio, firm. Mr. Lake is the president of Applied Sciences, Inc. (ASI), a small business that specializes in producing carbon nanofibers and diamond thin films, primarily for military applications. The nanofibers produced by ASI are 100 to 200 nm in diameter and have a two-tier structure (see Fig. 1-3). The inner layer of the carbon fiber, which is catalytically grown, has a highly graphitic structure. The outer layer is deposited later and has a "turbostratic" structure (the graphite planes are not directly registered with adjacent planes).

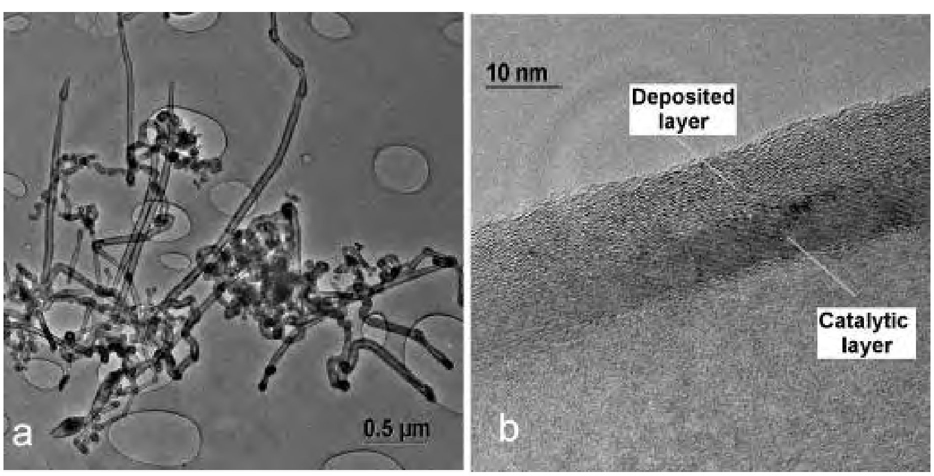


Fig. 1-3. The ASI carbon nanofibers (a) and the two-tier microstructure (b).

The goal of this HTML project is to study the microstructure of the carbon nanofibers using HTML's remote high-resolution TEM capability. It is noteworthy that Mr. Lake is wheelchair-bound as a result of polio. The remote capability on the Hitachi HF-2000 TEM made it possible for him to participate in real time in the TEM session from his Ohio office. ASI is very pleased with the TEM results, and the study will continue. Future remote sessions are already scheduled.

MAUC News

Drs. Larry Allard and Doug Blom were part of a team (Fig. 1-4) that won a UT-Battelle "Engineering Development by a Team" award at the 2006 Awards Night ceremony. The citation read, "For exceptional collaboration in the conceptualization, plan, design, and supervision of construction of a unique, state-of-the-art, environmentally stable laboratory that ensures optimum operation of advanced electron optical instruments." At the December ceremony, the team was also presented with a Director's Award in the team category, for "Outstanding Accomplishment in Science and Technology." The Director's Award included a small monetary token of appreciation from UT-Battelle, which made the holidays a bit brighter for the awardees.

Fig. 1-4. Engineering Team members (left to right, sitting) John Mayo, Larry Allard, and Doug Blom; (standing) Lynn Degenhardt, Bill Sides.

The Microscopy Society of America (MSA) notified Drs. Allard and Blom that they were elected, respectively, leader-elect and secretary-treasurer of the MSA Focused Interest Group on "Materials Research in an Aberration-Free Environment." The elected leader of the group is Dr. Ian Anderson, former group leader of the Microscopy, Microstructures, and Microanalysis group in the Materials Science and Technology Division (of which Larry and Doug are members) at ORNL. Dr. Anderson recently moved to a new position at the National Institute of Standards and Technology). Therefore, beginning January 2006, the new leadership of the Focused Interest Group will have a strong ORNL flavor. Larry and Doug look forward to working with Ian to further the science of aberration-corrected electron microscopy (ACEM) over the next several years through their activities in the group (e.g., workshops, symposia).

2. FRICTION, WEAR, AND MACHINABILITY USER CENTER (FWMUC)

User Center Members

Peter Blau, Leader
Christine Goudy, Group Secretary
Ron Chand
Cory Fletcher
Brian Jolly
Randy Parten
Jun Qu

The mission of the FWMUC is to enable the selection, development, and use of advanced materials and surface treatments by characterizing their frictional behavior, their durability under wear-causing conditions, and their response to machining processes, especially grinding. The mission comprises three complementary areas of characterization, and the instruments that support these areas represent a wide spectrum of capabilities: (1) friction, wear, and hardness testing; (2) measurement of form and surface roughness; and (3) grinding and machinability.

Our extensive set of friction-, wear-, and hardness-testing instruments are largely the result of support from various DOE and other-agency programs. Some instruments, such as the pin-on-disk testers, the reciprocating pin-on-flat wear machines, and the continuous-loop abrasion tester, are more generic. Other instruments, including the high-temperature scuffing tester and the sub-scale brake materials tester, have been custom-built to simulate specific applications. ORNL has regularly participated in American Society for Testing and Materials (ASTM) standardization efforts and has led the development of four standards for friction, wear, and scratch hardness testing.

Several types of numerically controlled and manual machine tools are available to guest researchers at the FWMUC. Some are instrumented to permit real-time measurement of machining process parameters. Dimensional measurement instruments are available for the precise and accurate measurement of size, form, and surface topography. The range of measurable features spans eight orders of magnitude, from centimeters to a few nanometers.

FWMUC Instruments

Tribology

- Cameron-Plint Model TE-77 reciprocating sliding wear tester
- Cameron-Plint TE-53 multimode friction and wear tester
- CSEM instrumented scratch tester
- AMTI high-temperature pin-on-disk friction and wear test system
- Knoop and Vickers microindentation hardness testing machine
- Bud Labs loop abrasion testing machine (ASTM standard)
- Elevated temperature oscillating scuffing test rig
- Pin-on-disk friction and wear testing stations (ASTM standard)
- Reciprocating friction and wear tester (ASTM standards and other configurations)
- Sub-scale instrumented brake materials tester
- Teledyne Taber portable scratch-testing apparatus

Machining

- Chand grindability testing system
- Instrumented K. O. Lee creep-feed grinder
- Instrumented Weldon cylindrical grinder
- Precitech high-precision, single-point turning system

Dimensional Metrology

- 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
- Nanoscience E-AFM atomic force microscope

Selected Highlights

Nanoparticulate Aluminum Matrix Composites Show Potential to Reduce Wear

University Collaborator: Linan An

HTML Staff: Jun Qu

Dr. Jun Qu worked with Prof. Linan An of the University of Central Florida (UCF) to investigate the effects of dispersed nanoparticles of alumina (Al_2O_3) on the friction and wear of aluminum against bearing steel. Previous studies of metal matrix composites reinforced with fine particles have been limited to concentrations of just a few percent, but UCF researchers prepared concentrations of up to 20 vol % and found significant improvements in tribological characteristics. For example, compared with pure aluminum against the same steel, aluminum-based nanocomposites containing 15 vol % of 50-nm particles exhibited friction coefficients that were 57% lower. The two nanocomposites with Al2O3 particles with nominal sizes of 50 and 200 nm performed similarly on friction behavior. This is either because (1) the reported nominal particle sizes were provided by the particle manufacturer without detailed size distributions and a later SEM examination showed a large size overlap between these two groups of particles, 30–300 and 100–500 nm, respectively; and (2) when the particle size is below a certain threshold, say 500 nm, the size effect may become less significant. This needs to be confirmed by further study. In addition, the wear rate of the nanocomposites was more than 1000 times lower than that of pure aluminum and 20 times lower than that of Al 319 (See Fig. 2-1). For the same concentration, the composites with nanometer-sized hard particles significantly outperformed those with micrometer-sized hard particles.

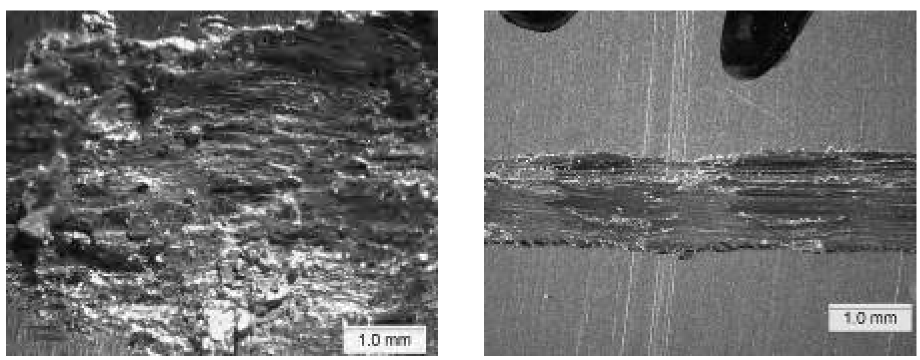


Fig. 2-1. Wear of pure Al (left) involves severe plastic deformation with adhesion and transfer to the slider, but the nanocomposite surface shows much less wear (right).

The encouraging results achieved in this study suggest a great potential of using lightweight, high-wear-resistance nanocomposite materials for critical engine bearing components including cylinder liners, cams, and fuel injectors. In addition, initial results from this project led to the award of an exploratory research effort sponsored by ORNL’s Laboratory Directed Research and Development Program.

New Slurry Abrasion Test Interests Oil Drillers

Industrial Collaborators: Ravi Menon, Frank LeClaire, and Jack Wallin
HTML Staff: Jun Qu and John Truhan

A great deal of energy is required to drill for oil and natural gas, so reducing the amount of energy in the drilling stack can reduce energy production costs. Energy is consumed not only to operate the drill head, but also to rotate pipe sections in slurry-filled casings, especially when the drilling stack turns corners deep below the ground to reach productive geological formations. Stoody Co. (Bowling Green, Kentucky) produces hard-facing alloys for use in abrasive environments such as those in drilling, and three of its engineers recently conducted an FWMUC project with Jun Qu and John Truhan (Fig. 2-2). The results of this project not only provided information on the lowest-friction, lowest-wear cladding material combinations, but also may lead to a new, cost-effective screening test for slurry abrasion.

Fig. 2-2. Researchers from Stoody Company using the multi-specimen machine in a block-on-ring configuration. Left to right: Ravi Menon, Frank LeClaire, and Jack Wallin.

ORNL staff and the three Stoody users developed a block-on-ring test in which a standardized slurry mixture, made of sand, clay, and water, is fed into the sliding interface during the wear test. Friction coefficients and wear rates can be measured in a few minutes; the much larger and more costly industry tests can take hours.

One of the interesting findings of the work was the relationship between the friction force and the wear losses of both the block and ring specimens. The wear in both cases was well represented by a polynomial expression of the form $y = ao + a1x + a2x2$, where y = weight loss (grams) and x = friction force (newtons) (see Fig. 2-3). The ratio of the block's wear loss with that of the ring varied between 4 and 11, indicating that it is important to consider the total wear of the system and to minimize the wear of the most expensive or the most difficult-to-replace component.

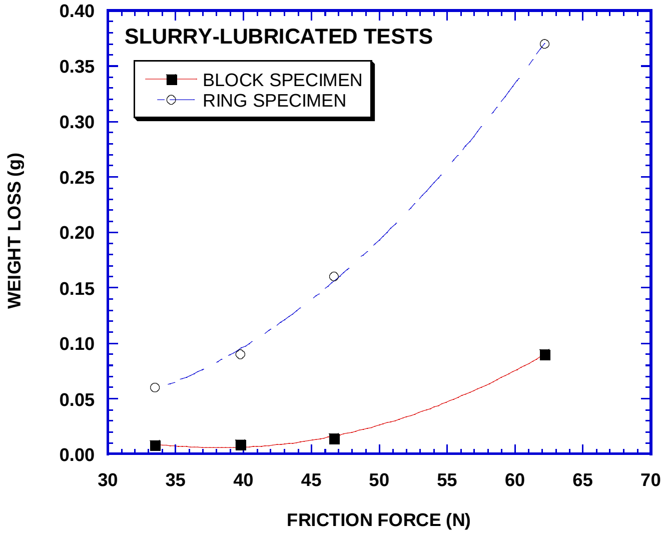


Fig. 2-3. The wear of the block and ring specimens was related to the measured friction force by a second-degree polynomial, $y = a_o + a_1x + a_2x^2$, where y = weight loss (grams) and x = friction force (newtons).

Ravi Menon, vice president of Stoody Co., and John Truhan, an FWMUC staff associate, jointly presented a summary of the preliminary slurry test results at a recent meeting of the American Petroleum Institute's (API's) casing wear working group in Houston, Texas. That group's objective is to develop an API standard for measuring casing wear, and this user project directly addresses that objective.

Deloro-Stellite Conducts High-Temperature Hardness Tests

Industry Collaborator: James Wu
HTML Staff: Peter Blau, Brian Jolly, and Jun Qu

Deloro-Stellite Corp. is a major supplier of high-temperature, high-performance alloys for the transportation industry and a variety of other demanding industrial applications. Working with Peter Blau, Brian Jolly, and Jun Qu, Dr. James Wu, chief technical officer for Deloro-Stellite in St. Louis, visited ORNL to continue a user project to characterize the elevated-temperature wear and frictional behavior of several of the company's alloys used as steel coatings. During the previous visit, a series of alloys was subjected to oscillating scuffing tests at 600°C. Several different criteria, such as time-dependent torque and post-test surface roughness, were used to rank the materials.

Dr. Wu's subsequent visit focused on measuring the effect of temperature on Vickers micro-indentation hardness of six cobalt-based alloys. Tests were performed on ORNL's high-temperature hardness testing system (Fig. 2-4a), which operates in a vacuum to retard oxidation of the test samples and the indenter. The selected test temperatures were room temperature, 200°C, 400°C, and 600°C. For certain alloys, such as Stellite 12, Vickers hardness decreased with test temperature (Fig. 2-4b). Other alloys with more complex microstructures were not as well-behaved.

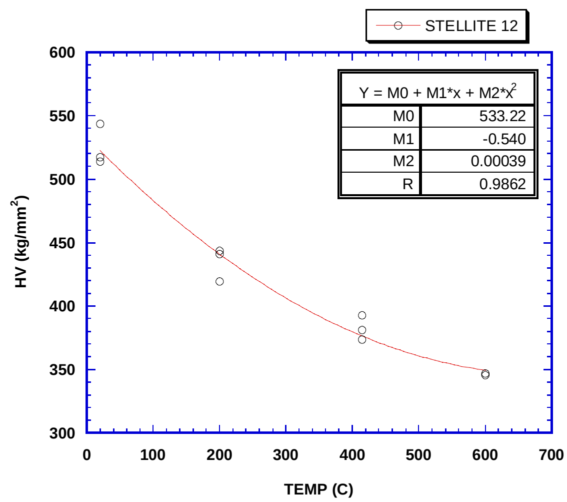


Fig. 2-4. High-temperature hardness tester vacuum chamber and lifting apparatus (left); effects of temperature on the Vickers microindentation hardness of Stellite 12 (right).

The data will be used to correlate the wear resistance of a set of cobalt-based alloys with their hardness numbers at similar temperatures, thus providing a better understanding of the effects of composition, hardness, and microstructure on the behavior of high-performance alloys in wear-critical applications. In particular, the HTML's unique hot hardness testing capability enables quantitative evaluations of high-temperature mechanical properties of specialty alloys, designed for high temperature applications such as components in exhaust-gas-recirculation (EGR) diesel engines.

Development of High-Speed Drilling for Titanium Alloys and Nanoindentation Testing of Machined Ti-6Al-4V Workpieces and Chips

University Collaborators: Rui Li and Albert Shih
HTML Staff: Peter Blau, Jun Qu, Laura Riester, Earl Shelton, and Thomas Watkins

Titanium alloys are attractive materials for aerospace and transportation technology applications because of their high specific strength, stiffness, and retention of mechanical properties at elevated temperatures. However, their widespread use in transportation technologies will depend on cost-effective manufacturing techniques. High-speed drilling technology has the potential to increase the cost-effectiveness of machining titanium and thereby promote its use in lightweight vehicles. A user project from Professor Albert Shih at University of Michigan, Ann Arbor, investigated the microstructural changes produced in titanium alloy Ti-6Al-4V from high-speed drilling. Changes studied included deformation at high strain rates and resulting alterations in microstructure, chemical composition, phase stability, and properties.

Staff from three user centers were involved in the project: Peter Blau, Earl Shelton, and Jun Qu (FWMUC) for drilling test specimens and analyzing the resulting microstructures; Thomas Watkins (DUC) for understanding the phase content in the hole walls and chips; and Laura Riester (MCAUC) for studying the localized changes in mechanical properties adjacent to hole walls and across the chip thickness using nanoindentation. University of Michigan Ph.D. student Rui Li (Fig. 2-5), conducted high-speed drilling experiments, with and without coolant, on the Sabre computerized numerically controlled vertical milling machine, which is capable of spindle speeds exceeding 16,000 rpm. The drilled titanium alloy workpieces were cross-sectioned to examine the effects of the high spindle speeds on the changes in hole-wall microstructure (Fig. 2-6). In addition to microstructural examination, XRD and nanoindentation studies were conducted. The nanoindentation work with a 4-mm-diam drill bit revealed that the load-displacement curves changed with location (Fig. 2-7 and 2-8, next page). Results indicate that using high speeds and minimal coolant suppresses the formation of the beta phase in titanium alloy and that the near-hole hardness increases, but only for drilling without lubrication. The results from this project will lead to cost-effective machining processes for titanium alloys, which have the potential for applications in the transportation industry.

Fig. 2-5. Rui Li, University of Michigan graduate student.

Mr. Li also worked with MCAUC researcher Laura Riester to quantify observed changes using a Hysitron Triboindenter in a region of a cylindrical hole formed with a 4-mm-diam drill bit. In particular, they investigated the effect of cutting speed, feed speed, and cooling fluid by obtaining indentation load vs. displacement data (Fig. 2-7). It was found that the load-displacement curves changed with location (Fig. 2-8). The results from this project will lead to cost-effective machining processes for titanium alloys.

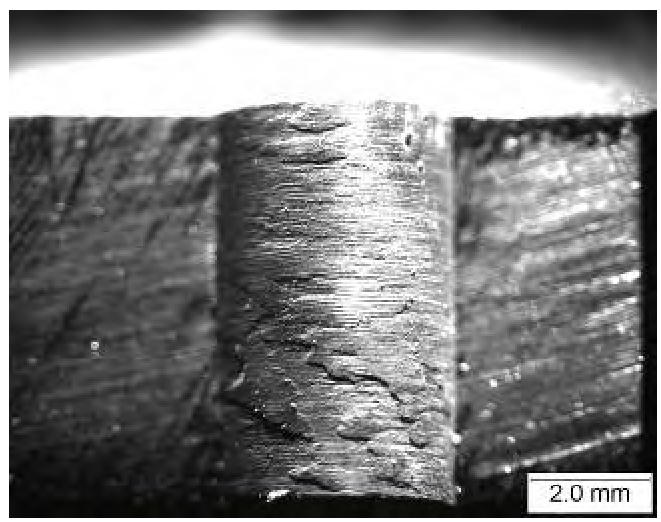


Fig. 2-6. Low-magnification view of the interior of a test hole in a Ti-6Al-4V alloy plate drilled without coolant. Deformation of material along the hole wall is evident.

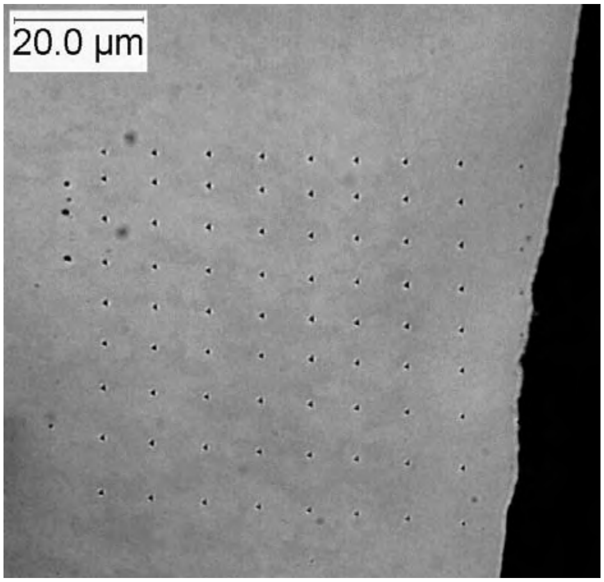


Fig. 2-7. Array of nanoindentations on the drill-hole wall in a Ti-6Al-4V workpiece obtained at a cutting speed 183 m/min and 0.051 mm/rev feed without supply of cutting fluid.

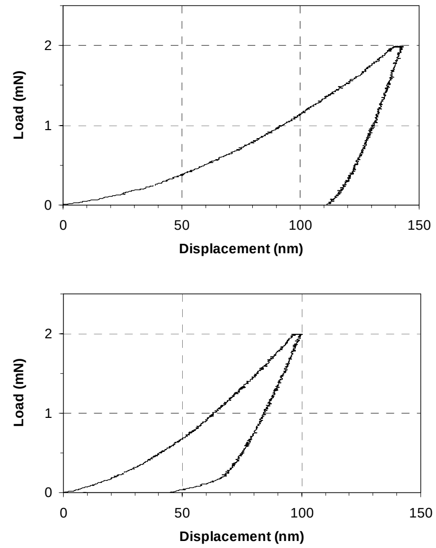


Fig. 2-8. Nanoindentation load vs displacement curves obtained on a drill-hole wall (upper) and on a region away from the drill hole (lower).

3. MECHANICAL CHARACTERIZATION AND ANALYSIS USER CENTER (MCAUC)

User Center Members

Edgar Lara-Curzio, Leader
Christine Goudy, Group Secretary
Donald L. Erdman III
James G. Hemrick
Michael J. Lance
Miladin Radovic
Laura Riester
Amit Shyam
Christopher O. Stevens
Rosa M. Trejo

The MCAUC specializes in the mechanical characterization of functional and structural materials. Numerous mechanical test frames with uniaxial and multiaxial capabilities are available to visiting researchers 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. MCAUC staff have expertise with a wide variety of materials, testing configurations, analytical modeling, failure analysis, finite-element stress analysis, and life-prediction analysis of materials and structures.

MCAUC Instruments

- Electromechanical, servohydraulic, pneumatic, and dead-weight testing machines for tension, compression, torsion, flexure, axial/torsion, and other loading configurations with capabilities up to 500 kN and 3000 Hz
- High-temperature furnaces (resistance and induction heating, quartz lamps) with capabilities up to 1700°C in air and 2000°C+ in vacuum or inert environments
- Integral electronic controllers for load, displacement, and strain control and computerized data acquisition
- Fixtures for uniaxial (3-point and 4-point) and biaxial (ring-on-ring) bending, Iosipescu shear testing, antibuckling compression, interlaminar shear by compression of double-notched specimens, and fracture toughness by double torsion
- Experimental facilities for creep, stress rupture, and stress relaxation testing at ambient and high temperatures, in air, vacuum, or controlled environments
- Rotary bend fatigue machine for testing cylindrical specimens in fully reversed cyclic loading
- Microturbine test facility for assessing the effects of stress and temperature on the durability of materials in combustion environments.
- Acoustic emission detectors, resonant ultrasound spectrometer, and impulse excitation instrumentation for characterizing mechanical integrity and elastic properties of materials and components
- 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 evaluating composite interfacial properties by means of single-fiber push-in and push-out
- Dynamic and thermomechanical analyzers
- Raman microprobe

Selected Highlights

Effect of Temperature and Loading Rate on the Tensile Behavior of TRIP Steels

Laboratory Collaborators: Elizabeth Stephens and Xin Sun
HTML Staff: Don Erdman, Mike Starbuck, David Pohlit, and Edgar Lara-Curzio

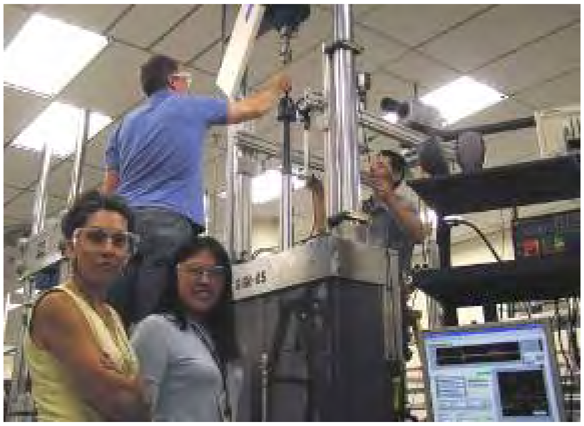


Fig. 3-1. Pacific Northwest National Laboratory researchers Elizabeth Stephens and Xin Sun. David Pohlit and Mike Starbuck are in the back preparing a test in ORNL's high-rate testing machine.

Considerable efforts have been dedicated in recent years to develop transformation-induced plasticity (TRIP) steels, which combine the high strength and good formability required for applications in the automotive industry. The high ductility of TRIP steels results from the transformation of metastable retained austenite to martensite under plastic deformation. This transformation is accompanied by a volume expansion and results in a localized increase in strain hardening during deformation, which delays the onset of necking and ultimately leads to uniform, larger elongations.

Researchers Xin Sun and Elizabeth Stephens (Fig. 3-1) from Pacific Northwest National Laboratory visited ORNL to participate in a user project in collaboration with HTML researchers Don Erdman, Mike Starbuck, David Pohlit, and Edgar Lara-Curzio. The project objective was to determine the effect of test temperature and loading rate on the tensile behavior of TRIP steels. Tensile tests were performed at temperatures between –40°C and 93°C using a unique mechanical testing machine equipped with an actuator capable of 18.5 m/s over a 400-mm range. Each test was recorded by a high-speed video system to determine the deformation of the specimen during the test (Fig. 3-2).

Currently, the test specimens are being characterized to determine the retained austenite in the microstructure and to further understand the deformation mechanisms in TRIP materials. The success of these efforts will lead to improved manufacturing processes for automobile components.

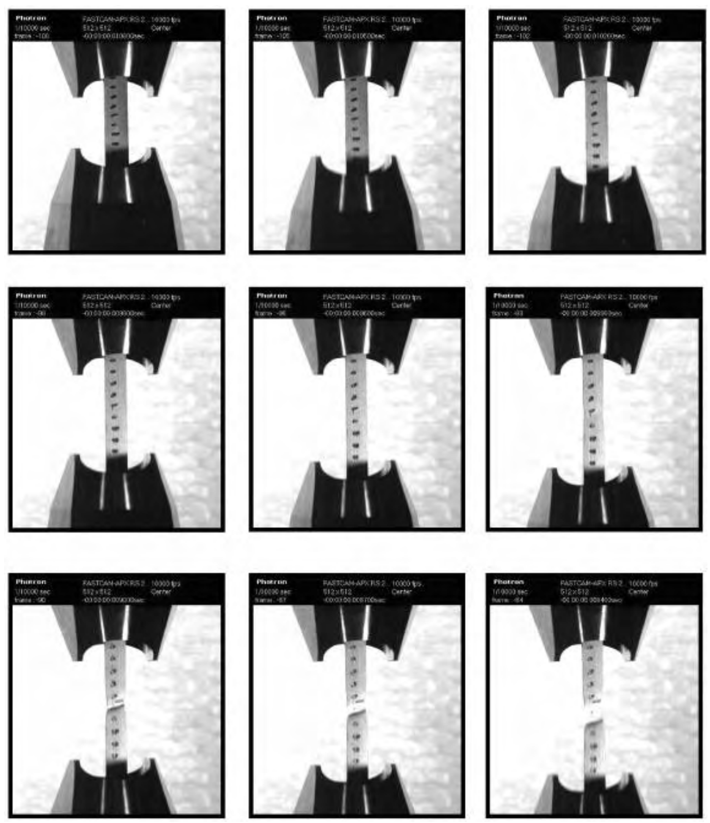


Fig. 3-2. Frames obtained using ORNL's high-speed video during the tensile evaluation of a TRIP steel test specimen at a crosshead displacement of 13 m/s. The time between frames is 0.0003 s. The surface markings on the test specimen are used to determine its strain.

Fatigue Behavior of Aluminum Squeeze Castings

Industry Collaborator: Gerald Gegel
HTML Staff: Don Erdman and Edgar Lara-Curzio

Squeeze casting is the solidification of liquid metal under pressure in a closed metal die. The resulting casting has improved properties and a uniform microstructure compared with those produced by traditional molten-metal fabrication techniques. Under subcontract to Eck Industries, Gerald Gegel of Material & Process Consultancy visited the HTML to initiate a user project in collaboration with researchers Don Erdman and Edgar Lara-Curzio. The goal is to compare the fatigue resistance of aluminum rods produced by an advanced squeeze-casting process to that of rods produced by a conventional squeeze-casting process. The success of this project could lead to more durable components for diesel engine air compressors.

Properties and Microstructure of Silicon Nitride

Laboratory Collaborators: Martin Pech-Canul and Ana Lilia Leal
HTML Staff: Edgar Lara-Curzio, Rosa Trejo, Larry Walker, Tom Watkins, and Robbie Peascoe-Meisner

The focus of the research work of Professor Martin Pech-Canul of Mexico's Center for Research and Advanced Studies (CINVESTAV) is to develop chemical vapor deposition processes in which solid silicon precursors (e.g., Na_2SiF_6) are used to synthesize silicon nitride. The appeal of this approach is the possibility of synthesizing silicon nitride at temperatures below 1000°C, much lower than the temperatures required with conventional processes.

Professor Pech-Canul and graduate student Ana Lilia Leal-Cruz (Fig. 3-3) visited the HTML to collaborate with ORNL researchers Edgar Lara-Curzio, Rosa Trejo, Larry Walker, Tom Watkins, and Robbie Peascoe-Meisner to characterize the microstructure and mechanical properties of silicon nitride materials synthesized over a wide range of experimental conditions (temperatures, gas flow, and pressure) (Fig. 3-4). Preliminary results from the investigations are encouraging and could lead to more cost-effective processes to produce components (e.g., ball bearings, exhaust valves) that could contribute to improving the efficiency of internal combustion engines.

Fig. 3-3. CINVESTAV's Professor Martin Pech-Canul and graduate student Ana Lilia Leal.

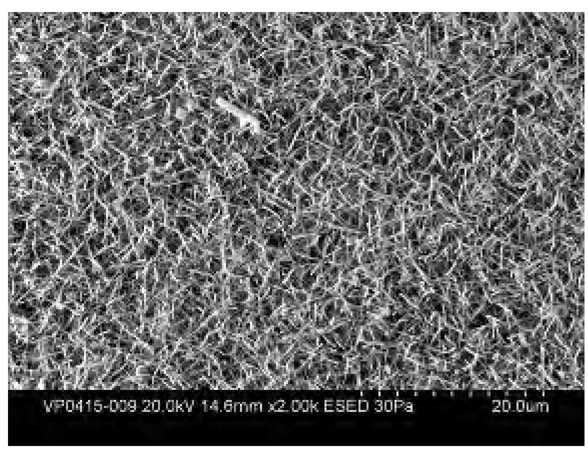


Fig. 3-4. Silicon nitride fibers obtained by the chemical decomposition of Na_2SiF_6.

Properties of Braided Ceramic Matrix Composites

Industry Collaborator: Todd Engel
HTML Staff: Edgar Lara-Curzio, James G. Hemrick, and Miladin Radovic

Fig. 3-5. Hyper-Therm engineer Todd Engel preparing to perform a tensile test.

Hyper-Therm High-Temperature Composites, Inc., is a small business located in Huntington Beach, California. Since 1992, Hyper-Therm has been active in the design and manufacture of C/SiC and SiC/SiC continuous fiber-reinforced ceramic matrix composites produced via isothermal/isobaric and forced-flow chemical vapor infiltration. SiC/SiC ceramic matrix composites are being considered for fabricating control rods for high-temperature gas-cooled nuclear reactors that produce clean electric power and hydrogen.

Braided fiber architectures, such as those needed for control-rod components, have been identified as a cost-efficient means of producing axisymmetric structures. However, there are no data on the thermal or mechanical properties of the latest generation of radiation-resistant SiC/SiC composites with such fiber architectures. Hyper-Therm engineer Todd Engel (Fig. 3-5) visited the HTML to work with MCAUC researchers Edgar Lara-Curzio, James G. Hemrick, and Miladin Radovic to determine the elastic constants, thermal expansion, and tensile behavior of SiC/SiC composites with braided fiber architectures. They used resonant ultrasound spectroscopy to determine elastic properties; a thermomechanical analyzer to determine thermal expansion; an electromechanical testing machine equipped with a high-temperature furnace, low-contact-force extensometry, and ORNL-designed grips for the tensile tests. The data generated will be used to produce preliminary designs for control rods and to help select the optimized fiber architecture. These data will also be useful for designing other high-temperature components.

Compressive Behavior of Nanocrystalline Fe-Ni Alloys

University Collaborators: Peter Liaw and Guojiang Fan
HTML Staff: Laura Riester and Rosa Trejo

Studies on conventional coarse-grain materials have revealed a strong size effect on their mechanical behavior. Nanocrystalline alloys are the focus of active research because of their superior mechanical properties, but to date, no size-effect studies exist for those materials. University of Tennessee, Knoxville (UT) Professor Peter Liaw and postdoctoral researcher Guojiang Fan (Fig. 3-6 next page) visited the HTML to work with MCAUC's Laura Riester and Rosa Trejo to evaluate how test specimen size of nanocrystalline Ni-Fe alloys (average grain size: 23 nm) affects the properties and mechanical behavior of those specimens. They used HTML's nanoindenter with a flat punch to subject cylinders of various diameters (ranging between 0.3 and 10 μm) to direct axial compression (Fig. 3-7 next page). The cylinders were prepared by FIB micromachining. Preliminary results revealed an unusual mode of failure of the cylindrical test specimens, which was associated with the procedure that had been used to prepare them. The success of the project could lead to the development of nanostructured miniaturized components with improved mechanical properties.

Fig. 3-6. UT postdoctoral researcher Guojiang Fan.

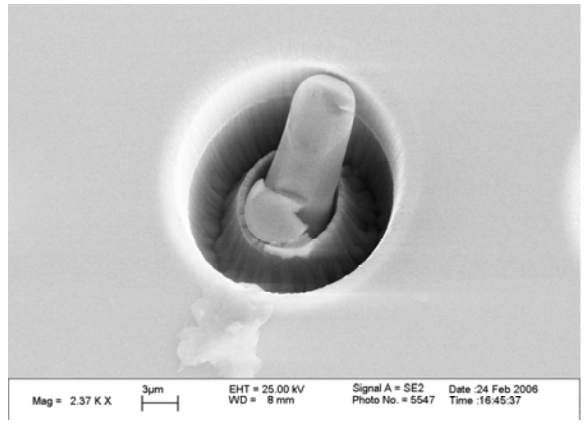


Fig. 3-7. Scanning electron micrograph of Ni-Fe nanocrystalline cylinder after compression testing using HTML's nanoindenter.

Reliability of Zirconia Electrolytes for Solid-Oxide Fuel Cells

CORNING

Industry Collaborators: Sujanto Widjaja and Scott Pollard
HTML Staff: Edgar Lara-Curzio, Rosa Trejo, and Miladin Radovic

Fuel cells are electrochemical devices that convert the chemical energy in a fuel into electric energy. Among fuel cells, solid-oxide fuel cells (SOFCs) are particularly attractive because they can achieve high efficiency and can operate with multiple fuels. In 2001, Corning, Inc., initiated an R&D program to overcome the challenges associated with the high-temperature operation of SOFCs, which currently limit their durability and reliability. Corning's approach to improving the robustness of SOFCs is based on the use of an electrolyte-supported planar SOFC. At the heart of the design is a flexible 20-µm-thick 3YSZ membrane. Corning engineers Sujanto Widjaja and Scott Pollard (Fig. 3-8) visited the HTML to work with MCAUC researchers Edgar Lara-Curzio, Rosa Trejo, and Miladin Radovic to determine the thermomechanical performance of thin 3YSZ membranes and the effect of temperature gradients on their fracture behavior (Fig. 3-9). The information obtained in this project will help Corning engineers design durable and reliable SOFCs.

Fig. 3-8. Corning engineer Scott C. Pollard preparing a tensile test.

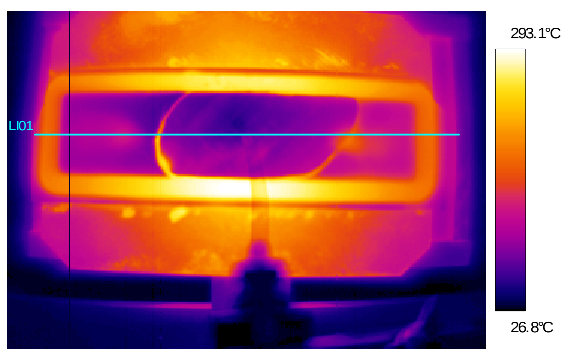


Fig. 3-9. Temperature distribution over a 20–µm-thick 3YSZ membrane right after failure as captured by MCAUC's infrared camera. The constrained membrane had been heated uniformly using an infrared lamp and cooled in the middle with a stream of air to generate a temperature gradient over the membrane.

Properties of Materials for Micromolding Replication Technologies

University Collaborators: Wen Meng and Jing Jiang
HTML Staff: Edgar Lara-Curzio and Chris O. Stevens

The commercialization of microscale devices and systems, such as microscale chemical reactors, heat exchangers, and electromagnetic relays, requires the availability of fabrication technologies that can achieve low-cost mass production of metallic high-aspect-ratio microscale structures (HARMS). Aluminum-based HARMS, by virtue of a favorable combination of thermal conductivity and density, are particularly important for heat transfer applications. Many metallic-based HARMS microdevices have significant relevance to transportation technologies. For example, high-efficiency, miniaturized heat exchangers and microchemical reactors are relevant for fuel-cell-powered electric and hybrid vehicles.


Fig. 3-10. MCAUC's Chris Stevens and LSU graduate student Jing Jiang.

Professor Wen Meng and graduate student Jing Jiang (Fig. 3-10), of the Louisiana State University Mechanical Engineering Department, are developing and implementing micromechanics-based models to improve micromolding replication technologies. They have focused their work on the LiGA approach, which combines deep lithography (*lithographie*), electrodeposition (*galvanoformung*), and molding replication (*abformung*). This approach represents an important strategy for low-cost mass production of metallic-based HARMS. Professor Meng and Mr. Jiang visited the HTML to work with MCUAC researchers Edgar Lara-Curzio and Chris Stevens to determine the mechanical properties of aluminum, copper, and nickel alloys, which are used to fabricate mold inserts. Of particular interest were the tensile properties of the materials at high temperature and as a function of strain rate. These data, which are unavailable in the open literature, have allowed professor Meng and his team to provide realistic input data for validating their micromechanical models. Their research will lead to the design, development, and manufacture of reliable low-cost components for transportation and other technologies.

Thermal Resistance between Internal-Combustion Engine Components

Industry Collaborator: Scott Sochor
HTML Staff: Hsin Wang and Miladin Radovic

Selecting materials for engine components requires detailed knowledge of the temperatures these components will experience during service. This is particularly important for mating components. In addition to the material thermophysical properties, the thermal resistance between mating components (e.g., valve seats and cylinder liners in internal combustion engines) must be known. General Motors (GM) Powertrain Division engineer Scott Sochor conducted a successful study of thermal contact resistance of engine components at HTML (Fig. 3-11). He worked with HTML researchers Hsin Wang and Miladin Radovic to determine the temperature distribution along mating components when subjected to compressive loading up to 150 MPa at temperatures between 20°C and 200°C (Fig. 3-12).

Fig. 3-11. Scott Sochor, GM Powertrain Division Engineer.

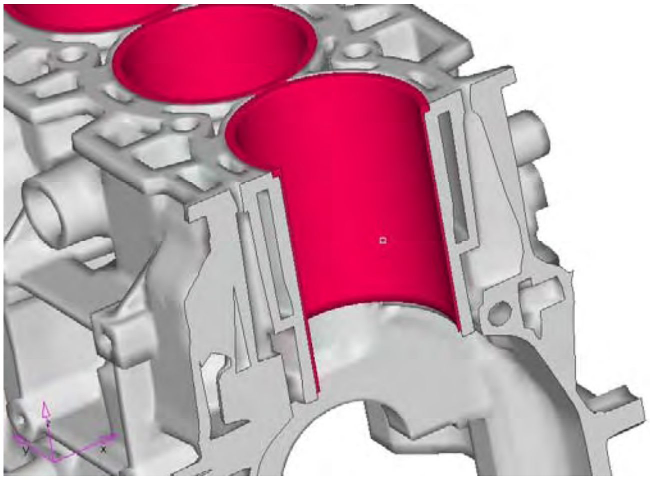


Fig. 3-12. GM Powertrain Division researchers studied temperature distribution along mating components of internal-combustion engines when subjected to compressive loading.

Eight groups of material pairs were studied during a series of tests using an electromechanical testing machine, a special furnace with an IR-transparent window, and an IR camera to determine the temperature distribution. Several measurements were obtained for combinations of several materials used to manufacture valve guides, valve seats, cylinder liners, cylinder blocks, and cylinder heads (e.g., Al356-T6, Al319-T7, 21-2N, 21-4N, Brico 3220, and gray iron). The data are being analyzed at GM Powertrain, where the information will be used in engine design to predict temperatures during engine operation. The results of this investigation are expected to lead to the design and manufacture of better engine components.

Properties of Polymer-Derived SiCN Ceramics for High-Temperature Sensors

Industry Collaborators: Bill Garrett and Wenge Zhang
University Collaborator: Linan An
HTML Staff: Rosa Trejo, Miladin Radovic, and Edgar Lara-Curzio

Sporian Microsystems, Inc., a small business in Boulder, Colorado, was formed in 2000 by University of Colorado graduates to develop and commercialize advanced microelectromechanical systems (MEMS), including sensors for environmental monitoring in power generation, water health management, homeland security, telecommunications, aerospace, and military applications. Recently, DOE awarded Sporian a Small Business Innovative Research contract to develop sensors to monitor temperature and pressure up to 1400°C inside power generation systems.

Sporian researchers Bill Garrett and Wenge Zhang partnered with Professor Linan An of the UCF to assess the feasibility of using polymer-derived SiCN ceramics to manufacture high-temperature sensors by evaluating their mechanical and physical properties (Fig. 3-13). Specifically, they worked with HTML researchers Rosa Trejo, Miladin Radovic and Edgar Lara-Curzio to determine the effect of processing parameters on the elastic properties, flexural strength, and thermal shock resistance of polymer-derived SiCN ceramics (Fig. 3-14). Successfully developing these sensors could enable the implementation of sensing and control strategies for automotive and turbine engines, combustors, gasification processes, and furnaces in a wide range of industries.


Fig. 3-13. Sporian's Bill Garrett and University of Central Florida's Professor An prepare a high-temperature flexural test (left); Sporian's Wenge Zhan uses resonant ultrasound spectroscopy (right).

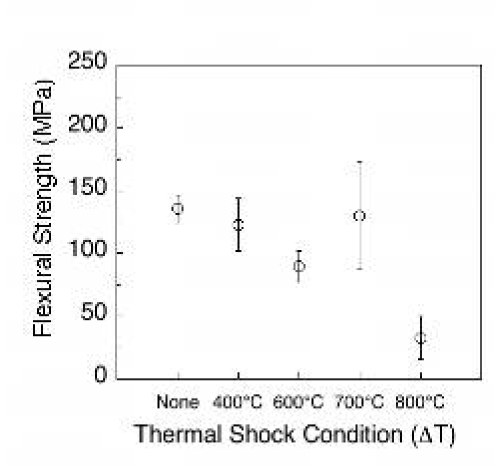


Fig. 3-14. Thermal shock resistance of polymer-derived SiCN ceramic.

Mechanical Properties of Titanium Matrix Composites

University Collaborator: Ratansingham Sooryakumar
HTML Staff: Rosa Trejo, Miladin Radovic, and Edgar Lara-Curzio

Professor Ratansingham Sooryakumar of the Physics Department at the Ohio State University is working with IAP of Dayton, Ohio, to process titanium matrix composites using magnetic compaction techniques. These materials are attractive for a wide range of applications because of their high specific stiffness and strength, corrosion resistance, and retention of properties at elevated temperatures. Professor Sooryakumar visited the HTML on several occasions during FY 2006 to work with MCAUC researchers Rosa Trejo, Miladin Radovic, and Edgar Lara-Curzio to assess the effect of temperature and concentration of TiC particulates on the elastic constants and flexural strength of these materials (Fig. 3-15). He used HTML's resonant ultrasound spectrometer to determine the resonant frequencies of the material, which when analyzed in combination with the density and geometry of the test specimens, provide values of their stiffness. The magnitude of the elastic constants of titanium matrix composites was found to increase with the concentration of TiC particulates as would be expected by the rule of mixtures, and to decrease with temperature (Fig. 3-16). It was also found that the flexural strength decreased with increasing concentration of TiC particulates, which appears to be consistent with features observed on the fracture surfaces, such as the debonding of particulates in the wake of a main crack (Fig. 3-17). Work continues to optimize the processing and properties of these materials.

Fig. 3-15. Professor Sooryakumar (Ohio State University) and MCAUC's Miladin Radovic using resonant ultrasound spectroscopy to determine the elastic properties of TiC-particulate-reinforced titanium matrix composites.

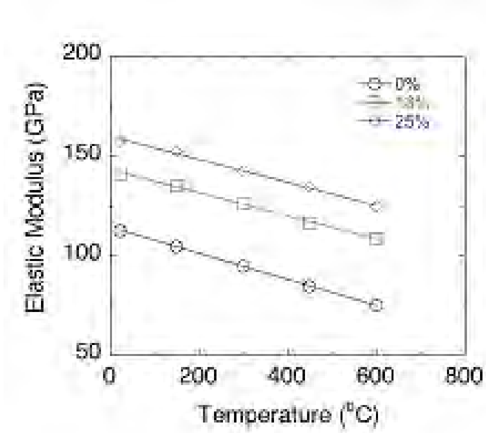


Fig. 3-16. Effect of temperature and concentration of TiC particulates on the elastic properties of titanium-matrix composites.

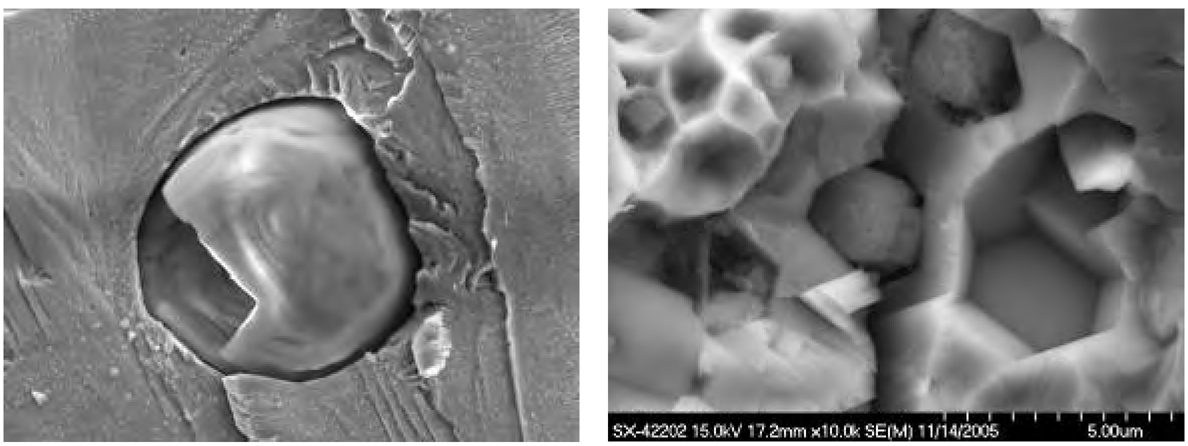


Fig. 3-17. Scanning electron micrographs of fracture surfaces of TiC-particulate reinforced titanium matrix composites.

MCAUC News

Fabio Pavia Graduates Summa cum Laude

On September 26, 2006, Mr. Fabio Pavia graduated summa cum laude after defending his thesis to receive an M.S. degree in Mechanical Engineering from the Polytechnic of Turin, Italy (Fig. 3-18). Mr. Pavia spent the spring and summer of 2006 working on the experimental part of his dissertation at the HTML under the guidance of ORNL researcher Edgar Lara-Curzio. The work of Mr. Pavia was focused on the development of test techniques to determine the biaxial strength of ceramic joints. Mr. Pavia's thesis advisors at the Polytechnic of Turin are Professors Massimiliano Avalle and Monica Ferraris. The development of reliable joining techniques and design guidelines will enable the use of ceramic components in industrial structural applications.

Fig. 3-18. Professor Massimiliano Avalle and Mr. Fabio Pavia.

Radovic Accepts Position at Texas A&M

MCAUC's postdoctoral researcher Miladin Radovic has accepted a tenure-track faculty position in the Department of Mechanical Engineering at Texas A&M (Fig. 3-19). Radovic, who has a Ph.D. in Materials Science and Engineering from Drexel University, joined ORNL in 2001 to work under the guidance of MCAUC's Edgar Lara-Curzio.

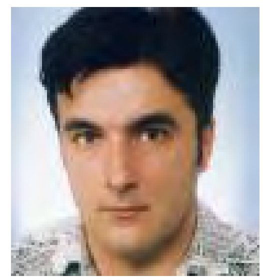


Fig. 3-19. Miladin Radovic.

Lara-Curzio Chairs Thirtieth Cocoa Beach Conference

MCAUC's leader Edgar Lara-Curzio chaired the Thirtieth International Cocoa Beach Conference on Ceramics and Composites in Cocoa Beach, Florida, January 22–27, 2006 (Fig. 3-20). The meeting, which has become the premier international meeting for ceramics and composites, was organized in 7 symposia with more than 600 papers submitted by researchers from 29 countries. Lara-Curzio is also serving as chair-elect of the Engineering Ceramics Division of the American Ceramic Society.

Fig. 3-20. MCAUC's Edgar Lara-Curzio receives a plaque from ACerS President Warren Wolf. (Projected image: the late Jim Mueller.)

4. DIFFRACTION USER CENTER (DUC)

User Center Members

Andrew Payzant, Leader
Geneva Worley, Administrative Secretary
Jianming Bai
Robbie Peascoe-Meisner
Claudia Rawn
Scott Speakman

The DUC uses variable-temperature laboratory, synchrotron X-ray, and neutron diffraction techniques to characterize metals, ceramics, and polymers. Users are scientists and engineers from academic, industrial, and DOE national laboratory backgrounds who collaborate with DUC staff and use the instruments when diffraction data are needed. The diffraction facilities are also extensively used by qualified staff members in the Materials Science and Technology 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 and participate in DOE- and ORNL-funded projects.

The DUC continues to accommodate a large number of user projects of current interest in the field of materials science and engineering. The results are used to relate materials processing and performance with phase transformations, lattice expansion, atomic structure, and phase stability issues that need to be determined before many of the new materials can be used as components with improved properties in new applications. Projects in FY 2006 included studying materials for potential use in fuel cells, multi-pollutant sorbents, photovoltaics, thin films for a variety of applications, ferroelectric liquid crystals, alloys with high melting points that retain strength at high temperatures, and materials used in medical imaging.

DUC Instruments

- Neutron powder diffractometer (low and high temperatures)
- 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 temperatures down to 15 K)
- Synchrotron X-Ray Beamline X-14A (low and high temperatures) at the Brookhaven NSLS.
- New Image Plate Capacity at HTML's Synchrotron Beamline X14A

The X14A beamline X14A at the National Synchrotron Light Source (NSLS) has provided the HTML with a high brilliance tunable x-ray source for both the Diffraction and Residual Stress user centers. In recent years, updated high-temperature attachments have extended the useful range of this instrument, enabling in-situ studies of materials. However, the advantage of high resolution has previously come at the price of comparatively long counting times using the existing point-detector system. This year, in collaboration with Dr. Trevor Tyson's research group at the New Jersey Institute of Technology, a new image plate based detector system has been designed and installed at X14A, which enables data to be collected simultaneously over a wide range of angles in and out of the conventional Bragg-Brentano diffraction plane. This new development cuts data collection times by over an order of magnitude, which is essential for applications looking at time or temperature evolution of materials, as well as providing the capability to study large-grained and/or highly textured materials.

In addition, this new capability greatly expands and enhances the HTML's capabilities to support the mission and objectives of EERE/FCVT with respect to characterization of crystalline materials by high-speed, high-resolution, x-ray diffraction analysis; in particular, with respect to our ability to characterize changes in materials in real-time under simulated operating conditions.

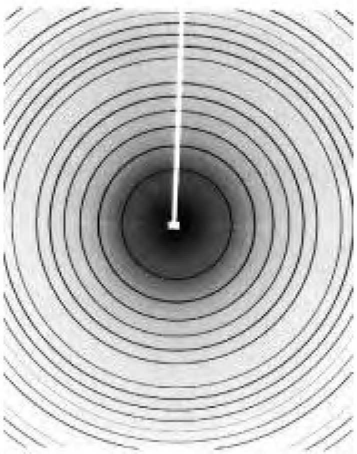


Fig. 4-1. IP measurement on the LaB_6 powder standard.

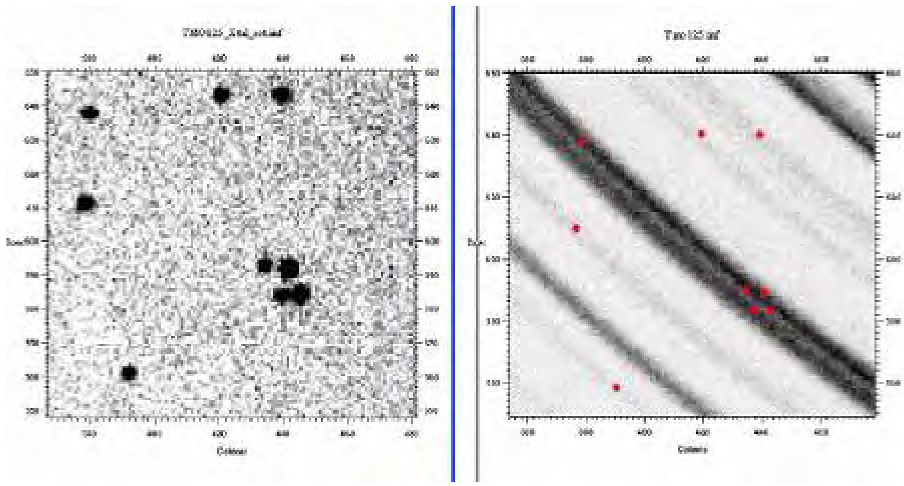


Fig. 4-2. An enlarged view of the Laue diffraction pattern of $TbMn_2O_5$ (left) and the powder pattern of the same structure (right).

Figure 4-1 shows an image plate measurement on a NIST LaB_6 powder standard reference material collected in just 5 minutes at an x-ray energy of 20 keV. At the selected sample-to-image plate distance (205 mm), the angular resolution was about 0.08°.

Figure 4-2 compares image plate measurements on a $TbMn_2O_5$ single crystal (spots) and powder (rings). The correspondence of the single crystal spots and Debye rings is clearly illustrated.

Selected Highlights

Cryomilled Lightweight Metals Characterized by X-Ray Diffraction

University Collaborator: Adam Maisano
HTML Staff: Andrew Payzant

XRD was used at HTML to study nanocrystalline metals produced at Virginia Polytechnic Institute and State University (Virginia Tech) by cryomilling. Crystallite size and strain in the materials are of interest to understand the effect of cryomilling on aluminum and magnesium alloys. Depending on alloy content, these lightweight metals increase in strength 50 to 400 percent, so they are desirable as replacements for denser structural materials. It was determined that cryomilling decreases crystallite size in Al but has little effect on Mg powders. Cryomilling time did not significantly influence crystallite size in Al; however, longer milling times led to a large increase in strain in the material. These results were incorporated into the Ph.D. thesis of Adam Maisano at Virginia Tech.

Oxidation Behavior of Nickel Aluminides Examined in situ

ORNL Collaborator: Bruce Pint
HTML Staff: Claudia Rawn and Scott Speakman

The January 2006 issue of *Journal of Microscopy* features a research paper written by ORNL corrosion expert Bruce Pint, describing recent in situ high-temperature XRD studies on advanced intermetallic alloys. These experiments were done in collaboration with HTML staff members Claudia Rawn and Scott Speakman.

High-Temperature X-Ray Reflectivity of AlN Films on 6H-SiC

University Collaborator: Daniele Stodilka
HTML Staff: Scott Speakman

UNIVERSITY OF FLORIDA

Dielectrics composed of AlN thin films on SiC substrates may be used as power invertors for electric vehicles or for the electric-drive component of hybrid vehicles. However, failure modes due to temperature must be understood to improve reliability in the service environment. Daniele Stodilka from the University of Florida worked with the HTML's Scott Speakman using high-temperature X-ray reflectivity to examine AlN films grown on SiC substrates at the HTML. This marks the first time that high-temperature X-ray reflectivity data have been collected at the HTML. As opposed to the diffraction peaks often used to characterize phase composition or residual stresses, X-ray reflectivity measures interference fringes to evaluate the thickness, surface roughness, and integrity of the thin films. The AlN film maintained integrity up to 700°C, indicating that their failure at lower temperatures may be due to electric breakdown or other mechanisms.

General Motors Studies Thermoelectric Materials with X-Ray Diffraction

HTML Staff: Claudia Rawn

Automobile manufactures are interested in the use of thermoelectric materials to convert waste engine heat into useful electricity. Compounds that contain an atom within a framework, such as the semiconductor clathrate compounds and the filled skutterudites, are of interest for their potential as thermoelectric materials. Previous work with single-crystal neutron diffraction has linked the atomic displacement parameter, or the thermal vibrations of the atom, to the lattice thermal conductivity of various compositions that crystallize with the clathrate and skutterudite structures.

More recent studies have indicated that "misch-metal" skutterudites have performance comparable to the rare-earth-doped material with considerable cost savings, the cost of misch-metal being about one-tenth that of pure rare earths. Researchers from General Motors are collaborating with HTML staff to investigate these materials. XRD powder data show that the GM samples are nearly single phase and that the diffraction maxima become broader as the misch-metal content and Fe content decrease (and as the Co content increases) in the samples (Fig. 4-3). Additional studies using neutron diffraction on samples at high temperature are in preparation.

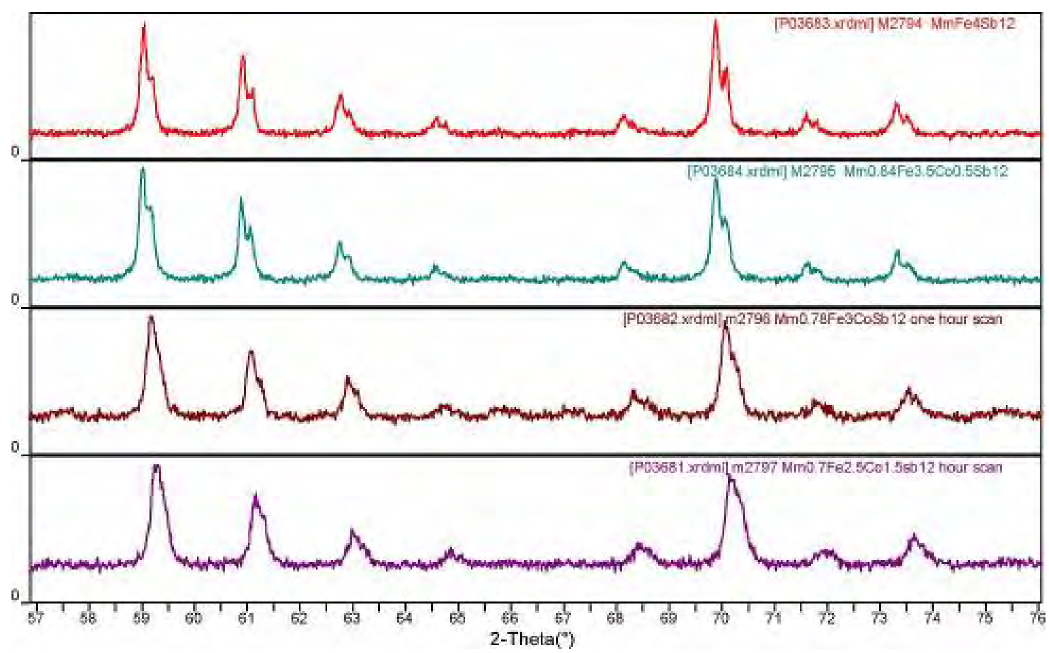


Fig. 4-3. Changes in diffraction pattern of thermoelectric compounds as the ratio of misch metal, iron, and cobalt is varied.

Neutron Diffraction Studies on Biosynthesized Magnetic Nanoparticles

ORNL Collaborators: Tommy Phelps, Lucas Yeary, Ji-Won Moon
HTML Staff: Claudia Rawn

Biologically induced mineralization of magnetite occurs by certain bacteria, creating the necessary chemical environment for magnetite precipitation on the surface of their outer membranes. Biologically induced mineralization is interesting because of the possibility of synthesizing large quantities of ferrite nanoparticles in an environmentally benign way. Magnetic nanoparticles are needed for rapidly expanding applications in the biomedical and engineering fields. The biomineralization process used for this study is of interest due to the synthesis at relatively low temperatures that produces nanoparticles without milling. These factors reduce processing costs and the method is promising for its scale up ability to produce large quantities of material. Additional cations substituting for the Fe can change the physical properties, thus tailoring the properties to specific applications (e.g., ferrofluids that need to act as a switch at a specified temperature). ORNL researchers, including HTML's Claudia Rawn, have focused on the inclusion of Zn2+ in the Fe_3O_4 structure, using time-of-flight neutron powder diffraction data collected at the ISIS neutron source (at the Rutherford Appleton Laboratory, England) to determine the location of the Zn in the magnetite structure. These data allowed both the magnetic and atomic structures to be refined and indicated that the Zn is replacing some of the Fe in the structure. The data also suggest that, as the Zn content increases, the background of the diffraction data increases, suggesting that disorder is occurring. The study results were presented at the American Crystallographic Association Annual Meeting and the American Conference on Neutron Scattering.

Synchrotron X-Ray Diffraction Measurements Help Determine the Structure of Amorphous Alloys

JOHNS HOPKINS UNIVERSITY

University Collaborators: En Ma, H. W. Sheng, W. K. Luo, and F. M. Alamgir
HTML Staff: Jianming Bai

Amorphous alloys, also known as metallic glasses, are materials of great potential because of their unique mechanical and magnetic properties. These properties originate from their disordered arrangement of the constituent atoms, the details of which were largely unknown. Recently, a Johns Hopkins University team lead by Professor En Ma reported results of its two-year investigation in the January 26 issue of the prestigious journal *Nature* (H. W. Sheng, W. K. Luo, F. M. Alamgir, J. M. Bai & E. Ma, "Atomic packing and short- to medium-range order in metallic glasses," *Nature*, **439**, 26 January 2006). In this work, the 3D atomic configuration of the amorphous alloys was reconstructed using the "reverse Monte Carlo" method from simultaneous fitting to both the extended X-ray absorption fine structure (EXAFS) and XRD measurements. The EXAFS data provide species-specific local coordination information; the XRD data provide both short- and long-range order. The XRD measurements were conducted at HTML's synchrotron X-ray beamline, X14A, under the guidance of beamline scientist Dr. Jianming Bai. Due to the weak scattering of the amorphous sample and the requirement for the data of high momentum transfer (so that they can be compared with EXAFS data in the same reciprocal space range), the high X-ray beam flux provided by the beamline was critical to completing the measurements.

Rensselaer Polytechnic Institute Researcher Uses XRD to Study High-Temperature Evolution of "Nanorod" Films

University Collaborator: Tansel Karabacak
HTML Staff: Robbie Peascoe-Meisner, Andrew Payzant

Rensselaer

Nanorod arrays, which could be useful in advancing optical communications and biological sensors, were produced by Dr. Tansel Karabacak at Rensselaer Polytechnic Institute by sputtering W, Ru, or Cu films on silicon substrates at oblique angles. The resulting metal grains were < 100 nm in diameter, but hundreds of nanometers long. The HTML's unique high-temperature XRD facilities were used to determine the temperature at which significant grain growth was initiated. The grain (crystallite) size could be estimated from measuring the diffraction peak widths, and the lattice thermal expansion could be derived from the changes in peak positions as the samples were heated. In addition, the W films transformed from the as-deposited (but metastable) face-centered cubic (fcc) β-phase to the body-centered cubic (bcc) α-phase at around 500°C. An observed anomaly in the thermal expansion of the Ru film is still under investigation.

University of Arkansas Researcher Studies Chromia Films

University Collaborators: Atul Khanna, Deepak Bhat
HTML Staff: Andrew Payzant

Dr. Atul Khanna used the HTML's high-temperature XRD capabilities to study the crystallization and grain growth of Cr_2O_3 thin films prepared by reactive AC magnetron sputtering on silicon and glass substrates. Such hard films have potential application as wear-resistant coatings in magnetic recording media and have been shown to aid the low-temperature deposition of corundum coatings.

DUC News

DUC Scientist Honored by University of Tennessee

Dr. Claudia Rawn, a DUC staff member, recently was presented the "Outstanding Young Faculty Researcher Award" for the Materials Science and Engineering Department at UT. Claudia has held a joint faculty appointment at the university for the past three years. Claudia Rawn serves in leadership roles for the Oak Ridge Chapter of ASM International and chairs the Powder Diffraction Special Interest Group of the American Crystallographic Association.

Andrew Payzant Elected as Fellow of the International Centre for Diffraction Data

In recognition of years of leadership and service to the ICDD, Dr. Payzant was elected as Fellow of the ICDD at the March Annual Meeting. The ICDD Board of Directors approved the designation of Fellow of the ICDD to Dr. Payzant, who was noted for giving time and talents well beyond that normally associated with membership toward improving the powder diffraction field and characterization of materials by XRD methods.

Dr. Payzant continues to serve on the editorial committee of *Advanced Materials and Processes* magazine; he organized and chaired sessions at the Denver X-Ray Conference.

HTML User Honored by Sigma Xi

Dr. Federico Guazzone received a 2006 Sigma Xi Outstanding Ph.D. Dissertation Award at Worcester Polytechnic Institute. His thesis, "Engineering of Substrate Surface for the Synthesis of Ultra-Thin Composite Pd and Pd-Cu Membranes for H_2 Separation," was supervised by Dr. Yi-Hua (Ed) Ma, and included in-situ XRD characterization at the HTML. Part of his thesis work has been published in *Industrial and Engineering Chemistry Research*.

HTML Hosts Tour for Materials Camp 2006

During the week of June 5, the Oak Ridge Chapter of ASM International sponsored a Materials Camp for local high school students. Twenty-three students took part in activities geared toward introducing them to the field of materials science and engineering. During the week, the students, along with two NASA materials engineers, conducted a failure analysis investigation of samples of recovered materials from the Space Shuttle Columbia. This year's camp was the third sponsored by the Oak Ridge Chapter and was co-organized by Claudia Rawn through her UT affiliation.

Speakman Takes Position at MIT

Dr. Scott Speakman has accepted a new position as research specialist for XRD at the Center for Materials Science and Engineering at MIT. A postdoctoral research associate at the DUC since the summer of 2003, Scott was the HTML point of contact on many user projects for the past three years.

5. RESIDUAL STRESS USER CENTER (RSUC)

User Center Members

Camden R. Hubbard, Leader
Geneva N. Worley, Administrative Secretary
Ke An
Jianming Bai
William Bailey
O. Burl Cavin
Hahn Choo
E. Andrew Payzant
Fei Tang
Thomas R. Watkins

HTML user projects and industry-related programs are increasingly concerned with life prediction and failure analysis of engineering structures and improving life via beneficial compressive stresses near the surface. In many cases, knowledge of engineered 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 for developing life prediction models.

The RSUC was established to meet this need by providing a facility for taking measurements and assessing methods for controlling residual stresses through modifications in the forming, surface treating, and finishing processes; changes in the design; or application of stress-relief procedures. Increasingly, HTML residual stress measurements are being used to check computational models, guide their improvement, and validate the best models.

RSUC users also characterize nonrandom grain distribution, known as texture, in materials and relate the results to directionally dependent materials properties using the same experimental facilities. Texture is very common in materials subjected to deformation as well as in thin films and coatings, which are materials of increasing technological importance. These diffraction facilities are not only used to measure both macro (long-range) and micro (short-range) residual stress and texture at the surface of and within polycrystalline materials, but also to provide fundamental knowledge of intergranular stresses arising during plastic deformation.

RSUC provides three principal stress measurement capabilities at three locations: the HTML X-ray residual stress facilities, the HTML-managed synchrotron beamline X14A at the National Synchrotron Light Source (NSLS), and the Neutron Residual Stress Mapping Facility (NRSF2) at HFIR. Together, the three facilities, unique in themselves, make RSUC an unparalleled resource, able to address a wide range of challenging stress and texture characterization needs for industry, academia, and laboratory.

RSUC Instruments

- Powder-texture-stress (PTS) four-axis goniometer with 18-kW X-ray rotating anode
- PTS four-axis goniometer with 2-kW X-ray tubes
- PANalytical X’Pert Pro X-ray diffractometer with high-temperature stages
- Large-specimen X-ray stress analyzer with automated mapping
- X14A synchrotron high-flux, highly parallel X-ray beamline
- NRSF2, a second-generation neutron residual stress mapping facility

The HFIR is one of the two highest-flux steady-state sources of neutrons in the world. The developmental operation of NRSF2 is leveraged on the operating, maintenance, and HFIR upgrade programs of DOE's Office of Basic Energy Sciences (BES). Similarly, the activities at the NSLS X14A beamline benefit greatly from the BES sponsorship of the NSLS. The RSUC also gratefully acknowledges the consignment of a high-temperature XRD system by PANalytical (formerly Phillips Analytical) and Anton Paar.

Instrument Development

Spallation Neutron Source-VULCAN Progress

The VULCAN diffractometer, a large-scale engineering instrument used to analyze structural materials, employs a neutron beam aimed at a material sample. By studying how the neutrons scatter or bounce off a given material, scientists can see how its atoms are arranged. Groundbreaking ceremonies for the VULCAN building at SNS were held June 23, 2006 (Fig. 5-1). Present were John Haines, newly appointed director for SNS-HFIR's Technology Development Division; Dr. Thom Mason, Associate Laboratory Director for SNS; Dr. John Root, Director of the Canadian Neutron Beam Centre, Chalk River, Canada, and representative of the Canadian Foundation for Innovation (VULCAN's major funding source); Dr. Xun-Li Wang, SNS, VULCAN's Principle Scientist; Dr. Ian Anderson, Division Chief, SNS Experimental Facilities. Representing the other contributors VULCAN's construction and instrumentation funding were Ken Herwig (SNS, Group Leader, Experimental Facilities Division), Camden Hubbard (RSUC Leader, HTML), and Peter Liaw (UT).

Fig. 5-1. VULCAN external building groundbreaking, June 23, 2006. Left to right: John Haines, Dr. Thom Mason, Dr. John Root, Dr. Xun-Li Wang, and Dr. Ian Anderson.

Significant progress has been made on three of the VULCAN subsystems that involved HTML staff and resource contributions.

1. The draft specifications for the load frame and furnace accessories were reviewed with potential vendors and with UT, HTML, and Spallation Neutron Source (SNS) team members. The National Science Foundation-sponsored UT-ORNL team drafted the final specifications, and UT initiated procurement.
2. HTML team members, in collaboration with the VULCAN instrument scientist, helped develop specifications for the sample positioning system. ORNL-SNS initiated the procurement of this subsystem after review comments were addressed.
3. The off-line/on-line sample alignment systems requirements have been defined and the best system has been identified. Under HTML staff leadership, procurement was initiated in June 2006, and laser trackers were delivered in August. (See the following discussion for more detail.)

Off-Line/On-Line Alignment System Designed; Capital Equipment Components Procured

Supported by the EERE Office of FreedomCAR and Vehicle Technologies (FCVT) in partnership with SNS and VULCAN, the design for VULCAN's off-line/on-line sample alignment system was finalized and procurement and training were completed. The design concept is to perform detailed sample alignment and define measurement location off-line using laser trackers, define a few fiduciary locations, and then use the fiduciary locations to relocate the sample on VULCAN, NRSF2, or other stress-measurement instruments. Once completed, productive neutron strain measurements can start essentially immediately, virtually eliminating the need for neutrons for sample set-up, and, equally important, reducing chances for errors. From past experience at NRSF2, approximately 25% of the total available neutron time for a study will be saved. Compared to theodolites and neutron diffraction alignment, the off-line laser-tracker alignment method also significantly improves the accuracy of measurement locations, for example, as needed in studies of quenched parts, welds, and buried interfaces.

NRSF2 Commissioning and User Projects Begun

The HFIR beam room and the HB-2 tunnel were completed, and installation of the NRSF2 was begun in FY06 with plans to complete in FY07. The HB-2 tunnel holds the NRSF2 double-crystal focusing monochromator system built for ORNL by the University of Missouri. Other parts of the NRSF2 system included the instrument beam shutter, beam stop, sample position system, detectors, LabView data collection, and data processing software. Accessories completed in FY06 include the load frame for in situ deformation studies. Adapting components from the earlier system allowed us to simultaneously conduct user projects while waiting for the fabrication of components that were optimized for the new system. During the fiscal year, several studies were completed including:

- measuring stresses in cylinder lines with Third Wave Systems,
- measuring stresses under applied load in Al-SiC MMC with Arizona State University,
- determining surface and through-thickness stresses in cruciform welds with Caterpillar,
- demonstrating the potential of neutron scattering to understand the role of applied loads on Corning soot filter materials,
- determining the surface and through-thickness stresses with IQ Technologies in support of developing validated models,
- strain mapping around fatigue cracks in 316SS before and after an overload event (with UT), and
- assessing the potential of neutron scattering mapping ZrH^2 content in Zircaloy-4 rods that were subject to hydrogen charging (also with UT).

The NRSF2 detector array was designed, initial components were received, and the seven-position sensitive detectors were mounted. Tests were conducted to determine the optimum level of fast neutron and gamma ray shielding required. Optimized incident and diffracted beam collimation and slit holder systems will be designed after the detector shielding is completed in the coming year, along with upgrades to the instrument calibration software.

Selected Highlights

The RSUC continues to address critical industrial and academic problems, typically using a combination of RSUC and other HTML facilities. The following highlights were selected to display the scope of RSUC activities and the increased use of a combination of RSUC instruments to accomplish the RSUC goals. These goals include a comprehensive measurement or mapping of stress to enhance life prediction, understand failure, guide improvement in manufacturing processes, validate models, and address fundamental science such as materials deformation mechanisms.

HTML's New Neutron Residual Stress Mapping Facility Highlighted

More than 200 scientists, engineers, and students attended the SNS-HFIR User Group Meeting held at ORNL Oct 10–12, 2006. One of 6 oral sessions and 10 posters highlighted the HTML's 2nd Generation NRSF2. The technical session "Engineering Materials Behavior Studies with Neutrons" included talks on engineering materials and material behavior studies using neutrons. Dr. Camden Hubbard, RSUC-ORNL, summarized the status of commissioning and performance of the HTML's new NRSF2 at the HFIR and the status of construction of VULCAN at SNS. Initial NRSF2 performance results promise that this will be a world-class facility. The highlights and status of design and construction of VULCAN, the engineering instrument at SNS, indicate that VULCAN will be the world's best instrument for studies of materials behavior when it comes on line in summer of 2008. Other presentations highlighted the future role of neutrons for engineering and materials science research. Prof. H. Choo, UT Joint faculty with ORNL, presented "In Situ Neutron Diffraction Studies of Mesoscopic Deformation Behavior of Structural Alloys," which highlighted the rich and unique understanding of materials deformation that can be obtained using neutron and in situ load frames with furnaces. Dr. S. Babu from EWI-Columbus summarized prior studies of materials following and during thermo-mechanical processing via neutron scattering. He closed his presentation with a vision of the future studies that the new facilities at ORNL should enable. Prof. Les Butler of LSU gave the last presentation, addressing the opportunities and unique data that could be obtained if a world-class tomography and radiography facility were to be developed at the SNS. In crystalline phases, scattering from Bragg peaks often is a major neutron transmission contrast mechanism. With neutron energy analysis at a pulse neutron source the "Bragg edges" can be used to enhance the images. This can best be taken advantage of at the SNS. Later in the day, results from eight research studies using NRSF2 were presented in posters.

Changes in Lattice Strain Profiles around a Fatigue Crack through the Retardation Period after Overloading

University Collaborators: J. Sun, P. Liaw
HTML Staff: Ke An, W. B. Bailey, C. R. Hubbard

A user research project at NRSF2 advanced the understanding of overload phenomena during fatigue. The study's findings were reported at the MECA-SENS 3 International Conference in Santa Fe, New Mexico, October 2005. The project aimed to reveal the mesoscale changes that retard crack growth rate following an overload. Using neutron diffraction, UT users mapped the elastic/plastic strains in front of the crack tip, which can aid in the development of micromechanical modeling. The strain mapping revealed that the overload increases the damage zone and changes the stress distribution compared to that of the steady-state fatigue damage zone (Fig. 5-2). The quantitative results are being used to improve the micromechanic models for the fatigue life of materials.

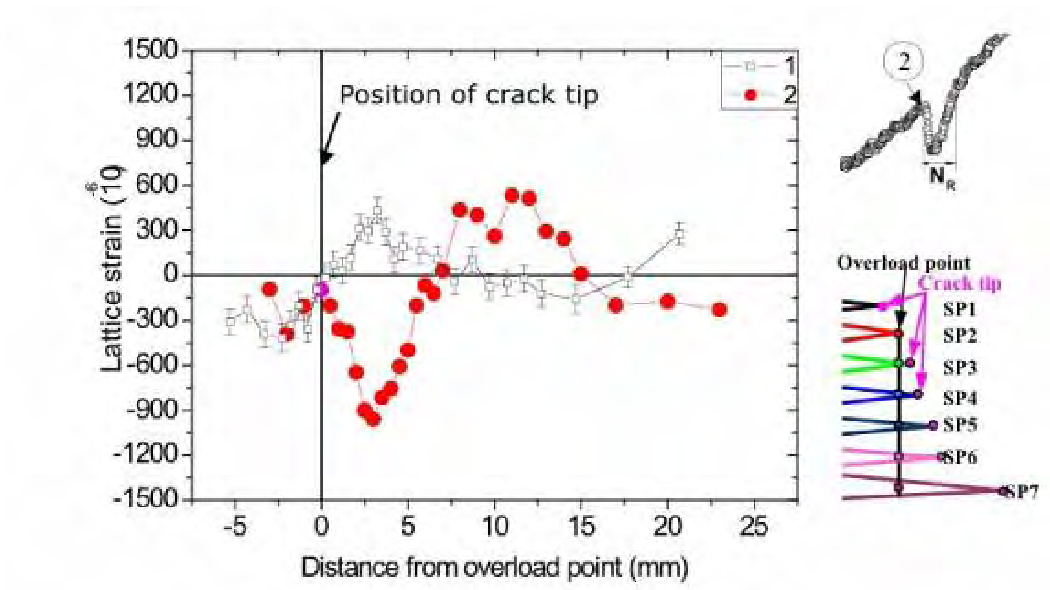


Fig. 5-2. Transverse lattice strain profile for specimen 2, immediately after overload, compared to that of specimen 1, prior to overload.

Residual Stresses in Ni-Cr Superalloys due to Fatigue and Corrosion

University Collaborator: B. R. Barnard
HTML Staff: T. R. Watkins

UT researcher Bryan Bernard visited the HTML to study the development of residual stresses in Ni-Cr superalloys due to fatigue and corrosion. When these superalloys are sufficiently heated, they form protective surface oxides, which generate residual stresses. Generally, superalloy components are subjected to applied stresses and cyclic temperature changes. The focus of the study was on the residual stress distribution (in alloy and oxide) of specimens of two mostly Ni-Cr superalloys subjected to: (1) isothermal, high-temperature conditions under no applied stress; (2) cyclic temperature changes under no applied stress; and (3) isothermal, high-temperature conditions under applied stress. The results indicate that the largest residual stress in the first alloy (74 Ni-20 Cr- bal. other) was produced by condition 2, followed by 3, and then 1. All residual stresses were compressive. Interestingly, the largest compressive residual stress was formed under condition 3, followed by 1 and 2 at comparable values in the second alloy (57 Ni-22 Cr-14 W-bal. other). This suggests that there was some plastic deformation. The greatest residual stress distribution among the oxides of the first alloy was observed to be that under condition 1, then 2, then 3. The oxides that experienced the greatest residual stress distribution for the second alloy were subjected first to condition 3, then 2, and finally 1. This work is part of an ongoing Ph.D. thesis in Material Science that aims to also examine the effect of cyclic temperature changes under applied stress. These alloys are being considered for use in aircraft applications.

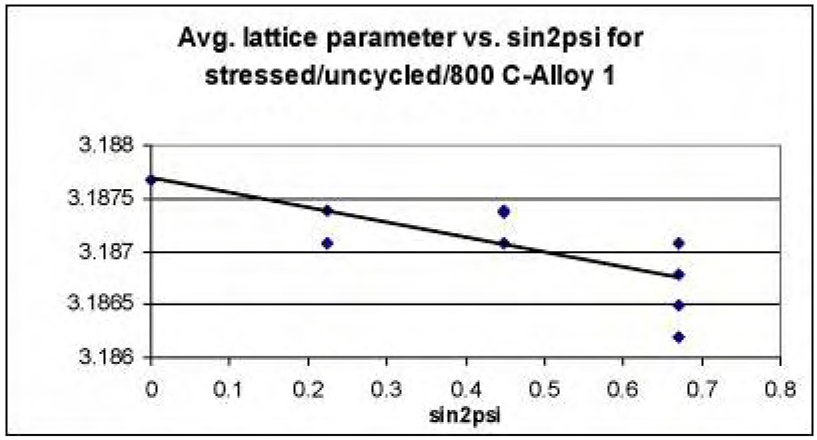


Fig. 5-3. Average lattice parameter as a function of $\sin^2(2\psi)$ for alloy 1 in the stressed/isothermal condition (3). The trend line indicates compressive residual stresses in the alloy.

University of Michigan studies the alpha-to-beta-phase fraction in Ti-6Al-4V

University Collaborators: A. J. Shi, R. Li
HTML Staff: T. R. Watkins, E. A. Payzant

This research effort is an outgrowth of the project "Cost-Effective Machining of Titanium Materials," sponsored by DOE. A drilling study of commercially available alloys is being conducted to analyze the effect of drilling process parameters, such as cutting speed, feed, and fluid on product quality. Alterations of Ti workpiece properties and chip formation, detailed investigations of change of chemical composition, phase transformation, microstructure, and material properties of both workpiece and chips are required. The University of Michigan researchers expect to acquire a better understanding of the machinability and mechanics of the material deformation in drilling of Ti. Another goal of this work was to provide guidelines for selecting machining process parameters for drilling of Ti alloys for industrial and transportation uses.

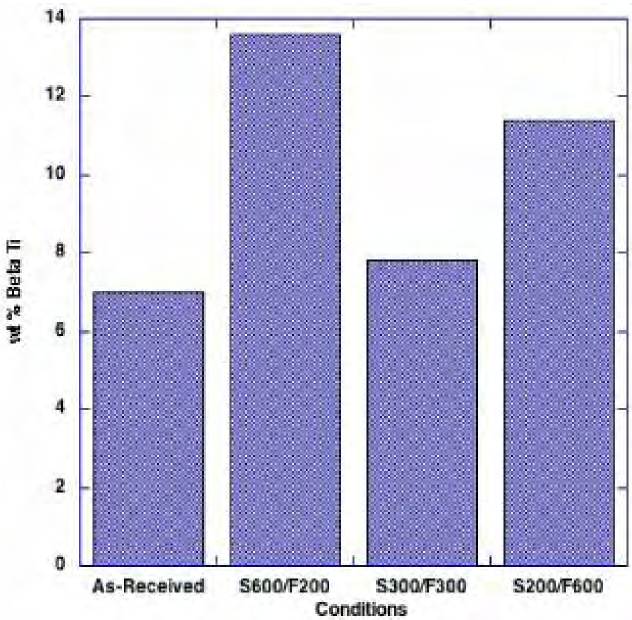


Fig. 5-4. The weight % beta Ti for the various sample conditions considered at constant machining power (S = drilling speed: ft/min.; F = feed rate: in/revolution).

Ti-6Al-4V is a heat-treatable alpha-beta alloy; therefore, the properties can be tailored by controlling the amount of retained high temperature beta phase. Here, the workpiece and chips were examined by XRD using a quantitative Rietveld analysis. Figure 5-4 shows a minimum when increasing the drilling speed and decreasing the feed rate while holding the "drilling power" constant (= speed × feed) and using a cooling fluid. Although the chips were also studied, their inherent shape and two-sidedness precluded meaningful data collection. Because of the success of this project, future experiments at the HTML's X14A synchrotron X-ray source were proposed.

Ti alloys offer a high strength-to-weight ratio up to a high temperature level, which is needed for light-weight material applications. The project's significance is primarily related to the energy savings possible in applying Ti components for heavy-duty trucks. Potential applications include the turbocharger compressor, drive shaft and axles, and framework and suspensions.

Residual Stresses due to Explosive Welding in Pressure Vessels

Industry Collaborator: D. J. Taylor
HTML Staff: T. R. Watkins, C. R. Hubbard

TPL, Inc., of Albuquerque, New Mexico, visited the RSUC to characterize the stresses introduced by their cladding process. Refractory metal liners are explosively welded into pressure vessels made of mild steel to increase wear and erosion resistance. The cladding increases the useful lifetime of these vessels by a factor of 3 up to possibly 10. The increase represents a potential cost savings not only in fewer vessels used, but also in time, logistics, and labor to replace vessels less frequently. Since the residual stress profile of these vessels is important to their use and lifetime, the effect of the explosive cladding process on residual strain must be determined to compensate for it in production.

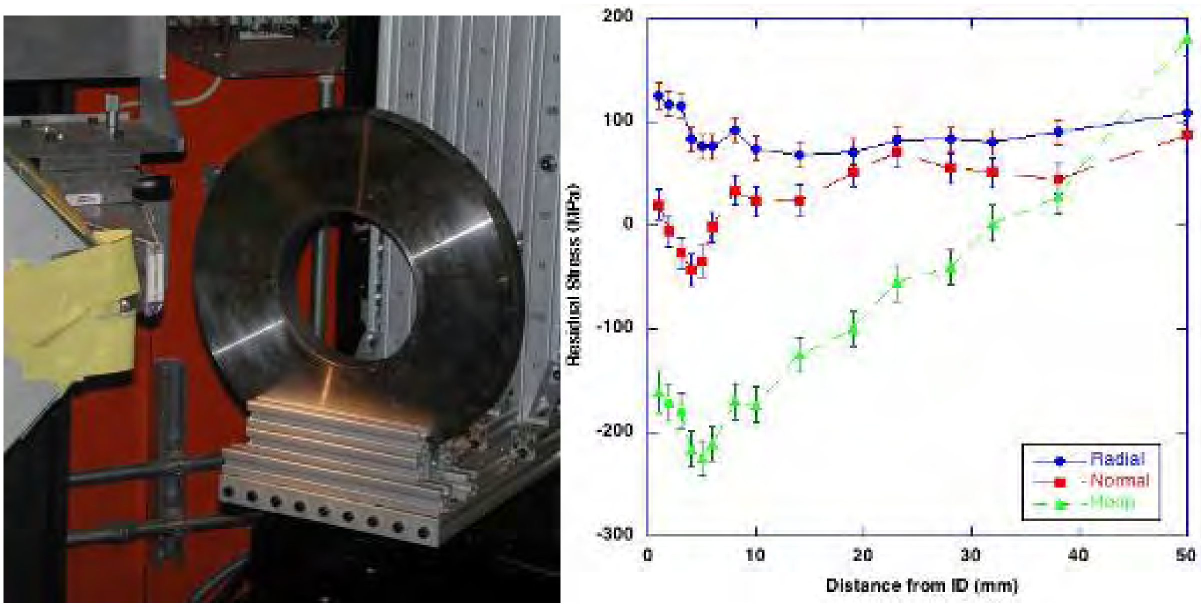


Fig. 5-5. Clad ring sample mounted on the NRSF2's XYZ goniometer at HFIR (left); the residual stresses in an initially stress-free vessel that was then clad (right).

Three ring samples were cut from larger vessels: (1) as-received non-clad (approximately stress-free), (2) clad within a stress-free vessel (Fig. 5-5), and (3) clad within a pre-stressed vessel. The ring slices were examined using the NRSF2 at HFIR for through-thickness mapping of the residual stresses. The stress values are calculated from the three orthogonal strains measured for each location along the diameter. Near the inside diameter (ID), evidence of stress changes due to the cladding is apparent. All residual stresses appear to level off within the first 20 to 40 mm from the cladding on the ring's ID. Comparing the clad samples with and without pre-stressing suggests that two-thirds of the peak compressive residual stress in the hoop and radial directions is due to the pre-stressing process. This peak residual compressive stress occurs 2 to 5 mm out from the ID. Most of the stresses in the normal direction were relieved by the slicing. This work was presented at the American Conference on Neutron Scattering in June 2006.

IQ Technologies and EMTEC Characterize Residual Surface Stresses in Heat-Treated Steel Parts

Industry Collaborators: P. J. Gros, Jr., M. Aronov
HTML Staff: C. R. Hubbard, W. B. Bailey, Fei Tang

Under the HTML User Program, IQ Technologies and Edison Materials Technology Center (EMTEC) are conducting a comprehensive study to evaluate residual stress conditions due to a new rapid quench heat treatment for a variety of steel samples, several of which are typical steels used in the automotive industry. This is a significant study, because carburized and quench-hardened steels and induction-hardened steels are used in both land and air transportation power-train systems where fatigue is a common limiting condition. Compressive residual stress in component surface layers is known to improve fatigue life, so characterizing the magnitude and depth of residual compression from steel heat treatment is a significant factor in improving ground and air transportation reliability or reducing component weight.

Oil quenching for some steels and induction hardening for others is widespread in the automotive industry. Over the last several years, IQ Technologies, Inc., of Akron, Ohio, has been developing an alternative method of hardening steel parts known as IntensiQuench® (IQ), an interrupted quenching technique that uses highly agitated, environmentally friendly plain water as the quench media instead of oils or polymers. Table 5-1 shows the improvement in compressive residual surface hoop stresses due to the IQ process.

Table 5-1. Improvement compressive residual surface hoop stresses due to IQ process

Test sample diameter, mm	Steel	Heat treatment	Surface residual hoop stresses, MPa
38	8620	Carburized and oil quenched	-137
		Carburized and intensively quenched	-606
29	52100	Intensively quenched	-959
38	1050	Induction case hardened	-668
		Intensively quenched	-1,035
25	5160	Oil quenched and shot peened	-563
		Intensively quenched and shot peened	-839
		Intensively quenched with no shot peening	-939

Neutron mapping for the 5160 steel rods was completed in December. Figure 5-6 below shows the neutron hoop strain measurement results from ~0.3 to over 2 mm from the sample surface. During the next HFIR operating cycle, neutron diffraction will be used to characterize the depth profile for all the samples. Results from this one neutron measurement, however, show that the IQ-imposed stresses likely extend to several millimeters in depth, a very deep case.

Intensive quenching achieved a higher magnitude of the beneficial residual compression than either oil quenching or induction hardening. Armed with unbiased and high quality data from ORNL, IQ Technology can now push for further implementation of the IntensiQuench process to extend the fatigue life of critical components for the U.S. transportation industry.

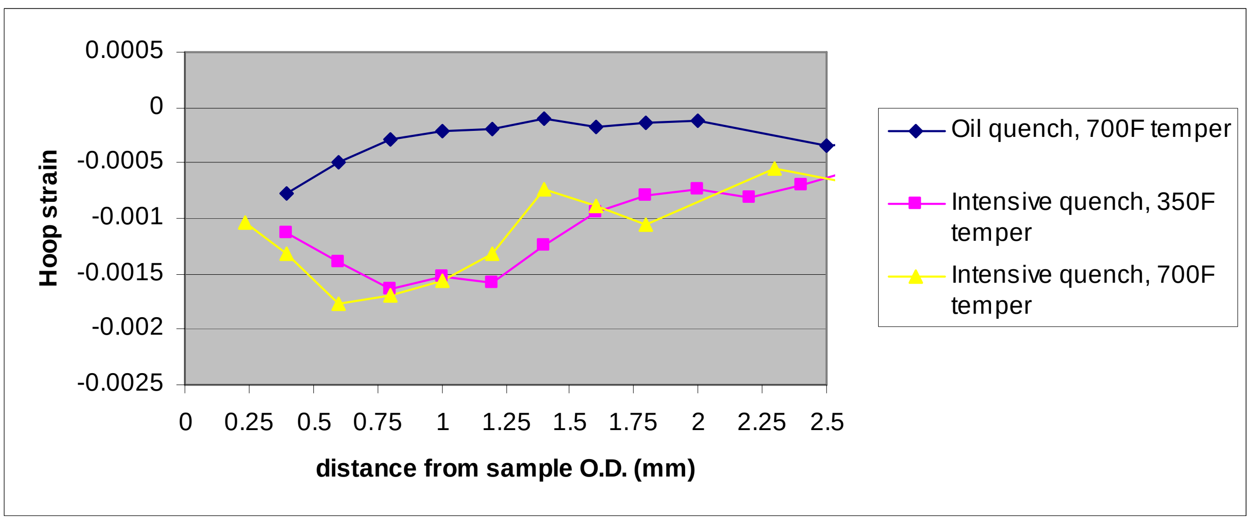
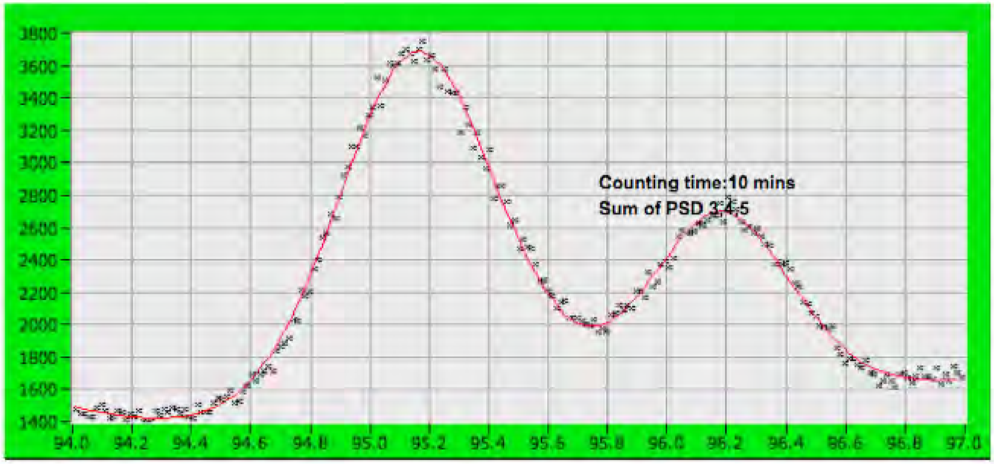


Fig. 5-6. Hoop strains in 5160 steel rod as measured at the High Flux Isotope Reactor (HFIR).

Neutron Strain Mapping Shown to be Applicable to Corning's Cordierite

Industry Collaborators: J. Webb, D. Wilcox
HTML Staff: C. R. Hubbard, W. B. Bailey, K. An

Drs. James Webb and David Wilcox of Corning, Inc., assessed the potential use of neutron strain mapping within highly porous cordierite ceramic parts. The trials showed that selected diffraction peaks from cordierite could be measured on NRSF2 (Fig. 5-7 next page). During the assessment, residual micro-strains parallel and perpendicular to the extrusion direction were examined for four different diffraction peaks. The results imply that there are different residual microstresses in the two directions and that there is significant hkl dependence due to crystal anisotropy (Fig. 5-8b). Subjecting the ceramic to compressive load on NRSF2 showed that the neutron method is capable of measuring changes in strain as a function of load as small as 100 psi.



hkl's	intensity	FWHM (deg 2θ)	Position (deg 2θ)	d-spacing (nm)
[173]	2800±93	0.600±0.008	95.161±0.001	0.1275669±0.000012
[1133]/[1043]	1641±56	0.517±0.017	96.180±0.002	0.1265431±0.000022

Fig. 5-7. Peak fitting by NRSF2's multi-peak fitting function with Gaussian profile model. Sample was reference cordierite powder in a glass tube, and the neutron wavelength was 0.188349 μm.

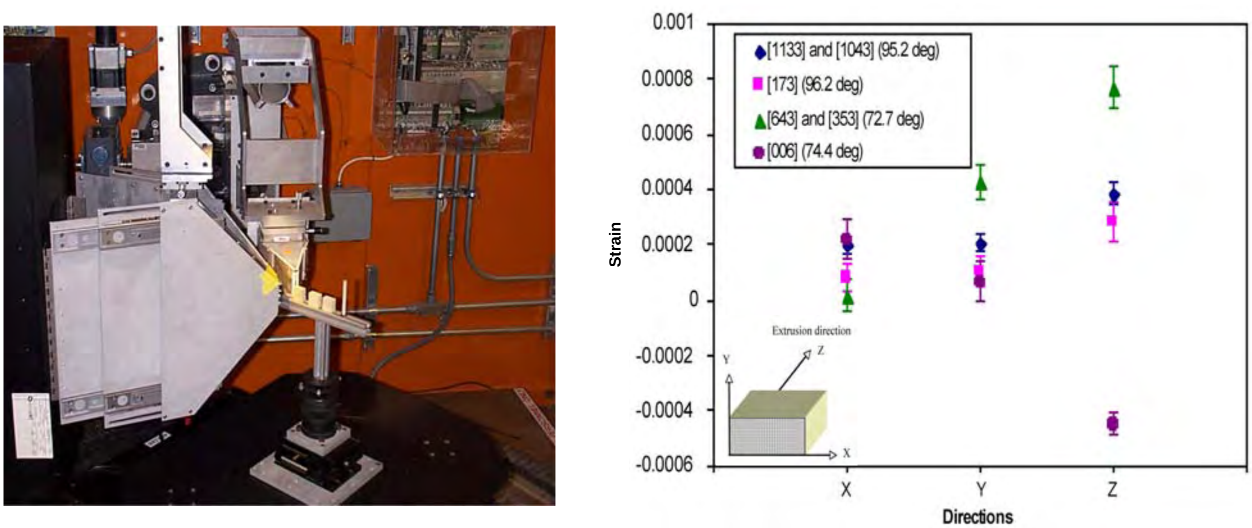


Fig. 5-8. The NRSF2's XYZ goniometer at HFIR (left); microstrains parallel to the extrusion direction (z) and perpendicular (x,y) for four different hkl in porous cordierite ceramics (right).

In-situ Real-time Neutron Diffraction Study of Materials Behavior under Thermally Induced Stresses

LDRD Partners: W. Woo, Z. Feng, X.L. Wang, S. David
HTML Staff: K. An, W. B. Bailey, C. R. Hubbard

Material state changes (e.g., phase, stress, microstructure and temperature) under rapid, severe thermal and mechanical deformation are a key topic in materials science and engineering, since such deformation processing is widely used in the transportation industry for items such as extruded beams and stamped auto body panels. The deep penetration capability of neutrons enables a greater understanding of the mechanical properties and structures within the bulk. However, due to insufficient fluxes, gathering data for neutron diffraction has been primarily limited to "static" behavior; capturing the changes of material state related to dynamic processes has not been possible.

A new technique being explored in this ORNL-Laboratory Directed R&D (LDRD) project is in-situ time-resolved neutron measurement based on the quasi-steady state (QSS) phenomenon. In the QSS, the distribution of material state is stationary in the *Eulerian* coordinate system where the observation point is fixed to the processing zone instead of the *Lagrangian* coordinate system observed from the laboratory point of view. The purpose is (1) experimental accomplishment of the direct (*Lagrangian*) measurements and QSS (*Eulerian*) measurements, and (2) establishment of the equivalency of the two measurements. The demonstrated equivalency (Fig. 5-10 next page) shows that neutron mapping methods can be applied to many processes and be used to develop and validate process models, e.g., friction stir welding and processing or extrusion.

In the basic experimental setup, the thermal stresses were imposed on a local area of 6061-T6 aluminum alloy plates with an electric heating gun (Fig. 5-9). A careful temperature control imposed a uniform thermal distribution within the neutron scattering volume (approximately 5 × 5 × 2 mm). Direct measurements were performed by having the moving heat source approach the fixed beam position. Neutron diffraction measurements every 20 seconds were sufficient for the in-situ real-time data.

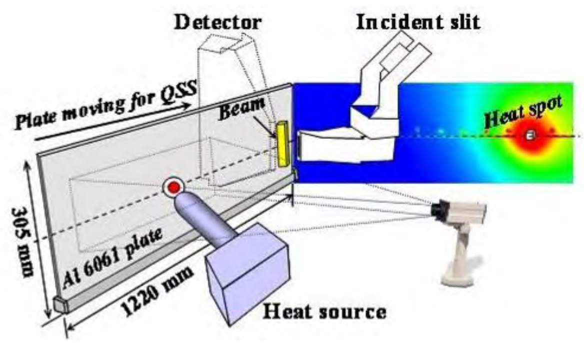


Fig. 5-9. Setup for moving an Al plate past a fixed heat source.

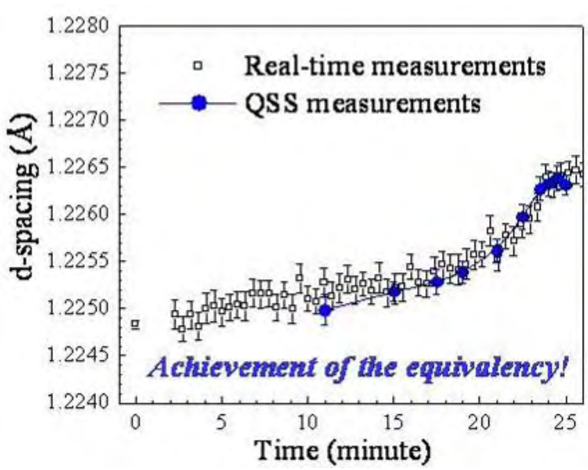


Fig. 5-10. Comparison of direct and quasi-steady-state d-spacing results.

For the QSS measurements, the relative distances between the fixed neutron beam and heat source position were set up for each data point. For the QSS data, 10-minute measurements were taken while the plates were moved in a uniform manner. The lattice d-spacings obtained (sub sampling) within the QSS period were constant, indicating that the material state remained steady. Figure 5-10 compares the direct measurements and QSS results of the transient response by the localized heat source using in-situ real-time neutron diffraction. The results from the two methods are essentially identical and thus validate the QSS method. Thus, for characterizing the materials state when a QSS condition exists (e.g., welding, extrusion, rolling) the neutron collection time can be made independent of the rate of change in the processes.

UT Engineers Visit RSUC to Compare Residual Stresses Following Deformation Arising from Torsion vs. Tension in a Cylindrical Rod

University Collaborators: X, Luo, D. Penumadu
HTML Staff: C. R. Hubbard, K. An, W. B. Bailey, and T. R. Watkins

University of Tennessee Civil Engineering Department researchers visited the RSUC to explore the intergranular residual stress effects of combined axial/tension plus torsional testing (Fig. 5-11). The samples were prepared using a system designed to investigate the effect of rotation of principal stress axes on the stress-strain behavior of frictional materials. The stress and strain state within the cylindrical steel rods during combined axial/tension and torsional loading is complex and is often interpreted using a fundamental assumption that the globally applied load results in a state of strain or stress throughout the specimen independent of the type of applied load. This assumption has not previously been tested. Diffraction methods can reveal grain-to-grain stresses and strain localizations due to the formation of narrow bands called shear bands. Initiation of shear bands is most pronounced under the application of the external torque or twisting of a specimen. The deformation behavior under pure tension versus pure torsion and the strain variation along the height of the specimen is being studied for a set of deformed steel tubes using both neutron and x-ray diffraction methods. Neutron diffraction data were collected at HFIR, and x-ray diffraction studies were undertaken at the HTML to help determine the surface residual stress and importantly the reference/stress-free lattice parameter and consequently, the stress-free interplanar spacings. This parameter is particularly important as it is required to calculate the strains from the neutron data. An initial value for stress-free d-spacing of 2.8672 Å was obtained. The neutron diffraction data show clear evidence of significant differences in intergranular stresses for the two deformation mechanisms. The study provided added evidence that the load frame being specified for use at VULCAN should include a torsion actuator. Having biaxial loading capabilities at VULCAN will make it a unique facility for studies of material behavior under biaxial loads and/or fatigue.

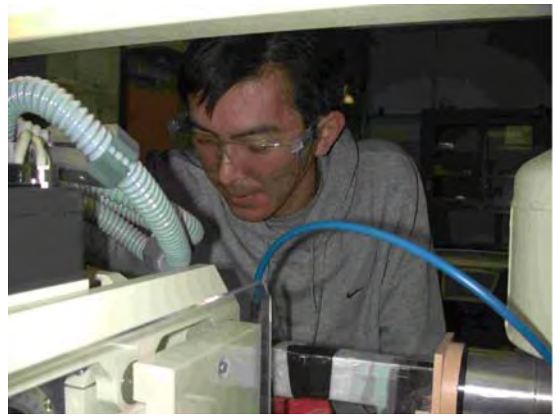


Fig. 5-11. UT civil engineering doctoral candidate Xin Luo mounts a specimen on the PTS tube x-ray diffractometer.

Stresses in Gears Measured Using X-ray Diffraction

University Collaborators: R.L. LeMaster, B. Boggs, J. Bunn
HTML Staff: C. R. Hubbard, T. R. Watkins, W. B. Bailey

UT Martin professor Robert LeMaster and students Bryan Boggs and Jeff Bunn worked at the HTML under a Faculty and Student Team (FaST) program to study the effect of machining of carburized gears. B&R Gear prepared the gears specifically for this study (Fig. 5-12a next page). This research sought to quantify the change in residual stress in a carburized gear as it progresses through the finishing process. The HTML's Large Specimen X-Ray Residual Stress Mapping Facility was used to characterize residual stresses in both finished and unfinished carburized gears. The gears were mapped across and along the face width of a gear tooth at 10 radial positions and at 8 positions across the tooth width (Fig. 5-12b next page). The unfinished gear showed little variation of compressive residual stresses while the finished gear showed a significantly larger variation with up to a 50% reduction (Fig. 5-13 next page), which correlated generally with the amount of material removed during grinding. The HTML's coordinate measuring machine was used to obtain metrology data. This gear machining research is part of a larger research project in which neutron diffraction will be used to penetrate deeper into the specimen and to complement the near surface x-ray stress data.

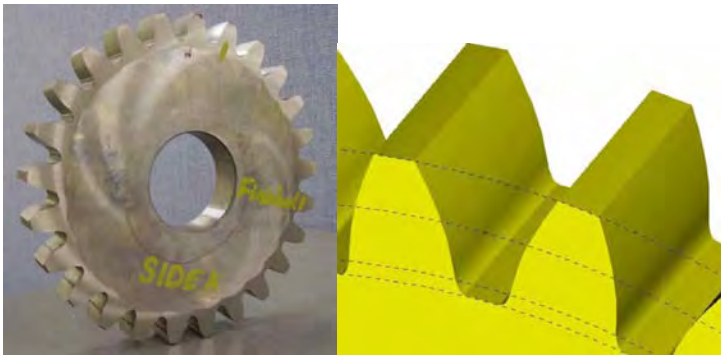


Fig. 5-12. Carburized gear prepared for residual stress study (left); the gears mapped across and along the face width of a gear tooth at 10 radial positions and 8 positions across the tooth width (right).

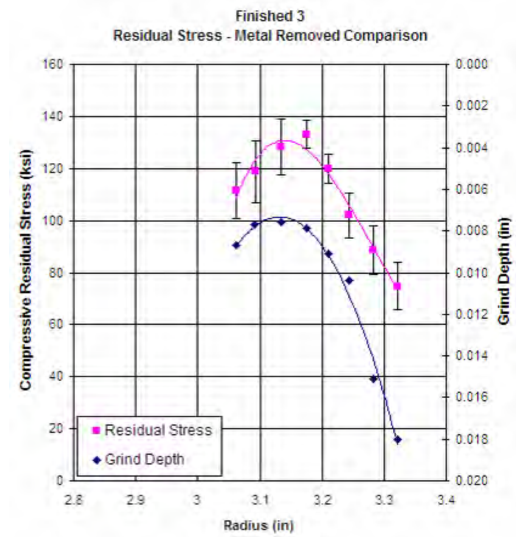


Fig. 5-13. The unfinished gear showed little variation of compressive residual stresses; the finished gear showed a significantly larger variation.

Direct in-situ characterization of surface and through thickness stresses in gears has not been possible previously. The unique combination of x-ray and neutron strain mapping facilities at the HTML will provide in-depth understanding with potential for improvement of models for gear systems. Such understanding could increase the power density of transmissions and reduce probability of failure.

RSUC News

Camden Hubbard Elected Fellow of the American Ceramic Society

The American Ceramic Society Board of Directors, at its May 12, 2006, meeting, unanimously elected Dr. Hubbard to the grade of Fellow of the Society. This achievement was recognized at the Honors and Awards Banquet at our 108th Annual Meeting, Monday, October 16, 2006, in Cincinnati, Ohio, USA.

Camden Hubbard continues to serve on the International Centre for Diffraction Data board of directors; he organized and chaired sessions at the Denver X-Ray Conference.

Camden Hubbard Elected to Distinguished Fellow of the International Centre for Diffraction Data

At the March Annual Meeting of the International Centre for Diffraction Data (ICDD), Dr. Hubbard was elected to Distinguished Fellow in recognition of his extensive contributions to the ICDD and field of x-ray powder diffraction. The ICDD is an international organization that aims to advance the science and database used for phase identification, quantitative phase analysis, and materials characterization using diffraction methods. Hubbard developed the editorial review program NBS*AIDS83 that continues as the basis of the editorial process, lead the ICDD Research Associateship at NBS (now NIST) that generated nearly 2000 high quality reference powder patterns of important phases, developed key XRD analysis methods, certified SRMs for diffraction, and made significant contributions to advancing quantitative XRD. Since joining ORNL he has been actively involved in both XRD and neutron diffraction efforts including the Diffraction and Residual Stress User Centers of the High Temperature Materials Laboratory.

ASM International Heat Treating Society R&D Committee Met at ORNL

The ASM International Heat Treating Society R&D Committee held its "face-to-face" meeting at ORNL on March 30, 2006. The meeting was sponsored by EERE-ITP Program Manager Dr. Peter Angelini, and hosted by Committee Chair Dr. Gerard Ludtka of ORNL's Materials Science and Technology Division. In the 1990s, this Committee developed the Roadmap and Vision 2020 documents to support technology needs in parallel with the ITP Program goals. Currently, this team has revised the priorities of the technology challenges and will be publishing these in three articles in the Heat Treating Progress magazine over the next several months. These articles will deal specifically with the three main thrusts of: "Improved Process and Materials Technology," "Energy and Environmental Technology," and "Equipment and Hardware Technology."

A significant outcome of this meeting in support of the first thrust area is the initiation of collaborative efforts with ASM International to develop electronic materials and processing databases. The initial effort will focus on establishing a phase transformation kinetics and dilation strain database to support leading-edge microstructure evolution and process modeling endeavors within industry. This pedigreed, archival database will have significant ramifications and benefits to the heat-treating industry and as a consequence, the transportation industry.

During the working lunch, Dr. Ludtka gave a presentation on "Exploring Magnetic Field Processing for Developing Customized Microstructures with Enhanced Performance." This exciting new technology promises aid in the development of the next generation of structural and functional materials.

As part of this full-day meeting, three tours were given to highlight several of ORNL's major User Programs. These included the Materials Processing Group, the High Temperature Materials Laboratory, and the SNS. Feedback included "I was in awe of ORNL's capabilities" and "I felt like a freshman on his first day in college." The ASM meeting generated several industrial requests to pursue collaborative and User Program R&D with ORNL.

Awards

Wanchuck "Chuck" Woo (right), a Ph.D. candidate in the Department of Materials Science and Engineering, University of Tennessee, won the "2006 Jerome B. Cohen Student Award." This award recognizes the outstanding achievements of his thesis research as evidenced by his paper, "In-Situ Time-Resolved Neutron Diffraction Measurement of Transient Material States during a Thermo-Mechanical Process Based on Quasi-Steady State Principle." The award was given at the 55th Annual Denver X-ray Conference, held August 7–11 in Denver, Colorado. This award, instituted in the memory of Professor Jerome B. Cohen, one of the leaders in the field of X-ray stress analysis and in the training of students in x-ray analysis, recognizes the outstanding achievements of student research in this field. Woo's paper was based on research conducted on the HTML's NRSF2 instrument at HFIR and the SMARTS instrument at Los Alamos National Laboratory in New Mexico.

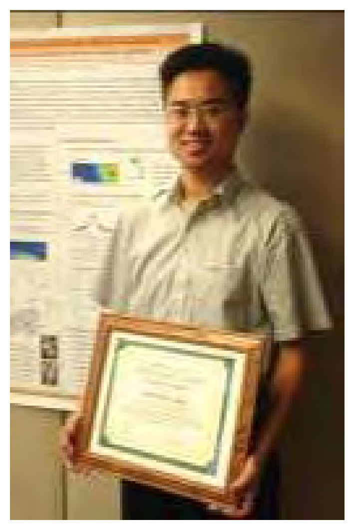


Fig. 5-14. Chuck Woo, University of Tennessee Ph.D. candidate.

Elena Garlea, also a University of Tennessee graduate student, was recognized for her research presentation on determining hydrogen distribution in zircalloy alloys. The NSSA Prize for Outstanding Student Research (at right) was presented by the Neutron Scattering Society of America at the 2006 American Conference on Neutron Scattering, June 18-22, 2006. The work presented was performed on the HTML's NRSF2 instrument at HFIR and at the ENGIN-X instrument at ISIS, Rutherford Laboratory, England. The study demonstrates the potential for mapping a hydrogen concentration gradient within samples using incoherent scattering and diffraction methods. Plans are to use this foundation to conduct in situ studies on the effects of hydrogen charging on strains around crack tips.

Publication

Thomas R. Watkins, O. Burl Cavin, and Camden R. Hubbard (HTML), Beth Matlock (TEC-Knoxville), and Roger D. England (Cummins Engine), "Considerations Regarding the Alignment of Diffractometers for Residual Stress Analysis," Rigaku Journal, Vol 23, May 2006. This invited paper and extended appendix summarize the presentations given at the Residual Stress Diffractometer Alignment Workshop presented at the Denver X-Ray Conference, 2001 and 2005. The paper can be retrieved at http://www.rigakumsc.com/journal/volume23-1.html

6. THERMOGRAPHY AND THERMOPHYSICAL PROPERTIES USER CENTER (TTPUC)

User Center Members:

Ralph B. Dinwiddie, Leader
Geneva N. Worley, Administrative 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, carbon, and metal matrix composites; ceramic precursors; superconducting materials; carbon materials; powders; and foams.

The TTPUC continues to develop capabilities in the field of thermography 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 for a wide variety of materials processes, in-service performance characterizations, temperature mapping, and nondestructive evaluation (NDE) inspections.

TTPUC Instruments

- Laser flash thermal diffusivity system (cryogenic temperatures to 2500°C)
- Xenon flash thermal diffusivity system (room temperature)
- Hot disk thermal constants analyzer (cryogenic temperatures to 750°C)
- Three-omega thermal diffusivity system (room temperature to 450°C)
- Radiance-HSX IR camera (3–5 microns)
- Alpha TE cooled IR camera (7.5–13 microns)
- Omega uncooled IR camera (7.5–13 microns)
- Phoenix mid-wavelength IR camera (1.5–5 microns)
- Phoenix near-IR camera (0.9–1.7 microns)
- Hyperspectral imaging lens (3–5 microns)
- IR microscope (3–5 microns)
- High-speed two-color single-point IR detectors (100 kHz)
- ThermoSonix instrument for nondestructive evaluations
- Netzsch differential scanning calorimeter (room temperature to 1600°C)
- Dual-push-rod dilatometer (cryogenic temperatures to 1600°C)
- Simultaneous thermal analyzer (room temperature to 1500°C)
- Chan large-sample thermogravimetry analysis (room temperature to 1700°C)
- Total hemispherical emittance and electrical resistivity (100° to 1400°C)
- Seebeck coefficient (room temperature to 800°C)
- Visible light spectrometer for reflectance and transmittance studies
- Near-IR spectrometer (0.9–2.6 microns)
- Signatone Pro4 (electrical resistivity)

Selected Highlights

Miami University Studies Thermal Conductivity of Paper Coatings at TTTPUC

University Collaborator: Lei Kerr
HTML Staff: Ralph B. Dinwiddie and Hsin Wang

Fig. 6-1. Professor Lei Kerr.

Lei Kerr, an assistant professor from the Department of Paper and Chemical Engineering at Miami University, Oxford, Ohio (Fig. 6-1), visited the TTPUC as an HTML user to study the thermal conductivity of paper coatings. The objective of the project was to gain an understanding of fundamental science and engineering aspects of thermal conductivity of paper coatings to advance the paper industry's ability to make better products for various digital printing applications and to be more globally competitive. Thermal conductivity is very important for many applications in which paper is used (e.g., thermal printing; electrophotographic printing or laser printing, in which dry toners are used; and Indigo printing, where liquid toners are used). Other applications include flooring underlayment, high-pressure laminate, and insulation.

Lower thermal conductivity would help to trap and retain heat at or near the printing surface of the paper during toner fusing, ink drying, and image development. For example, in the fusing stage of the laser printing process, heat is applied to the toner and the paper as the paper passes through the fuser assembly. The heat melts the toner, which then permanently attaches to the paper as it cools. Thus, the study will benefit the hardware manufacturers, who will be able to produce devices such as laser printers that are more efficient. These devices are designed to handle paper having controlled heat-transfer properties.

During the 3-day visit, more than 40 paper sheets with different coating thicknesses and variable coating conditions were tested in a hot-disk thermal conductivity measurement system. Thin-film mode was used for all the measurements. The results were consistent with those of printing performance tests at Miami University. A follow-up visit is planned for heat-capacity and thermal-diffusivity measurements. Bulk coating material will also be brought for measurements during the next visit.

Understanding materials structures such as those in paper has potential for application in better filtration systems in a variety of energy-related uses.

Researchers from State University of New York at Stony Brook Continue Studies of Thermal Barrier Coatings at TTPUC

University Collaborators: Weiguang Chi, Yang Tan, and Sanjay Sampath
HTML Staff: Hsin Wang

STONY BROOK UNIVERSITY

Two graduate students from the State University of New York at Stony Brook made a second visit to TTPUC to study the relationship between microstructure and thermal transport properties of plasma-sprayed thermal barrier coatings (TBCs). (Results from the previous visit were presented at the International Thermal Spray Conference, Seattle, Washington, May 15–17, 2006). As part of the research project, undertaken at the Center for Thermal Spray Research (CTSR) under the direction of Dr. Sanjay Sampath, the researchers characterized the microstructure of a series of TBCs.

Based on results obtained during the summer, an updated test matrix was designed. As-sprayed TBCs were heat-treated at high temperatures to stabilize the microstructure. Thermal diffusivity tests of 36 specimens (6 groups of 6 samples) were conducted from room temperature to 1200ºC.

IR imaging was also used to get cross-section temperature maps of TBCs. A microscope lens with 5.5-micron/pixel resolution was used in the study. The temperature maps will be compared with micro-Finite Element Analysis (FEA) results generated at Stony Brook to better understand heat flow in TBCs.

University of Central Florida and Sporian Microsystems, Inc., Visited TTPUC

University Collaborator: Linan An
Industrial Collaborators: Wenge Zhang and Bill K. Garret
HTML Staff: Hsin Wang, Wally D. Porter

The University of Central Florida (UCF) and Sporian Microsystems, Inc. have jointly developed polymer-derived ceramics (PDCs) for MEMS applications and for high-temperature sensor applications. A professor from UCF and two researchers from Sporian visited HTML. Thermophysical properties of PDCs are not available, especially at elevated temperatures. Thermal diffusivity, specific heat, and thermal expansion tests were conducted at TTPUC on SiCN and SiAlCN from room temperature to 1000ºC. The newly installed Signatone Pro4 probe station system was used to measure the electrical resistivity of some semiconducting PDCs at room temperature. During the visit, the mechanical properties of PDCs were studied at facilities in the Mechanical Characterization and Analysis User Center (see page 17f).

The UCF researchers also conducted initial testing for a newly approved proposal on aluminum metal matrix composites (MMCs). With the addition of nano-sized alumina particles, the thermal conductivity of the MMCs decreased. Thermal conductivity was studied as a function of particle size and composition. Wear properties of the MMCs were also investigated at the Friction, Wear, and Machinability User Center (see page 11f).

Ford Conducted IR NDE Inspection of Automotive Spot Welds

Industry Collaborators: Bill Charron and Todd Cleaver
HTML Staff: Hsin Wang

Two researchers from Ford Motor Company tested 75 spot-weld specimens at HTML (Fig. 6-2) as part of the USCAR/DOE study on non-destructive evaluation (NDE) of spot welds. Participants in the project also included General Motors and the Lawrence Berkeley Laboratory. The specimens had been studied by other NDE techniques and were going to be studied destructively after the HTML study. One of the important requirements to leave the surface undisturbed (e.g., no painting was allowed because it would alter the surface reflection).

Fig. 6-2. Todd Cleaver, Ford Motor Co., examining a spot weld specimen.

Weld quality was studied using an infra-red (IR) camera and image analysis software developed at HTML. The purpose was to evaluate NDE techniques that can be used by automakers to replace a labor-intensive, destructive tear-down process. All 75 specimens tested in one day demonstrated the rapid inspection capability of the technique. The IR images are being analyzed at HTML and will be used to correlate weld quality and results from other NDE techniques.

Northwestern University Studied Biomorphic SiC at HTML

University Collaborators: Kristen Pappacena and Kathy Faber
HTML Staff: Hsin Wang

A Northwestern University researcher studied thermal transport properties of SiC converted from natural wood at TTPUC. Biomorphic silicon carbide is a ceramic material derived from natural wood precursors. The processing, performed by collaborators from the University of Seville, Spain, includes a several-step procedure, which begins with pyrolizing the wood in an inert environment, leaving behind a carbon scaffold with the same cellular microstructure as the wood. The carbon scaffold is subsequently infiltrated with liquid silicon at 1550°C, and the reaction Si + C -> SiC occurs. This results in a SiC cellular structure, which closely resembles the initial microstructure of the original wood. Treatment in an HF/HNO_3 etch removes the excess silicon from the reaction. The project's goal is to understand the microstructure-property relationships in the material. To establish the relationship between the structure and properties of biomorphic silicon carbide, several different woods with silicon carbide materials of different densities and pore distributions were examined.

Using the Anter laser flash system, 72 specimens were tested, and the specific heat of several of these was measured with the differential scanning calorimeter (DSC) (Fig. 6-3). Several groups of carbon and graphite materials with the same wood textures before conversion to SiC were also studied. The specimens were grouped by axial and off-axial orientations. The off-axial specimens showed lower thermal conductivity; the axial specimens showed 30 to 40% higher thermal conductivity. A special SiC coating was used to seal the top surface pores to prevent laser penetration. The thermal transport data will be used along with thermal shock resistance test results to evaluate the potential for application as an automotive filter/converter.

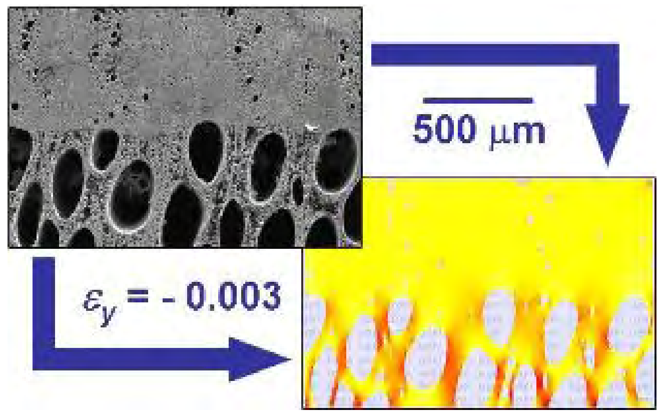


Fig. 6-3. Wood sample (left) prior to silicon infiltration and resultant structure after conversion to SiC (right).

Saint-Gobain Continues Study on Intrinsic Thermal Transport Properties of Doped and Undoped Zirconia Powders

SAINT-GOBAIN

Industry Collaborator: Gaëtan Bonhomme
HTML Staff: Hsin Wang and Wally Porter

A Saint-Gobain researcher continued his study on intrinsic thermal transport properties of zirconia powders at the TTPUC. In previous visits, materials with different doping levels were sintered to near theoretical density and designed porosity levels. It was determined that the effect of porosity is equal to, if not larger than, the effect of doping on thermal conductivity. A new group of specimens was prepared with selected porosity and doping levels. A total of 18 specimens was tested using the Anter laser flash system from room temperature to 1400ºC. Room-temperature diffusivity values were measured using the xenon flash system before and after each test. The specific heat of two to three compositions was analyzed using the DSC. The study was designed to confirm findings of previous visits and to finalize the study of zirconia powders. Zirconia is currently being used to manufacture several engine components, including fuel injectors and thermal barrier coatings (TBCs).

Adiabatics, Inc., Studied Low Cost TBCs for Diesel Engines

Industry Collaborators: Dorsaf Saad and Philippe Saad
HTML Staff: Hsin Wang

Two researchers from Adiabatics, Inc. studied low-cost TBCs intended for diesel engines. The aim of the project is to test thermophysical properties of coatings on steel and aluminum substrates. The coatings were sprayed onto the substrates and then cured at about 400ºF. DSC tests found continued curing in some coatings. Thermal diffusivity measurements on two-layer samples were conducted on the Anter system. It was found that lower-thermal-conductivity stainless steel (instead of carbon steel or aluminum) should be used, as well as the thin-film mode of the Hot Disk system. Thermal conductivity of uncured

TBCs was obtained. This initial visit gave the researchers first-hand information on their coatings. Experiments to improve on heat treatment and fabrication of samples with a higher coating-to-substrate thickness ratio are being conducted at Adiabatics.

TTPUC News

TTPUC Recognized as Leader in Characterization of Thermoelectrics

At the recent International Thermoelectric Conference (ITC' 06) held in Vienna, Austria, and at the Direct Thermal Energy Conversion Workshop (organized by the Office of Naval Research, the Defense Advanced Research Projects Agency, the National Aeronautics and Space Administration, and DOE), HTML staff presented results of thermoelectric material characterization. The addition of high-temperature Seebeck and electrical resistivity measurement capabilities and the traditional TTPUC strength in thermal transport properties measurements have enhanced the HTML's position as a leader in high-temperature thermoelectric characterization. Most state-of-the-art high-temperature thermoelectrics have been tested in a short time, and the results were cross-checked with other laboratories at industries and universities. The TTPUC is currently participating in a waste heat recovery program sponsored by the Office of FreedomCAR and Vehicle Technologies (FCVT).

TTPUC Installs ULVAC ZEM-2 for High Temperature Seebeck Coefficient and Electrical Resistivity Testing

Auto exhaust, at temperatures averaging 300º–600ºC, is ideal for thermoelectric power generation. The ULVAC ZEM-2 system was successfully installed at the TTPUC (Fig. 6-4). A capital investment by the HTML, this system will be able to measure Seebeck coefficient and electrical resistivity of thermoelectric materials from room temperature to 800ºC; temperature range with future upgrades could be expanded to –80ºC and 1000ºC. Initial testing indicated that the system met all the technical specifications. It will be used to characterize high-temperature thermoelectric materials for an FCVT waste heat recovery project and other user projects.

Fig. 6-4. The ULVAC ZEM-2 system installed at the HTML.

NEW HTML USER AGREEMENTS EXECUTED DURING FY 2006

Executed	Agreement #	User Agreement Partner
10/5/2005	UR-05-483	Sporian Microsystems, Inc., Lafayette, CO
10/9/2005	UR-05-478	Applied Science, Inc., Cedarville, OH
10/20/2005	UR-05-480	Edison Materials Technology Center and IQ Technologies, Dayton, OH
10/14/2005	UR-05-482	The Queen City Forging Company, Cincinnati, OH
11/22/2005	UR-05-485	SemiSouth, Starkville, MS
11/30/2005	UR-05-487	Biohorizons, Birmingham, AL
2/24/2006	UR-06-527	Hyper-Therm High Temperature Composites, Inc., Huntington Beach, CA
4/12/2006	UR-06-520	GrafTech International LTD, Lakewood, OH
4/21/2006	UR-06-536	Trex Enterprises, Inc., Lihue, HI
7/6/2006	UR-06-557	Adiabatics Technology, Inc., Columbus, IN
7/12/2006	UR-06-561	State University of New York at Binghamton, NY
7/31/2006	UF-06-523	Component Composite Coatings International, LLC, Alpharetta, GA
8/10/2006	UR-06-563	ThyssenKrupp VDM USA Inc., Tipton, IN
8/10/2006	UR-06-556	Novatek, Inc., Provo, UT
8/18/2006	UR-06-568	Capstone Turbine Corporation, Chatsworth, CA
6/12/2006	UR-06-550	ECK Industries, Inc., Manitowoc, WI
9/26/2006	UR-06-576	AppliCote Associates, LLC, Sanford, FL
9/22/2006	UR-06-559	Novelis, Kingston, Ontario, Canada
9/28/2006	UR-06-555	Toyota Technical Center USA, Inc., Ann Arbor, MI

PUBLICATIONS AND PRESENTATIONS FY 2006

Journal Articles

An Ke, K. L. Reifsnider, C. Y. Gao, "Durability of $(Pr_{0.7}Sr_{0.3})MnO_{3\pm d}$ 8YSZ composite cathodes for solid oxide fuel cells," *J. Power Sources* **158**, 254-262 (2006)

Aytug, T., M. Paranthaman, K. J. Leonard, H. Y. Zhai, M. S. Bhuiyan, **E. A. Payzant**, A. Goyal, S. Sathyamurthy, D. B. Beach, P. M. Martin, D. K. Christen, X. Li, T. Kodenkandath, U. Schoop, and M. W. Rupich, "Assessment of chemical solution synthesis and properties of $Gd_2Zr_2O_7$ thin films as buffer layers for second generation high-temperature superconductor wires,"*J. Mater. Res.* **20**, 2988-2996 (2005)

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