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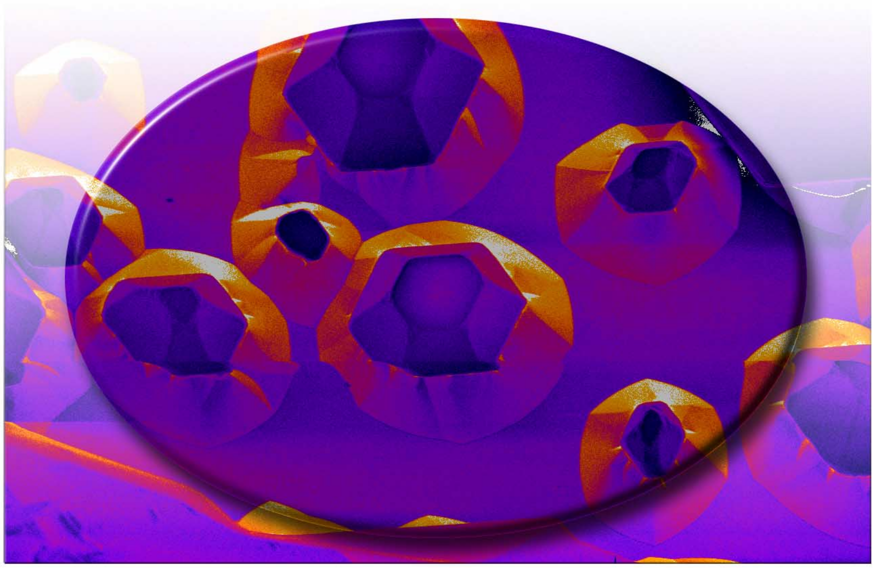
ORNL/TM-2001/185

High Temperature Materials Laboratory Annual Report

FY 2000 (October 1, 1999–September 30, 2000)



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HIGH TEMPERATURE MATERIALS LABORATORY

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High Temperature Materials Laboratory

THIRTEENTH ANNUAL REPORT:
OCTOBER 1999 THROUGH SEPTEMBER 2000

A. E. Pasto
B. J. Russell

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ADVANCED MATERIALS CHARACTERIZATION AT THE HIGH TEMPERATURE MATERIALS LABORATORY

Arvid E. Pasto, Director
High Temperature Materials Laboratory

The Facility

The High Temperature Materials Laboratory (HTML) is designed to assist American industries, universities, and governmental agencies develop advanced materials by providing a skilled staff and numerous sophisticated, often one-of-a-kind pieces of materials characterization equipment. It is a nationally designated user facility sponsored by the U.S. Department of Energy's (DOE's) office of Transportation Technologies, Energy Efficiency and Renewable Energy. Physically, it is a 64,500-ft^2 building at the Oak Ridge National Laboratory (ORNL). The HTML houses six "user centers," which are clusters of specialized equipment designed for specific types of properties measurements.

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

The User Centers

Materials Analysis User Center (MAUC)

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

Mechanical Characterization and Analysis User Center (MCAUC)

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

Residual Stress User Center (RSUC)

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

Diffraction User Center (DUC)

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 vacuum and up to 1500°C in air. DUC users have access to the National Synchrotron Light Source.

Thermophysical Properties User Center (TPUC)

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

Machining and Inspection Research User Center (MIRUC)

This center employs instrumented surface and cylindrical grinders to study hard material grinding on ceramics and special alloys. These dynamometer-equipped machine tools provide unique capabilities for studying grinding forces and their roles in controlling the topography and mechanical and wear properties of the resulting surfaces. Other capabilities include instruments for determining the cylindricity and circularity of axially symmetric objects. This center also uses a Silicon Graphics workstation for extensive computer modeling and graphical capabilities. Also available are an instrumented, electrochemical capable creep feed grinder system for grinding research on ceramics and composites and a coordinate measuring machine. In addition, this center contains equipment for measuring friction and wear, including fretting, rolling, and sliding.

The Programs

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

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

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

Work is performed for other branches of DOE via direct funding or through cooperative research and development agreements (CRADAs), which typically consist of a cost-sharing arrangement between 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, but not all, projects involve materials primarily related to the transportation industry. Ceramics, metal- and ceramic-matrix composites, lightweight materials such as aluminum and magnesium alloys, steels, and electronic materials have all been characterized at HTML.

HTML DIRECTOR'S REPORT

The High Temperature Materials Laboratory (HTML) User Program continued to work with industrial, academic, and governmental users this year, accepting 86 new projects and developing 50 new user agreements. The table on the following page presents the breakdown of these statistics. The figure on page 2 depicts the continued growth in user agreements and user projects. You may note that our total number of proposals is nearing 1000, and we expect to achieve this number in our first proposal review meeting of FY 2001. The large number of new agreements bodes well for the future. A list of proposals to the HTML follows this section; at the end of the report, we present a list of agreements between HTML and universities and industries, broken down by state.

Dr. Arvid Pasto serves as the Director of the HTML. Arvid holds a doctorate degree in ceramics and is a graduate of the State University of New York College of Ceramics at Alfred University.

Program highlights this year included several outstanding user projects (some of which are discussed in later sections), the annual meeting of the HTML Programs Senior Advisory Committee, the completion of a formal Multiyear Program Plan (MYPP), and finalization of a purchase agreement with JEOL for a new-generation electron microscope.

A User Forum was held in August 1999, with the primary goal of collecting information from our users about their expected future needs for materials characterization. This was followed by an off-site meeting for the HTML Program staff and the User Center leaders, where together we sifted through the information gathered at the Forum, at various conferences in which HTML staff had participated, and from direct interaction with customers. The outcome of this meeting was the development of an MYPP for HTML, which will help guide our future growth and provide input to our sponsors concerning strategic directions and our need for operating and capital resources to accomplish our mission. The MYPP was finalized and published in March 2000. It can be found on our website, along with our Annual Report for FY 1999 (http://www.ms.ornl.gov/htmlhome).

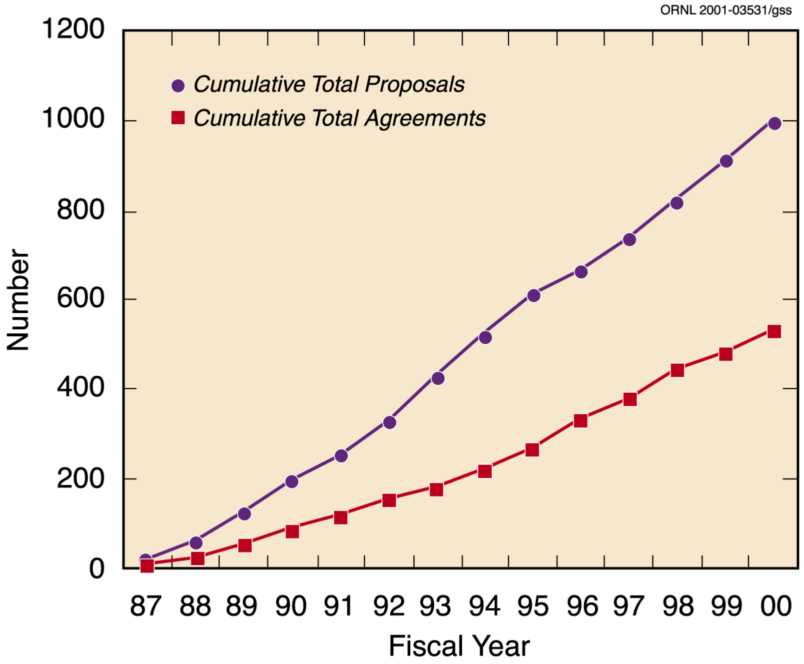
As noted in our last report, we had for several years been following the development of a spherical aberration corrector for electron microscopes. An aberration-corrected microscope would allow significant improvement in resolution, allowing information to be gathered to below 1 angstrom. As a result of the efforts of several individuals and groups, the HTML Program received $3M in FY 2000 to purchase such an instrument.

After development of an extensive specification and a request for quotation, two bids were received, and a contract was awarded to JEOL in September 2000. The new instrument will take several years to build, with delivery expected around May 2003. Additional details are provided in the Materials Analysis User Center (MAUC) section of this report.

The aberration-corrected microscope requires a facility with very low amounts of acoustic and mechanical vibration and extremely low magnetic and electric fields to achieve maximum resolution. The current HTML building will not suffice and cannot effectively be remediated, so we will pursue a new facility to house the microscope, the Aberration-Corrected Electron Microscope (ACEM), as well as several other sophisticated electron beam instruments in the Metals and Ceramics Division.

Statistics of HTML operations for FY 2000 showing user proposals and user agreements

Total	New Proposals			Total	Cumulative Proposals		
	Industrial	University	Other		Industrial	University	Other
86	32	48	6	992	419	545	28
Total	New Agreements			Total	Cumulative Agreements		
	Industrial	University	Other		Industrial	University	Other
50	38	8	4	531	321	190	20



Graph depicting continued growth in user agreements and user projects.

HTML PROPOSAL LIST

Proposal No.	Organization	Lead Center	Title	Spokesperson	Staff Contact
2000-001	Northwestern Univ. (8)	MAUC	Electron Holography of Nanostructures	Vinayak Dravid	Ted Nolan, Edgar Voelkl
2000-002	Minco (1)	MAUC	Improved MgO Performance for Sheathed Electrical Heating Element Applications	Steven White	Ted Nolan
2000-003	ORNL (09)–Chemical Technology Div.	DUC	Probing Into the Mechanism of Zeolite Crystal Nucleation and Growth from Nanoscale Gel Particles	Michael Hu	Camden Hubbard, Ted Nolan, Andrew Payzant, Larry Allard
2000-004	AdTech Nepth Inc. (3)	DUC	Study of Possible Reaction Mechanisms During the In-Situ Formation of Nd 123 Superconductors from Nepth Precursors	Shome Sinha	Camden Hubbard, Claudia Rawn
2000-005	AdTech Nepth Inc. (4)	DUC	Study of Possible Reaction Mechanisms During the In-Situ Formation of Y123 Superconductors from Nepth Precursors	Shome Sinha	Camden Hubbard, Claudia Rawn
2000-006	PCC Airfoils (2)	TPUC	Determination of Shell System Thermophysical Properties	Thomas Moreland	Camden Hubbard, Ralph Dinwiddie
2000-007	Univ. of Tennessee (65)	TPUC	Fatigue Behavior of 316 Stainless Steel Studied by Infrared Thermography	Peter Liaw	Camden Hubbard, Hsin Wang
2000-008	Univ. of South Carolina (2)	RSUC	Residual Stress Determination of Friction-Stir-Welded 2024-T3 Aluminum	Michael Sutton	Camden Hubbard, David Wang
2000-009	Univ. of Alabama–Birmingham (18)	RSUC	X-ray and Neutron Diffraction for Measurement of Bone Stress	Alan Eberhardt	Camden Hubbard, Thomas Watkins, April McMillan
2000-010	Milacron Marketing Company (2)	MIRUC	Optimizing Grinding Performance of Combination of Grinding Wheels and Grinding Fluids	Ralph Kelly	Sam McSpadden, Tom Morris
2000-011	Univ. of Tennessee (66)	TPUC	Thermographic Behavior of Haynes HR 120 Alloy During Low-Cycle Fatigue and Crack Propagation	Peter Liaw	Camden Hubbard, Hsin Wang
2000-012	Rice Univ. (04)	TPUC	Thermal Conductivity Reduction in Thermal Barrier Coatings Through the Use of Carbon Nanotubes	Enrique Barrera	Hsin Wang, Camden Hubbard
2000-013	Smith International, Inc. (1)	MCAUC	Failure of Polycrystalline Diamond (PCD) Materials for Abrasive Applications Under Static Applications Under Static and Cyclic Hertzian Contact Loading	Anthony Griffo	Kristin Breder, Peter Blau
2000-014	Huntington Alloys (1)	MAUC	Diffusivity of Cr in Various Heat-Resistant Alloys	Mark Harper	Ted Nolan, Larry Walker
2000-015	Univ. of Tennessee (67)	MAUC	Effect of Implantation Temperature and Ionizing Radiation on the Microstructure of Ion-Implanted Sapphire	Carl McHargue	Ted Nolan, Larry Allard
2000-016	NASA – Marshall Space Flight Center (1)	MCAUC	The Effects of Radiation on the Mechanical Properties of Polycrystalline Silicon and Polycrystalline Diamond Thin Films	Robert Newton	Edgar Lara-Curzio, Laura Riester
2000-017	Monofrax/Univ. of British Columbia (2)	MCAUC	Compressive Creep of Spinel and Alumina Refractories	Steven Winder	Edgar Lara-Curzio, Andy Wereszczak
2000-018	Alfred Univ. (15)	MAUC	Oxidation of CVD Diamond at Low-Oxygen Pressure	Linda Jones	Ted Nolan, Dave Braski
2000-019	Northwestern Univ. (09)	DUC	X-ray Scattering Studies of Metal/Perovskite Oxide Interfaces	Alexander Kazimirov	Camden Hubbard
2000-020	ORNL (10)–Metals And Ceramics	DUC	Control of Thermal Expansion Anisotropy in Ternary Mo5Si3 Intermetallics	Joachim Schneibel	Camden Hubbard, Claudia Rawn
2000-021	Milacron, Inc. (3)	MAUC	Accurate Determination of Diamond Concentration in Metal Matrices	Ralph Kelly	Ted Nolan, Larry Walker, Sam McSpadden
2000-023	Kansas State Univ. (4)		Bulk-Aluminum-Nitride Crystal Growth by Vapor Transport	James Edgar	Ted Nolan, David Braski
2000-053	Ohio Univ. (1)	MAUC	Electron-Energy-Loss Spectroscopy and TEM of Amorphous Wide-Band Gap Semiconductors	David Ingram	Ted Nolan
2000-022	North Carolina State Univ. (19)	MIRUC	Wear and Temperature Measurement in SiC and Diamond Scratching of Zirconia	Albert Shih	Sam McSpadden, Peter Blau
2000-024	PCC Enterprises (1)	MAUC	Microscopy of Inorganic Membranes	David Johanns	Ted Nolan
2000-025	Univ. of Louisville (1)	DUC	Time-Resolved Diffraction of Dehydration Reactions in Hydroxides	George Lager	Camden Hubbard, Claudia Rawn, Andrew Payzant
2000-026	Huntington Alloys (2)	MAUC	Cyclic Oxidation Resistance of Incotherm® Alloy G	Shailesh Patel	Ted Nolan, Larry Walker

HTML PROPOSAL LIST (cont.)

2000-027	ORNL (12) – Chemical Technology	MAUC	Synthesis of Organically Modified Molecular Sieves	Sheng Dai	Camden Hubbard, Claudia Rawn
2000-028	Univ. of Nevada–Reno (2)	DUC	High Temperature X-Ray Diffraction Studies on NH4NO3-KNO3 Solid Solutions (Materials Used in Automobile Air Bag Gas Generators)	Dhanesh Chandra	Camden Hubbard, Claudia Rawn
2000-029	NASA Glenn Research Center (8)	DUC	Identification of Phases in Ti-A1-O Alloys at 1300 to1600°K	Nathan Jacobson	Camden Hubbard, Claudia Rawn
2000-030	Clemson Univ. (19)	MCAUC	Effects of Materials Inhomogeneity and Nanomechanical Properties on Polymeric Wear	Martine Laberge	Edgar Lara-Curzio, Laura Riester
2000-031	Northwestern Univ. (10)	RSUC	Neutron Diffraction Measurement of Thermal Mismatch Stresses in Directionally Solidified Eutectic Oxide Composites	Vinayak Dravid	Camden Hubbard
2000-032	Vanderbilt Univ. (14)	MAUC	Characterization of Pt Films Deposited Using Ion-Beam-Assisted Chemical Vapor Deposition	Bridget Rogers	Dave Braski, Ted Nolan
2000-033	LANL (3)	RSUC	Residual Stresses in a PIGMA Welded Be-Ring	Mark Bourke	Camden Hubbard, Steve Spooner
2000-034	Dow Corning Corp. (11)	MAUC	Structural and Interface Properties of Low-k Dielectric Thin Films	Wei Chen	Ted Nolan, Larry Allard
2000-035	Univ. of California–SB/Univ. of California–Santa Cruz (1)	TPUC	Thin Film Microcoolers	Ali Shakouri	
2000-036	Ohio State Univ. (4)	MCAUC	Carbon-MEMS	Marc Madou	Edgar Lara-Curzio, Laura Riester
2000-037	Univ. of Tennessee (68)	RSUC	Neutron and X-ray Detections of Shape-Memory Alloy-Reinforced Aluminum Metal- Matrix Composites Subjected to Fatigue	Peter Liaw	Camden Hubbard, David Wang, Tom Ely
2000-038	Poco Graphite/Dallas Optical Systems (1)	MCAUC	Development of a Rapid, Cost-Effective Silicon-Carbide Component Fabrication Technique for Precision/Ultra-Precision Applications	Abuagela Rashed	Edgar Lara-Curzio, Sam McSpadden, Matt Ferber, Ralph Dinwiddie
2000-039	Ohio State Univ. (5)	MCAUC	Reliability and Durability Testing of Ceramic Gas Sensors (Carbon Monoxide)	S. Akbar	Edgar Lara-Curzio, Laura Riester, Peter Blau
2000-040	Univ. of Tennessee (69) (UT Space Inst.)	MAUC	Scanning Auger Microprobe	Mary Helen McCay	Ted Nolan, David Braski
2000-041	Howmet Research Corporation (3)	MAUC	Effect of EB-PVD TBC Deposition Conditions on Alumina Phase Constituents	Kenneth Murphy	Ted Nolan, Karren More
2000-042	Caterpillar, Inc. (12)	MAUC	Development of Advanced Durable Lean NOx Catalysts for Diesel-Engine Aftertreatment	Michael Ready	Ted Nolan, Karren More
2000-043	Honeywell Engines and Systems (1)	MCAUC	Fast Fracture Behavior of Ceramic Tubes	L. Portolese	Edgar Lara-Curzio, Ted Nolan
2000-044	Goal Line Environmental Technology (1)	MAUC	Evaluation of the Deactivation Mechanism of a Sulfur-Exposed Nox Trap Through Microstructure Characterization	Gregory Wagner	Ted Nolan
2000-045	ASEC Manufacturing (1)	MAUC	Investigation of Soot Deposition Mechanism In a Filter by Means of TEM Analysis	Michel Molinier	Ted Nolan
2000-046	Georgia Institute of Technology (35)	MCAUC	Low Dielectric Constant Porous Spin-on-Glass for Microelectronic Applications	Paul Kohl	Edgar Lara-Curzio, Laura Riester
2000-047	W. L. Gore & Associates, Inc. (2)	MAUC	Microscopic Analysis of Polymer-Electrolyte Fuel-Cell Membrane Electrode Assemblies	Simon Cleghorn	Ted Nolan, David Stinton, Douglas Blom
2000-048	Cummins Engine Company (18)	RSUC	Measurement of Retained Austenite in Diesel-Engine Components Using XRD Whole-Pattern Fitting	Roger England	Camden Hubbard, Thomas Watkins
2000-049	Caterpillar, Inc. (13)	MCAUC	Characterization of Elevated Temperature Mechanical Properties of Mg-Zro2 and Zirconia Toughened Mullite (ZTM)	Mark Andrews	Edgar Lara-Curzio, Matt Ferber
2000-050	Caterpillar, Inc. (14)	MCAUC	Characterization of Tribological Films on Silicon Nitride for Ceramic Valve-Train Applications	Phil McCluskey	Edgar Lara-Curzio, Hua-Tay Lin, Andy Wereszczak
2000-051	Polytechnic Univ. (2)	MCAUC	High-Temperature Deformation of A15 Type V-25si	Sung Whang	Edgar Lara-Curzio, Andy Wereszczak
2000-052	Minco (2)	TPUC	Improved Mgo Performance for Sheathed Electrical Heating-Element Applications	Steven White	Camden Hubbard, Hsin Wang
2000-053	Ohio Univ. (1)	MAUC	Electron Energy-Loss Spectroscopy and TEM of Amorphous Wide-Band Gap Semiconductors		

HTML PROPOSAL LIST (cont.)

2000-054	Univ. of Tennessee (70)	RSUC	X-Ray Characterization of the FCC to HCP Phase Transformation due to Temperature and/or Mechanical Deformation of Ultimet Alloy Subjected to Room and High-Temperature Fatigue, and Neutron Detection of Residual Strains in the Ultimet Alloy Resulting from Fatigue	Peter Liaw	Camden Hubbard, David Wang, Tom Ely
2000-055	Southern Illinois Univ. (04)	RSUC	Studies of Surface Layers Developed After Friction Tests	Peter Filip	Camden Hubbard, Thomas Watkins
2000-056	Fisk Univ. (1)	DUC	High-Temperature X-Ray Diffraction of AgGaTe2	Arnold Burger	Camden Hubbard, Claudia Rawn
2000-057	Georgia Institute of Technology (36)	MCAUC	An Investigation Into the Effects of Surface Integrity on Component Service Life in Hard Turning	Shreyes Melkote	Edgar Lara-Curzio
2000-058	Siemens Westinghouse Power Corp. (11)	MCAUC	Effect of Temperature on the Time Evolution of the Elastic Properties of Thermal Barrier Coatings (TBCs)	Matthias Oechsner	Edgar Lara-Curzio, Matt Ferber
2000-059	Siemens Westinghouse Power Corp. (12)	MCAUC	Evaluation of the Fracture Toughness of the Ceramic-Bond Coat Interface in TBC Systems	Matthias Oechsner	Edgar Lara-Curzio, Matt Ferber
2000-060	Ohio State Univ. (6)	RSUC	Residual-Stress Measurement of Cast A1 Engine Block Using X-Ray Diffraction	James Williams	Camden Hubbard, Thomas Watkins
2000-061	NanoTechnologies, Inc. (1)	MAUC	Assessment of Thermal Conductivity of Nano Aluminum Nitride as a Function of Exposure to Oxygen and Water Vapor	Dennis Wilson	Camden Hubbard, Ted Nolan, Edgar Lara-Curzio
2000-062	Univ. of Tennessee (71) (UT Space Institute)	TPUC	Thermophysical Property Analysis Proposed Research	Mary Helen McCay	Camden Hubbard
2000-063	Vesuvius Research (1)	MCAUC	Mechanical Characterization of Refractories	Quentin Robinson	Edgar Lara-Curzio, Matt Ferber
2000-064	Georgia Institute of Technology (37)	MCAUC	Nanoindentation Testing of Copper and Pearlite Steel Samples	Ashok Saxena	Edgar Lara-Curzio, Laura Riester
2000-065	Scientific Manufacturing Technology (1)	MIRUC	Magnetic-Abrasive Technologies and Equipment	Gennady Kremen	Sam McSpadden
2000-066	Research & Technology Corp. (01)	MCAUC	Advanced Basalt Fiber Composites for Reinforced Concrete Applications	Vladimir Brik	Edgar Lara-Curzio, Ted Nolan, Camden Hubbard
2000-067	Valv Technology/Univ. of Houston (1)	MIRUC	Effect of Grinding Parameters on the Performance of Silicon Nitride/BAS Ceramic Composite	Lorant Olasz	Sam McSpadden, Tyler Jenkins
2000-068	Univ. of Tennessee (72)	DUC	Flow and Fracture Behavior of ODS Alloy Systems	George Pharr	Camden Hubbard, Claudia Rawn, Wallace Porter
2000-069	Penn State Univ. (27)	TPUC	Effect of Nanocrystalline Grain Sizes in (0–3 mol%) Yttria-Stabilized Zirconia on the Tetragonal Phase Stability	Merrilea Mayo	Camden Hubbard, Wallace Porter, Claudia Rawn
2000-070	Texas A&M Univ. (04)	RSUC	Measurement of Texture in Heavily Deformed Copper	K. Hartwig	Camden Hubbard, Andrew Payzant
2000-071	Univ. of Tennessee (73)	RSUC	Texture Analysis in Metallic Alloys and Composites Processed Through Equal-Channel Angular Pressing	Peter Liaw	Camden Hubbard, Andrew Payzant, Grigoreta Stoica, Sean Agnew
2000-072	Univ. of Alabama–Birmingham (19)	MCAUC	Influence of Crack Path on Crack Resistance of Ceramic Matrix Composites	Burton Patterson	Edgar Lara-Curzio, Matt Ferber
2000-073	U.S. Chrome (1)/Adv. Comp. & Materials (1)	MIRUC	Tribological Characterization of Nickel SiC Composite Coatings for Aluminum Engine Cylinder Linings	John Carpenter	Peter Blau, Sam McSpadden
2000-074	Univ. of North Texas (1)	MCAUC	Nanoindentation and Wear Resistance of Cerium Oxide and Layered Silicate Coatings	Nandika D'Souza	Peter Blau, Edgar Lara-Curzio, Laura Riester, Arvid Pasto
2000-075	Louisiana State Univ. (8)	MIRUC	Correlations of Acoustic Emission Signals and Quality of Conventional and Creep-Feed Ground Alumina and Silicon Nitride Materials	Warren Liao	Sam McSpadden
2000-076	SUNY Stony Brook (1)	MAUC	Defects, Interfaces, and Boundaries in Friction Stir Weldments	Henry White	Ted Nolan
2000-077	Siemens Westinghouse Power Corp. (13)	MCAUC	Mechanical Properties of Oxide/Oxide Ceramic Matrix Composite Combustor Liner Tubes	Eric Carelli	Matt Ferber, Edgar Lara-Curzio
2000-078	Georgia Institute of Technology (38)	DUC	The Influence of Processing Conditions on Cation Ordering in Magnetic NiFe2O4 Nanoparticles: A Resonant Powder XRD	Angus Wilkinson	Jian Ming Bai, Claudia Rawn

HTML PROPOSAL LIST (cont.)

2000-079	Univ. of Tennessee (80) (UT Space Inst.)	MAUC	Scanning Auger Microprobe Analysis of Laser-Marked Surfaces	Mary Helen McCay	Dave Braski
2000-080	Motorola Energy Systems (ESG) (2)	TPUC	Thermography Application in Studying the Electronic Packaging Soldering Joint	Hossein Maleki	
2000-081	Univ. of Nevada–Reno (03)		High Temperature X-ray Diffraction Studies on Zr2Fe and (ZrHf)2Fe Hydrides for Long –Term Hydrogen/Tritium Storage	Dhanesh Chandra	
2000-082	Univ. of Central Florida (2)	TPUC	Correlation of Thermal Conductivity of TBC with Electrochemical Impedance Spectroscopy	Vimal Desai	Ralph Dinwiddie, Hsin Wang
2000-083	Rice Univ. (5)	TPUC	Application of Carbon Nanotubes and Fullerenes for Thermal Management	Enrique Barrera	Hsin Wang
2000-084	Tuskegee Univ. (6)	MAUC	Microstructural Analysis of Fractured Woven Sic/Sinc Composites Under Tensile and Fatigue Loading at Room and Elevated Temperatures	Anwarul Haque	Ted Nolan
2000-085	Univ. of Tennessee(78)	TPUC	Damage Evolution Assessment of Ultrafine-Grained Magnesium Alloys By Infrared Thermography	Peter Liaw	Hsin Wang
2000-086	Univ. of Tennessee (79)	MIRUC	Effect of Crosslinking on the Tribological Properties of Ultra-High Molecular Weight Polyethylene	Ray Buchanan	Peter Blau

1. MATERIALS ANALYSIS USER CENTER (MAUC)

Group Members
Ted Nolan, Group Leader
Cheryl Lee, Secretary

Larry Allard
Doug Blom, Postdoctoral fellow
Dave Braski
Dorothy Coffey
Bernhard Frost, University of Tennessee research associate
David Joy, ORNL–University of Tennessee distinguished scientist
Frank Kahl, Postdoctoral fellow
Karren More, Guest researcher
Edgar Voelkl
Larry Walker

The MAUC uses electron microscopy and surface chemical analysis techniques to characterize the structure and chemistry of advanced structural materials. The information obtained from these characterizations is used to elucidate the mechanisms that control material performance.

MAUC Instruments Include

- Hitachi S-800 field-emission gun (FEG) scanning electron microscope (SEM)
- Hitachi HF-2000 FEG analytical electron microscope (AEM)
- Hitachi HD-2000 FEG scanning transmission electron microscope (STEM)
- JEOL 733 electron microprobe
- Hitachi S4700 FEG SEM with energy-dispersive spectroscopy (EDS) and phase ID systems
- JEOL 4000EX transmission electron microscope (TEM)
- PHI 680 FEG scanning auger nanoprobe (SAN)
- Hitachi FB-2000 focused ion beam (FIB) micromill

Remote and Digital Microscopy

Running instruments in our laboratory via the Internet has proven very effective and beneficial to our users. Besides the benefit of a reduction in travel costs to user organizations, there is an advantage of interorganizational collaborative research.

Selected Highlights

Acquisition of an Aberration-Corrected Electron Microscope (ACEM)

An order was placed in September 2000 with JEOL USA, Inc., for the first aberration-corrected, combination scanning transmission/conventional transmission electron microscope in the nation. This STEM/TEM instrument is being built to HTML specifications by JEOL Ltd. (Akishima, Japan), in collaboration with CEOS Gmbh (Heidelberg, Germany), the developers of the first working aberration correctors in the history of electron microscopy. The instrument was purchased with $3M funding from the Office of Transportation Technologies, and is expected to be delivered in the late spring of 2003.

Aberration correctors can be used to compensate (primarily) the spherical aberration in either the objective lens or the condenser lens of the electron microscope. The latter type of corrector was chosen for the HTML instrument, to give an incident electron beam with a diameter of 0.7 angstroms (0.07nm). This will produce images recorded in scanning transmission mode, using a dark-field imaging technique that will show sub-angstrom resolution (i.e., better than an average single atom diameter). The major advantage of this technique is that it is sensitive to the presence of single atoms of a heavy metal species, as found, for example, on the surface of oxide supports in catalytic materials.

Coupled with conventional TEM images at a resolution of 1.9 angstroms (0.19nm), the new instrument should prove to be a powerful tool for characterizing nanostructured materials at the atomic level. In addition to its outstanding, revolutionary high-resolution imaging capabilities, the ACEM will also incorporate an energy filter in the imaging lens system. This filter will allow images to be recorded using electrons that have lost specific amounts of energy during interaction with elements in the thin specimen, so that the distribution of the elements can be directly imaged. An advantage of this technique also is that an “energy-loss” spectrum can be recorded with the sub-angstrom beam stopped on a single column of atoms in the structure. This will allow both identification of chemical species and determination of chemical bonding effects in a crystal on an atom-column-by-atom-column basis.

Fig. 1-1. JEOL aberration-corrected electron microscope.

The ACEM is being designed to be totally computer controlled, so it will be operated using computers in a separate room. Because it is such a high-resolution instrument, it is very sensitive to environmental effects, such as magnetic fields, temperature variations, air pressure changes, and micro-vibrations. Remote control via a computer system will remove the operator(s) from the room, thus allowing better control of the ambient conditions and permitting the instrument to routinely achieve its design specifications. The inset figure shows a digital mock-up of the new microscope, with the aberration corrector and energy filter indicated. No laboratory in the HTML provides the environmental conditions required, so ORNL will construct a new laboratory building to house the ACEM and several other sensitive microscopes.

Hitachi HD-2000 Dedicated STEM Consigned to HTML

Hitachi Scientific Instruments Co. has made available for use in the MAUC its new HD-2000 dedicated scanning transmission electron microscope. This $1.4M instrument is currently the only commercially dedicated STEM in the world, since the Vacuum Generators Company ceased production of its STEM instruments several years ago. The HD-2000 uses the same electron gun and illuminating lens system as our HF-2000, and also the same specimen goniometer stage and holders. So samples from one machine can easily be transferred to the other instrument without removal from the specimen holder.

The HD-2000 allows us to make high-resolution secondary electron images, back-scattered electron images, and both bright-field and dark-field scanning transmission images. The dark-field technique readily images in bright contrast any heavy metal species in the image, since it uses only electrons scattered in a large angle annular ring. High-atomic-number materials scatter more strongly at high angles than low-atomic-number materials, so those areas will have greater intensity in dark-field mode. The HD-2000 is advantageous to researchers in that it offers multiple techniques for delineating ultrafine particles.

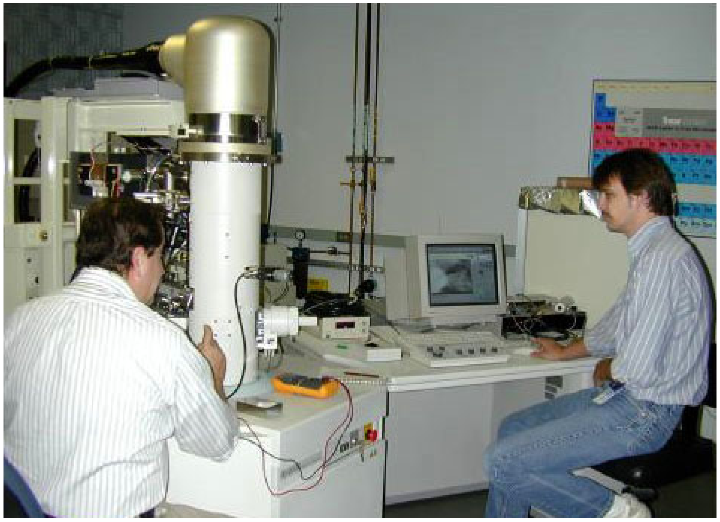


Fig. 1-2. Hitachi STEM during installation.

The second big advantage of the HD-2000 is that it has the largest collection angle for x-rays of any instrument in the world. It allows more than 20 times the collection rate for x-rays than the HF-2000, making it ideal for analysis of ultrafine particulates. For example, we have easily detected osmium in just a few small clusters of Os on MgO, which originally had only 20–30 Os atoms total.

Nucleation of AlN on 6H-SiC (0001) by a Sublimation Technique—Kansas State University

Kansas State: B. Liu, Y. Shi, J. H. Edgar
HTML: L. R. Walker

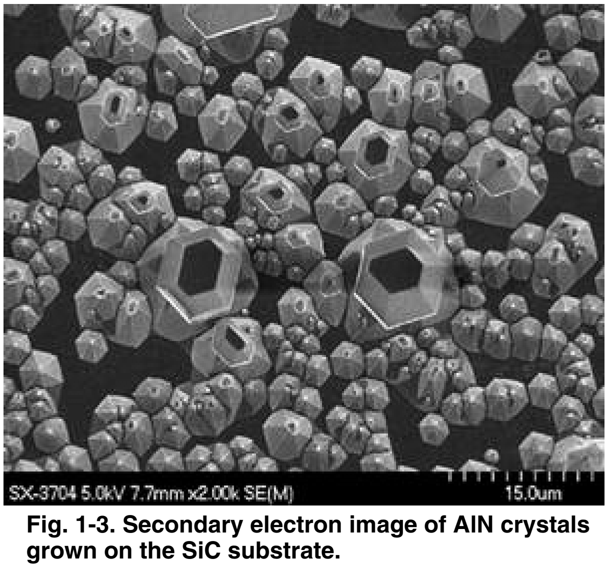


Fig. 1-3. Secondary electron image of AlN crystals grown on the SiC substrate.

The initial stage of aluminum nitride (AlN) growth on a SiC substrate by the sublimation-recondensation technique was studied to understand the origin of crystal defects in bulk AlN crystals. When SiC was used as a seed crystal, the AlN crystals did not completely coalesce, and individual grains were observed. Such crystal growth features were determined by the initial nucleation of the crystals. To determine the relative crystallographic orientations of AlN crystals and the SiC substrate, the Hitachi S-4700 SEM with back-scattered channeling pattern (BSCP) analysis was used. Channeling patterns are generated by electrons that scatter out of the sample from points deep within the crystal. These form linear parallel line pairs that have an orientation and spacing related to the crystallographic planes in the crystal.

Figure 1-3 on the previous page shows a secondary electron image of AlN crystals grown on the SiC substrate. Surprisingly, these crystals had facets that were not oriented as expected with respect to facets on the SiC substrates.

Figure 1-4 shows BSCPs of both an AlN crystal and a bare area of the SiC substrate. The patterns indicate that both the AlN and SiC hexagonal axes are parallel, but there is a misorientation of 30° between the $[11\bar{2}0]_{\mathrm{AlN}}$ and $[11\bar{2}0]_{\mathrm{SiC}}$ crystallographic planes. This result was consistent with SEM image observations and allowed the relative orientations to be precisely determined.

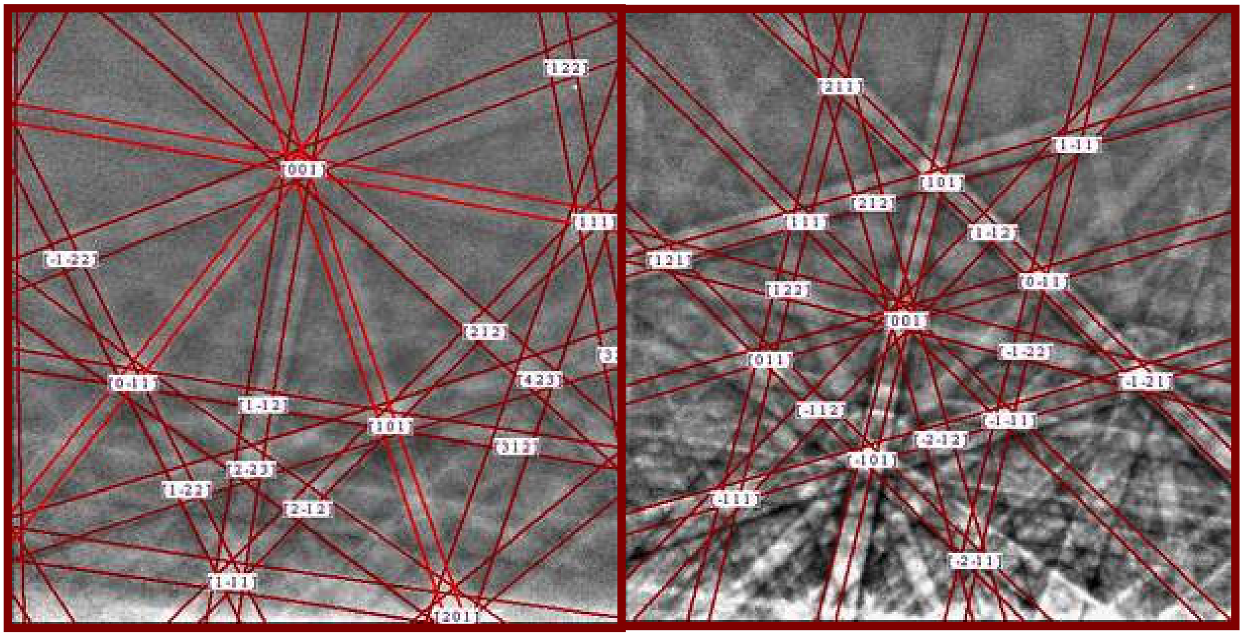


Fig. 1-4. Back-scattered channeling patterns of an AlN crystal (left) and a bare area of the SiC substrate.

Development of Ultramicrotomy as a New Technique for TEM Specimen Preparation for MAUC Users

A new specimen preparation technique was developed to allow proton exchange membrane (PEM) fuel cell membrane electrode assemblies (MEAs) to be characterized in a transmission electron microscope (TEM). MEAs consist of a proton-conducting polymer membrane (which is gas tight) sandwiched between two catalyst layers surrounded by two porous electrodes for gas diffusion and electrical conductivity. Specimens for TEM study must be extremely thin, on the order of 100 nm or less in thickness. Preparing thin specimens without disturbing the geometry and distribution of the components of an MEA is a specimen preparation challenge. In order to provide reliable information on the microstructure, the MEA must be disturbed as little as possible so we can be sure that we are characterizing the microstructure as it exists during operation.

Ultramicrotomy with a diamond was successfully used to cross-section PEM fuel cell MEAs. Diamond knife ultramicrotomy is a common technique for biological samples, but is less well known in the world of materials science. Following the development of the specimen preparation technique, a commercially available MEA was studied prior to use in a fuel cell. The first question addressed was the distribution and quantity of precious metal catalyst in the catalyst layers. The second question addressed was the relationship between the catalyst layer and the membrane. High-resolution electron microscopy provided atomic level microstructural information about the components of the MEA. Energy dispersive X-ray spectroscopy (EDX) provided chemical information about the MEA at a nanometer-level spatial resolution.

Our success with the ultramicrotomy of the fuel cell MEAs has led to the purchase of an advanced cryo-ultramicrotome for the HTML. The cryo option allows for sectioning materials at near liquid nitrogen temperatures. Cryo-sectioning offers several advantages for soft materials such as polymers and biological tissue. The extremely cold temperatures reduce the plasticity of the materials allowing for thinner sections with fewer sectioning artifacts. With the purchase of the cryo-ultramicrotome, the MAUC TEM specimen preparation ability is enhanced for several types of materials systems such as porous materials, ductile materials, polymers, and biological tissue.

2. MACHINING AND INSPECTION RESEARCH USER CENTER (MIRUC)

MIRUC Group Members
Sam McSpadden, Group Leader
Paula Miller, Secretary

Tyler Jenkins
Lawrence O'Rourke
Randy Parten
Earl Shelton
Peter Blau (Tribology)
Ronald Ott (Tribology)

The MIRUC provides basic facilities for the investigation of grinding processes for high-performance ceramic materials, design and fabrication of mechanical-property test specimens, dimensional characterization of test specimens and other components, and tribology.

Several types of numerically controlled grinders are available to guest researchers for MIRUC projects. The grinders were selected for their similarity to those used in manufacturing facilities throughout the United States. Grinders are instrumented to permit real-time measurement of key grinding-process parameters (including grinding forces, spindle horsepower, spindle vibration, acoustic emission, and coolant temperature). Data can be collected, displayed, stored, and analyzed using specialized Labview programs and other analysis software.

MIRUC Grinding Instruments Include

- Chand-Kare grindability test system
- Cincinnati Milacron Sabre multi-axis grinder
- Harig surface grinders
- Instrumented Nicco creep-feed grinder
- Instrumented Weldon cylindrical grinder
- Trihedral tripod grinder

Inspection Instruments Include

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

Physical testing and material analysis constitute a major portion of the work in the tribology laboratory. Experiments are designed to screen materials, effect simulations of components, or study the basic relationships between the microstructures and compositions of surfaces and their friction and wear behavior. Available machines fall into three categories: (1) commercially developed testing machines, (2) machines designed under subcontract, and (3) machines designed and built by ORNL for special purposes. Most of the testing machines are primarily for studying sliding wear, but abrasive-wear, impact-wear, and rolling-contact-wear tests are also available. The user center also offers the capabilities of tribology testing at high temperatures and in controlled atmospheres.

Friction and Wear Instruments Include

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

Selected Highlights

Optimizing the Performance of a Combination of Grinding Wheels and Grinding Fluids—Milacron, Inc.

Milacron, Inc.: M. K. Krueger, S. C. Yoon, D. Gong
HTML: S. B. McSpadden, Jr., L. J. O'Rourke, R. J. Parten

Metal manufacturing industries have a growing interest in finding economical ways to improve grinding productivity, part quality, and production cost. Milacron, Inc. worked with the MIRUC to evaluate the performance of several of its metalworking consumable products. The study extensively investigated the effects of the various types of grinding wheels, grinding fluids, and different combinations thereof on grinding ratio (G-ratio), specific energy (U), grinding efficiency (E), and surface roughness (R_a). Type 52100 tool steel was chosen as the work piece material for the studies. The wheels and coolants were evaluated over a wide range of specific material-removal rates. Figure 2-1 shows a typical grinding operation in progress. As a result of this study, the following findings were made:


Figure 2-1. The Weldon cylindrical grinder is used to test combinations of coolants and grinding wheels by grinding Type 52100 steel specimens.

- The specific material removal rate (Q') can be increased by more than 100% by using sol-gel wheels with metalworking fluids containing a high concentration of extreme pressure (EP) lubricants instead of conventional aluminum oxide wheels with fluids that do not contain EP lubricants.
- The sol-gel grinding wheel generates a G-ratio eight to ten times higher than that generated by a traditional aluminum oxide wheel.

- Metalworking fluids with two levels of EP lubricants were compared with a fluid that did not contain EP lubricants. The fluid with the highest EP lubricant level gives G-ratios six to nine times higher than the fluid without EP lubricants.
- The combination of the sol-gel wheel with the fluid containing the highest level of EP lubricants results in a G-ratio 56 times higher than that of the conventional aluminum oxide wheel with the fluids that do not contain EP lubricants.
- The grinding efficiency (E) remains constant as Q' increases with the combination of sol-gel grinding wheels and both of the EP-lubricant-containing grinding fluids. All other combinations of wheels and fluids demonstrated the expected trend of decreasing E as Q' increased.
- Based on a comparison of G-ratio, specific energy (U), and specific material removal rate (Q'), the total productivity improvement of sol-gel wheels with the fluid containing the highest level of EP lubricants is 130 times better than that of the conventional aluminum oxide wheel and grinding fluid with no EP lubricant.
- Using the same combination of wheel and fluid with high levels of EP lubricants, the surface finish of the ground parts is better than that obtained with other wheel/fluid combinations. As Q' increases, the rate of surface roughness increase is less with the sol-gel wheel and fluids containing high levels of EP lubricants than those obtained with other wheel-fluid combinations.

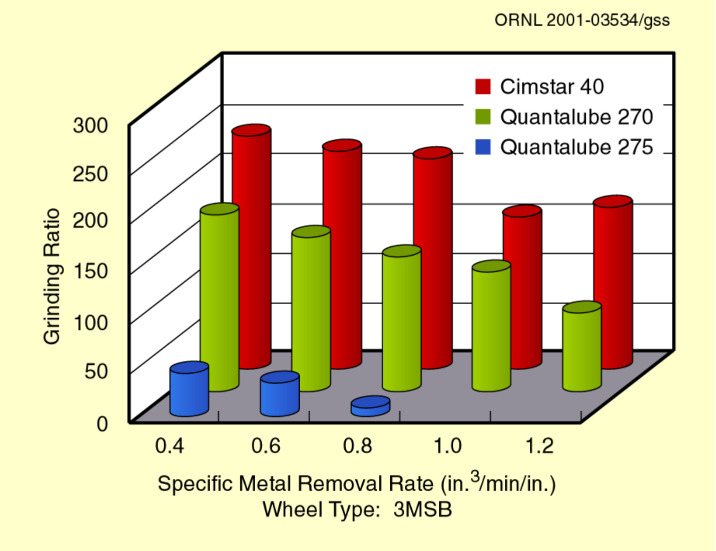


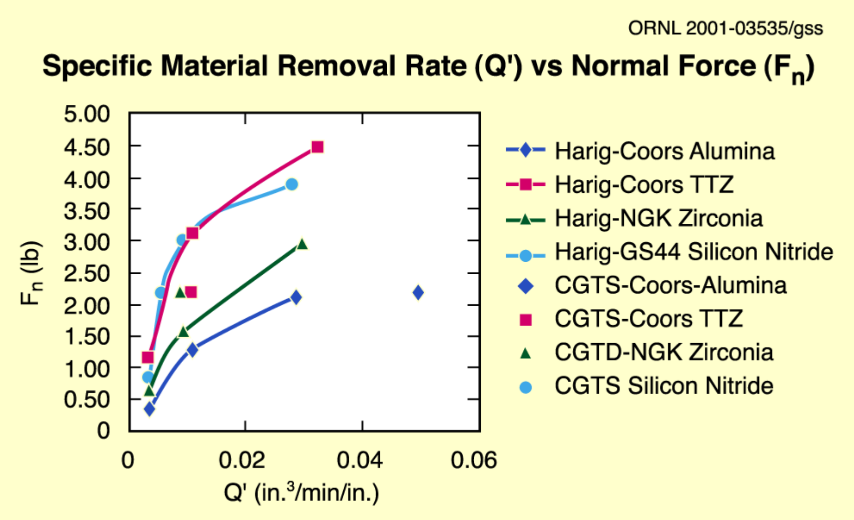
Fig. 2-2. Chart showing relationships among specific material removal rate, grinding ratio, and coolant type for a specific grinding wheel type.

Figure 2-2 is a three-dimensional chart showing the relationships among specific material removal rate, grinding ratio, and coolant type for a specific grinding wheel type. It can be seen that the grinding ratio increases as material removal rate decreases, regardless of the type of coolant used. Similar charts were developed to show the effect of wheel type on grinding ratio.

Study to Correlate Grindability Index With Surface Grinding Performance

The Compact Grindability Test System (CGTS) is designed to quickly determine the relative grindability of ceramic materials in terms of a grindability index. The grindability index can be easily converted to a specific material removal rate, which is a more generally used measure of how easily material can be removed from a workpiece. If the grindability index is to have any practical significance, it must accurately predict material removal rates under real-world grinding conditions. Initial work involved the testing of four materials on the CGTS to accurately determine their grindability indices. These same materials were then tested on the instrumented Harig surface grinder under conditions that closely approximated those present on the CGTS.

The CGTS operates at a constant normal force of 10 N. Because the Harig grinder is set up to operate in English units rather than SI, the CGTS force value was converted (10 N = ~2.2 lb_f). Figure 2-3 summarizes the comparison of the CGTS to a traditional surface grinding process on the Harig grinder. By locating the single point in the graph that represents the predicted Q' and moving along the abscissa to the intersection of the curve representing the Harig data, one can see that there is reasonably good correlation between the two sets of data for three of the four materials. The Harig grinder proved to be very difficult to control throughout the experiment. It has severe limitations as a research instrument because the table speed cannot be accurately set or maintained due to the speed variations inherent to the hydraulic drive system. A modern surface grinder is desperately needed to remedy this problem. Plans are to pursue leasing such a grinder next fiscal year if funds are available.


ORNL 2001-03535/gss

Specific Material Removal Rate (Q') vs Normal Force (F_n)

Fig. 2-3. Graph showing specific material removal rate versus normal force.

Portable Coolant System Placed in Service

The MIRUC received approximately $18,000 of pollution prevention/reduction funding, which was used to purchase a portable coolant tank with an integrated temperature control system. Whenever an experiment is conducted involving different types of coolant, the system can be moved to the appropriate grinding machine and quickly attached, in place of the larger coolant system normally used with the machine. Use of the system has greatly reduced the volume of coolant required to conduct experiments, in some cases by as much as 80%.

High-Speed, Portable Data Acquisition System Installed

This year, the MIRUC obtained a portable data-acquisition system designed by ControLink Systems, LLC. This system provides a cost-effective way to keep our data-collection hardware and software current by avoiding unnecessary duplication of instrumentation and computer hardware and software. The portable system is on a roll-around cart and consists of a Windows-based computer, a high-speed data-acquisition card, and updated data-collection software. Existing instrumentation was mounted in a sturdy metal cabinet alongside the grinders in each of four grinding labs. The cabinet contains interface cables to existing fixed data acquisition systems as well as to the new portable system. Therefore, the existing computer systems and data-acquisition software can be used independently of, or in conjunction with, the new portable system.

3. MECHANICAL CHARACTERIZATION AND ANALYSIS USER CENTER (MCAUC)

MCAUC Group Members
Edgar Lara-Curzio, Group Leader
Paula Miller, Secretary

Tim Kirkland
Ken Liu
Ralph Martin
Laura Riester
Christopher Stevens
Robert Swindeman

The MCAUC specializes in the mechanical characterization of a wide variety of materials and components (e.g., electronic materials, refractories, porous materials, metals and alloys, monolithic ceramics, polymers, metal and ceramic matrix composites, and thermal and environmental barrier coatings). Members of MCAUC perform mechanical testing and analysis; are involved in the development of standarized test methods and design codes; and conduct finite-element and life-prediction analyses for monolithic ceramics, structural alloys, ceramic composites, and ceramic coatings. Numerous mechanical test frames are available to visiting researchers from industry and academia to conduct uniaxial and multiaxial mechanical tests (tension, compression, flexure, shear, torsion, internal pressurization, rotary bending) and micromechanical tests. The MCAUC has developed unique facilities to perform tests in controlled environments and at elevated temperatures using standard and customized specimens.

MCAUC Instruments Include

- Uniaxial test facility
- Electromechanical test systems
- Servohydraulic test systems
- Ultrahigh-frequency test system
- Facilities for long-term testing
- Flexure test facility
- Multiaxial test facility
- Servohydraulic axial/torsional test systems with capabilities for internal pressurization of tubular components
- Environmental test facility
- Wide array of furnaces, heating sources, and environmental chambers for testing in inert and simulated industrial environments
- Micromechanical test facility
- Nanoindenter
- Hardness tester
- Push-out tester
- Universal micromechanical test system
- Resonant ultrasound spectrometer
- Ultrasonic modulus system
- Thermal shock facility
- Failure effects analysis/reliability analysis

Selected Highlights

The Effects of Hard-Turning Surface Integrity on the Mechanical Fatigue Resistance of Components—Georgia Institute of Technology

Georgia Institute of Technology: S. N. Melkote, S. R. Smith
MCAUC: K. Liu, L. Riester, C. Stevens, E. Lara-Curzio

Hard turning is defined as the machining of materials with a hardness of 45 Rockwell-C (HRC) or greater. The hardness of the material generates extremely high specific forces in the contact area between the tool and the workpiece, triggering thermal and mechanical mechanisms that influence the surface of the part. The advent of ceramic and cubic boron nitride tooling has made hard turning a viable machining option, but for this process to gain acceptance as a finishing process, it must be shown that it produces surfaces that meet the same quality standards as grinding.

Fig. 3-1. Georgia Tech grad student Steve Smith working with MCAUC's Laura Riester using HTML's nanoindenter.

Professor S. N. Melkote and Ph.D. student Stephen R. Smith of the G. W. Woodruff School of Mechanical Engineering at the Georgia Institute of Technology worked with MCAUC researchers Ken Liu, Laura Riester, Chris Stevens, and Edgar Lara-Curzio to study the relationship between surface integrity generated by finishing processes and component service life.

Specifically, fatigue testing was utilized to determine the impact of hard turning as compared to the traditional finishing process of grinding. Test specimens used in the experiments were generated under carefully controlled manufacturing processes and included five distinct surface conditions: hard-turned with a continuous white layer on the surface, hard-turned with no white layer, ground, and hard-turned and ground specimens subsequently superfinished to improve surface finish. In addition, the specimen's surface condition was thoroughly characterized through surface topography mapping, metallographic inspection, residual stress measurement, transmission electron microscopic analysis, and nanoindentation hardness measurements.

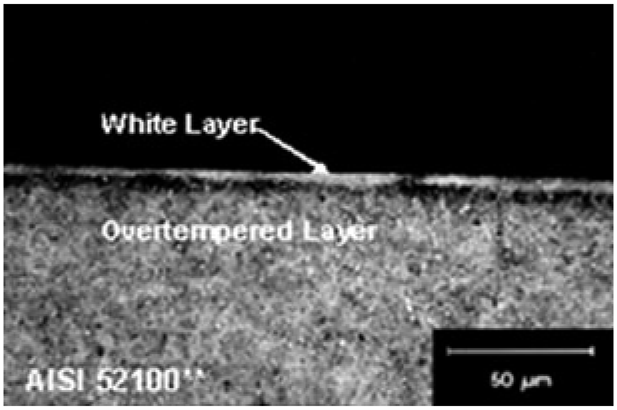


Fig. 3-2. Scanning electron micrograph of hard-turned steel showing structure of white layer.

It was found that the presence of white layers generated when using tooling with limited wear did not affect the fatigue life of the specimens. Furthermore, it was found that while the phase and crystallographic structure of the white layer is identical to the bulk material, the grains within the white layer have undergone significant refinement. Nanoindentation data showed that the white layer is harder than the base metal.

Mechanical Properties of Ceramic Matrix Composites for Gas Turbine Engines—Siemens-Westinghouse

Siemens-Westinghouse: E. Carelli
MCAUC: E. Lara-Curzio, R. Parten

The power generation industry has been under increasing pressure to reduce NO_x emissions from gas turbine engines while keeping up with market demands for increased power output and efficiency. These goals can be achieved in part through reductions in the amount of film cooling of combustor liners and turbine airfoils with attendant increases in the turbine inlet temperature.

In current gas turbine engines (Fig. 3-3), many of the superalloy-based components are operating near their upper use temperature, thereby precluding significant engine temperature increase using these alloys. To meet future environmental and performance standards, it is anticipated that the targeted temperature increases in turbine components will be accomplished through the use of continuous fiber-reinforced ceramic composites (CFCCs). Among the various CFCCs that have been developed to date, the ones that have attracted the greatest attention within the power generation industry in the past few years are those made from all-oxide constituents. The main advantage of all-oxide CFCCs over non-oxide ones (e.g., SiC/SiC) is their superior resistance to oxidation under typical turbine engine conditions.

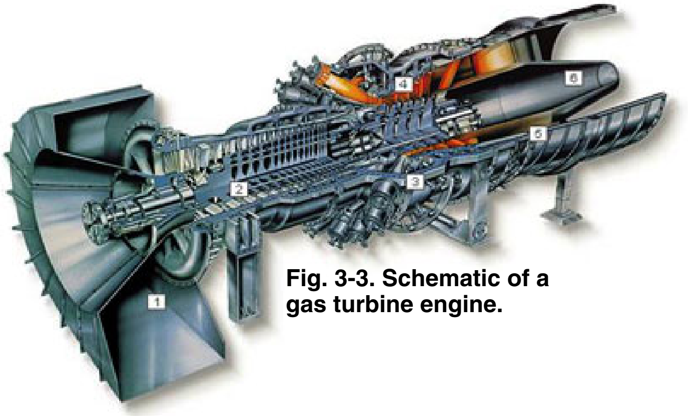


Fig. 3-3. Schematic of a gas turbine engine.

Fig. 3-4. Siemens-Westinghouse engineer Eric Carelli preparing the section of an all-oxide combustor liner for an internal pressurization test.

Researchers at Siemens-Westinghouse Science & Technology Center, Pittsburgh, Pennsylvania, are working with COI Ceramics, Inc., of San Diego, California, to develop all-oxide ceramic matrix composite combustor liners based on Nextel™ 720 fibers and aluminosilicate and alumina matrices for large gas-fired turbine engines. Siemens-Westinghouse engineer Eric Carelli (Fig. 3-4) is collaborating with MCAUC researchers Edgar Lara-Curzio and Randy Parten to evaluate the mechanical properties of 190-mm-diam combustor liners using a test method authored by Lara-Curzio that is in the process of becoming an American Society for Testing and Materials (ASTM) test standard. The results from this collaboration have helped identify the optimum fiber architecture and to determine the magnitude of manufacturing-induced residual stresses.

Mechanical Characterization of Low-Dielectric Constant Silica Films for Microelectronic Applications—Georgia Institute of Technology

Georgia Institute of Technology: P. Kohl, A. Padovani
MCAUC: L. Riester

The National Technology Roadmap for Semiconductors calls for the synthesis and preproduction of low-dielectric-constant (low-k) insulators for integrated circuit (IC) interconnections in the 1999 (k = 2.5–3.0) to 2006 (k = 1.5) timeframe. The need for low-k materials originates from the shrinkage in transistor area, creating signal propagation delays, cross-talk noise, power dissipation due to resistance-capacitance (RC) coupling and an increase in the number of interconnection levels.

Numerous materials are being investigated to replace SiO_2, which is currently being used for ICs (k~4.2). The incorporation of air (or other gases) into the dielectric material is a promising approach to achieving ultralow-k insulators, and nanoporous silica films have attracted considerable interest because the material is similar to the silica films currently used (i.e., precursors used in deposition, chemical processing, and physical properties). Thus, low-k performance can be achieved while retaining some of the silica-based properties and infrastructure. Dr. Paul Kohl and graduate student Agnes Padovani (Fig. 3-5) of the Chemical Engineering Department at the Georgia Institute of Technology are working on the development, characterization, and optimization of the properties of porous, low-k materials based on methylsilsesquioxane.

Fig. 3-5. Georgia Tech student Agnes Padovani operating HTML's nanoindenter.

The introduction of porosity at the nanometer scale has produced dramatic improvements in the electrical, optical, and mechanical properties of these films. Working with MCAUC researcher Laura Riester, Kohl, and Padovani have demonstrated, by using HTML's nanoindenter, that these films exhibit significant improvement in their fracture toughness (resistance to cracking) with increasing void fraction (Fig. 3-6).

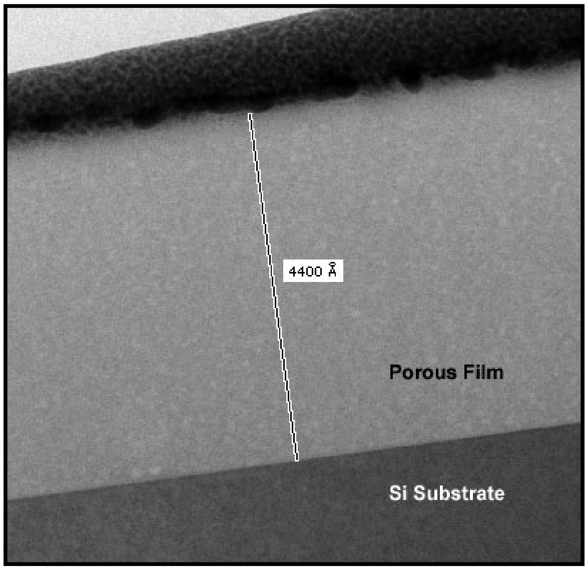


Fig. 3-6. Transmission electron micrograph of low-dielectric constant porous glass film.

Strength and Time-Dependent Deformation of Porous Alumina at Elevated Temperatures—University of Virginia Temperatures—University of Virginia

University of Virginia: D. M. Elzey, V. H. Hammond
ORNL: E. Lara-Curzio, T. Kirkland, M. K. Ferber

There is a class of fiber-reinforced ceramic matrix composites that derives its damage tolerance from a highly porous matrix, precluding the need for an interphase at the fiber-matrix interface. Although the efficacy of this material concept in enabling damage tolerance has been demonstrated, it remains to be established whether the matrix pore structure is stable against sintering and whether the desirable damage-tolerant characteristics can be retained for extended periods at the targeted service temperatures.

Professor Dana M. Elzey and graduate student Vincent H. Hammond from the Department of Materials Science at the University of Virginia worked with ORNL researchers Edgar Lara-Curzio, Tim Kirkland, and Matt Ferber to characterize the high-temperature properties and time-dependent deformation of porous alumina for ceramic matrix composite applications. Mechanical characterization of the porous alumina samples included fast-fracture flexural strength measurements as a function of test temperature and porosity. The materials studied had density values in the range of 52 to 70%; sample density was controlled by varying the sintering temperature used during processing (Fig. 3-7). Two important trends were found: for a given test temperature, the fracture strength increases as the sample's density increases, and for samples of a given density, a significant reduction in the flexural strength was observed as the test temperature was increased.

Examination of representative fracture surfaces by scanning electron microscopy revealed an increase in the number and size of planar facets present on particle surfaces as the density of the sample increased. These facets correlate to the degree of bonding that occurs between particles during sintering. The stronger interparticle bonding observed in the higher-density samples resulted in the increased fracture strength.

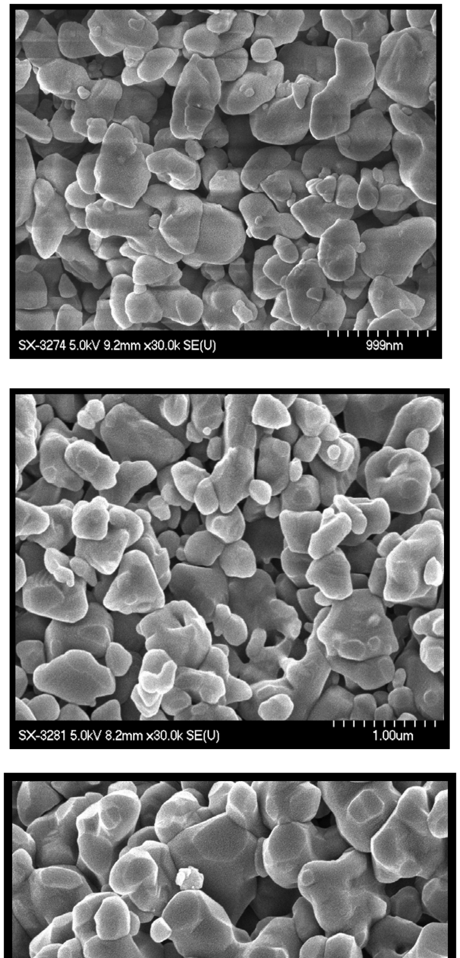


Fig. 3-7. Scanning electron micrographs of porous alumina microstructures with low (top photo), intermediate (middle photo), and high (bottom photo)

Micromechanical Properties of Knee Implants—Clemson University
Clemson University: M. LaBerge, S. Ho
ORNL: L. Riester

A total joint replacement (TJR) consists mainly of a CoCr alloy component articulating against ultrahigh molecular weight polyethylene. One of the major concerns relating to long-term clinical performance of these implants is the production of wear particles. Although the bulk form of the material is compatible, wear debris produced with polyethylene components have been shown to induce osteolysis, thus contributing to implant loosening.


Fig. 3-8. Clemson grad student Sunita Ho; inset: knee replacement components.

Professor Martine LaBerge and graduate student Sunita Ho (Fig. 3-8) of Clemson University worked with MCAUC researcher Laura Riester to understand polymeric wear of TJRs and to quantify the nanomechanical properties of a polymeric-bearing material as a function of the nanotopography at the buried interface of the contacting surfaces. Preliminary results yielded values of 2.29 ± 0.1 GPa for the elastic modulus along the surface of the compression molded tibial ultrahigh molecular weight polyethylene insert and hardness values of 0.07 ± 0.01 GPa. It is expected that these results will contribute toward the development of better and more durable TJRs.

Low-Cost Silicon Carbide for Mirrors—POCO Graphite, Inc./Dallas Optical Systems, Inc.
POCO Graphite, Inc./Dallas Optical Systems, Inc.: J. Casstevens, and A. Rashed
ORNL: E. Lara-Curzio, T. Kirkland, M. K. Ferber

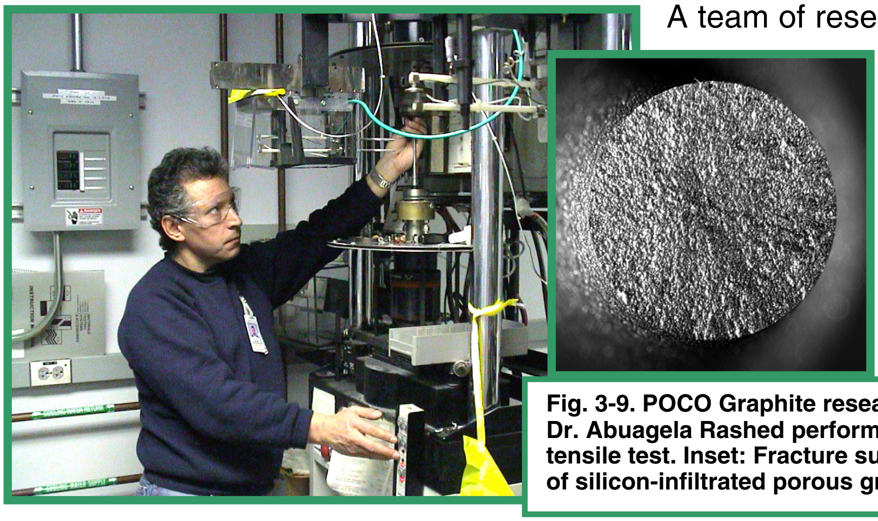


Fig. 3-9. POCO Graphite researcher Dr. Abuagela Rashed performing a tensile test. Inset: Fracture surface of silicon-infiltrated porous graphite.

A team of researchers from POCO Graphite, Inc., of Decatur, Texas, and Dallas Optical Systems, Inc., of Rockwall, Texas, led by Mr. John Casstevens and Abuagela Rashed (Fig. 3-9) worked with HTML MCAUC researchers Edgar Lara-Curzio, Tim Kirkland, and ORNL researcher Matt Ferber to support the development of a process intended to fabricate high-precision silicon carbide components at a reduced cost. To address the difficulty and cost of shaping silicon carbide, a specially formulated and processed graphite made by POCO Graphite is converted completely to silicon carbide (SUPERSiC™) following precision machining of the graphite. One of the immediate applications of this process is the manufacture of large mirrors.

Extensive work was carried out to measure the thermal and mechanical properties of the silicon carbide material at various points in the mirror-making process. Measurements have confirmed that the uniformity of the grain and pore size of the graphite material and the resulting converted silicon carbide have a large effect on the mechanical properties of the final material. Because the converted silicon carbide that was tested has approximately 20% porosity (see Table 3-1), it is expected that mechanical and thermal properties of the material will improve significantly when the porosity is filled by silicon infiltration.

Table 3-1. Summary of tensile and flexural results for SUPERSiC™ (20% porosity)

Tensile Strength	132 ± 4.93 MPa
Weibull modulus (tensile)	23.37
Young's modulus	252 GPa
Flexural Strength (4-pt bending)	148 ± 9.97 MPa
Weibull Modulus (flexure)	15.53

Properties of Aluminum-Intensive Automobiles—National Highway Traffic Safety Administration (NHTSA)

ORNL: C. Stevens, R. Parten, E. Lara-Curzio

To develop automobiles that triple the efficiency of today's cars without sacrificing performance, utility, cost of ownership, or safety, it will be necessary to reduce the weight of current models by as much as 40 percent. This dramatic reduction requires the use of lightweight metals, plastics, and composites and represents new challenges for designers and engineers. In collaboration with the NHTSA, ORNL researchers are developing detailed vehicle computational models to simulate the mechanical response of automotive structures in collisions, including one of an aluminum-intensive vehicle (Audi A8) (Fig. 3-10). The amount of energy and deformation involved in a collision are enormous and must be well understood to be harnessed into mechanisms that will protect vehicle occupants.

HTML researchers Chris Stevens, Randy Parten, and Edgar Lara-Curzio were responsible for determining mechanical data in uniaxial tension, compression, and shear required for modeling purposes. Several parts and components obtained directly from the Audi A8 model car that include motor mounts, shock absorbers, and body panels were evaluated.

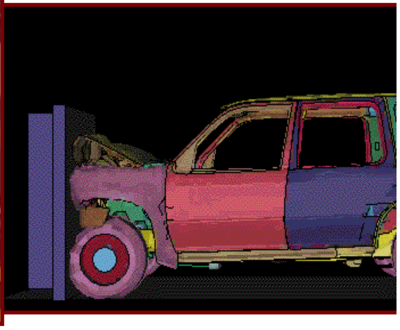


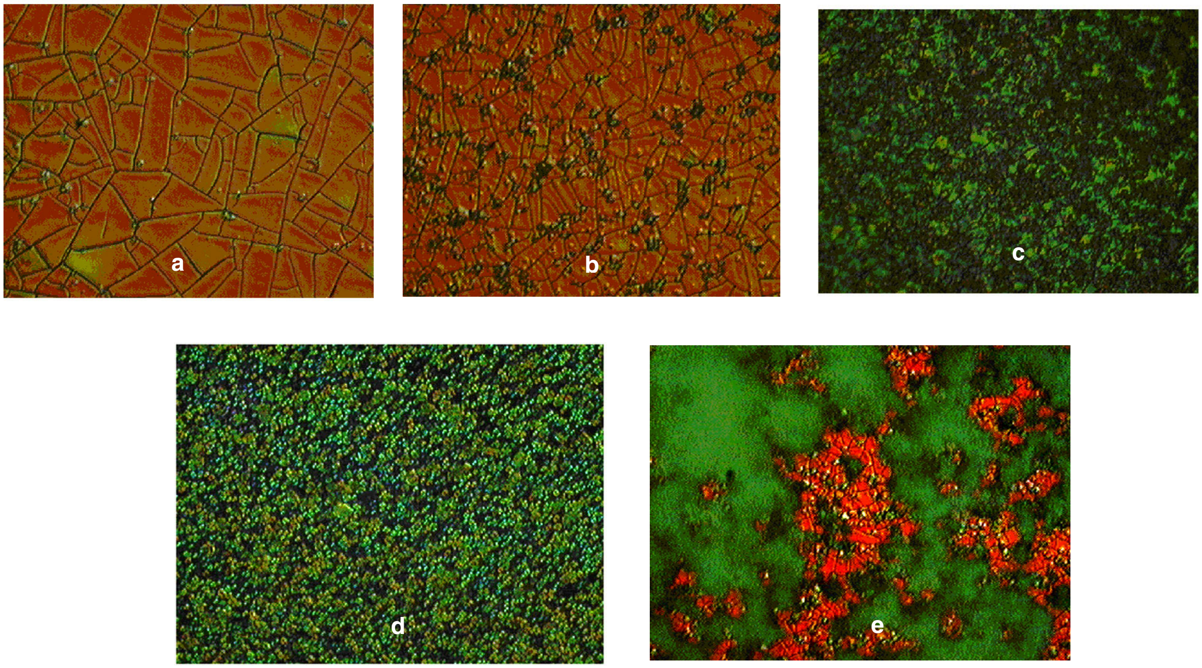
Fig. 3-10. Photo of crashed vehicle and computer simulation of crashed vehicle.

Researchers from the University of North Texas Investigate the Mechanical Properties of Electrolytic-Deposited Ceramic Coatings—University of North Texas

University of North Texas: N. A. D'Souza, and T. D. Golden
ORNL: L. Riester

Professors Nandika D'Souza and Teresa Golden from the University of North Texas worked with MCAUC researcher Laura Riester to determine the elastic properties and fracture toughness of cerium oxide (CeO_2) coatings deposited onto stainless steel by electrolytic deposition. Cerium oxide coatings are being investigated because they offer excellent protection against corrosion for several metals and because CeO_2 is a key component in three-way catalysts for the treatment of automobile exhaust gases. Electrolytic deposition has aroused considerable interest in the development of nanostructured ceramic laminates and in electronic applications because the low processing temperature is important for film integration in different devices.

Poor strength and toughness often limit the functionality of cerium oxide films, and one objective of the research of Professors D'Souza and Golden is to improve these properties. Their approach has been to incorporate platelet reinforcements to improve the strength and toughness of cerium oxide coatings.



Figs. 3-11a–e. Optical micrographs (800× magnification) of electrodeposited films: (a) cerium oxide on stainless steel; (b) cerium oxide/untreated nanoclay (1%) deposited by potential control; (c) cerium oxide/untreated nanoclay (3%) deposited by potential control; (d) cerium oxide/treated nanoclay (5%) deposited by potential control; and (e) cerium oxide/untreated nanoclay (5%) deposited by current control.

Nanoindentation measurements on electrodeposited films of cerium oxide and cerium oxide/nanoclay composites indicated that these had an elastic modulus of 62 GPa and hardness of 1.2 GPa. By comparison, cerium oxide/nanoclay composite films proved very difficult to measure because of their roughness. It was also found that addition of larger amounts of nanoclay to the solution gave a rougher, mottled appearance to the films and lowered hardness values (Fig. 3-11).

NASA Researcher Visits HTML to Measure the Mechanical Properties of Diamond Films for MEMS Applications—National Aeronautics and Space Administration

NASA: R. Newton
ORNL: L. Riester

The characterization of microelectromechanical system (MEMS) components and substrates is an area that has attracted great interest. While novel MEMS-based products continue to be designed, there are still many fundamental materials issues that require in-depth study. For example, for MEMS devices that will operate in outer space or in radiation environments, it is essential to determine the effects of irradiation on the physical and mechanical properties of these devices. Polycrystalline silicon (polySilicon) is the most commonly used material for MEMS device fabrication. However, due to its superior mechanical properties, polycrystalline diamond (polyDiamond) could be a more suitable substrate for MEMS devices designed for operation in radiation environments. NASA Marshall Space Flight Center's engineer Robbie Newton worked with MCAUC researcher Laura Riester to determine the hardness and Young's Modulus of irradiated polycrystalline silicon and diamond using nanoindention techniques.

The materials studied consisted of single-crystal silicon wafers coated with either a 15-μm-thick film of polySilicon deposited by low-pressure chemical vapor deposition (LPCVD) or a 10-μm-thick film of polycrystalline diamond. The latter were grown at Vanderbilt's microelectronics fabrication facilities by microwave plasma-assisted chemical vapor deposition (MPACVD). Both the polySilicon and polyDiamond films were undoped.

The selection of the film thickness for both materials was based on Transport of Ions in Matter (TRIM) calculations, which indicated that for the energy range to be studied, protons are adequately stopped by films of these thicknesses (i.e., protons of the same energy penetrate more than twice the depth in silicon as in diamond). Representative samples were taken from the bulk wafers and placed in the radiation exposure facility located at NASA's Marshall Space Flight Center. Both the polySilicon and polyDiamond were irradiated with 700-keV protons and 1-MeV electrons with a flux of ~1 nA/cm^2. The samples, along with nonirradiated control samples, were characterized using a nanoindenter fitted with a Berkovich indenter tip.

Cross-sectional indentation measurements on polyDiamond coatings revealed a hardness value of 89.9 GPa for the virgin material and 90.1 GPa for the irradiated sample, while the elastic modulus was found to be 1155 GPa and 1207 GPa, respectively. For the polySilicon coatings, hardness values of 11 GPa for the virgin material and 11.5 for the irradiated specimen were obtained, whereas significant changes were observed in the Young's modulus from 178 GPa for the nonirradiated material to a value of 149 GPa for the radiated material.

MCAUC Honors and Awards

HTML's Edgar Lara-Curzio participated in the prestigious National Academy of Engineering's "Frontiers of Engineering" Symposium

MCAUC's group leader, Edgar Lara-Curzio was invited to participate in the prestigious National Academy of Engineering's (NAE's) Sixth Annual Symposium "Frontiers of

Engineering" held at the Academy's Beckman Center in Irvine, California, September 14–16, 2000. The goal of the Frontiers of Engineering symposium is to bring together outstanding young leaders of the various engineering disciplines. According to NAE, "…the convening of top-notch people from diverse fields and challenging them to think about the developments at the frontiers of areas different from their own could lead to a variety of desirable results, including collaborative work, the transfer of new techniques and approaches across fields, and the establishment of contacts among the next generation of engineering leaders." The total number of participants at the symposium was 100 engineers, none older than 45, from industry, universities, and national laboratories.

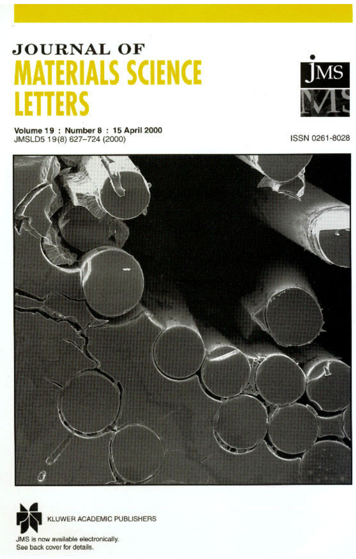
Lara-Curzio delivers presentation at the 10th Iketani Conference

MCAUC's Edgar Lara-Curzio gave an invited presentation entitled "Evolution of Fiber Microstructure and Its Effect on the Time-Dependent Deformation and Stress-Rupture Behavior of Oxide/Oxide Composites" at the 10th Iketani Conference on Materials Research held at the Karuizawa Prince Hotel, Karuizawa, Japan, June 26–30, 2000. This conference, sponsored by the Iketani Foundation, is the Japanese equivalent of the Gordon Conferences in the United States. The objectives of the Iketani Foundation are to promote science and technology, to contribute to the progress and the economy of society, and to support research of advanced materials and its related technologies.

In addition to presenting a paper, Lara-Curzio chaired a session entitled "Ceramic Matrix Composites." As part of the trip, Lara-Curzio also visited the Corporate R&D Center of UBE Industries, Ltd. in Ube City; JUTEM in Ube City; and the Institute of Advanced Energy of Kyoto University, where he gave a lecture entitled "Mechanical Properties of Oxide/Oxide Composites."

Work of Lara-Curzio featured on cover of *Journal of Materials Science Letters*

The cover of the April 15, 2000, issue of the *Journal of Materials Science Letters* shows a scanning electron micrograph that is part of the paper entitled "High-Temperature Interlaminar Shear Strength of Hi-Nicalon™ Fiber-Reinforced MI-SiC Matrix Composites with BN/SiC Fiber Coating," by MCAUC researcher Edgar Lara-Curzio and NASA-Glenn Research Center researcher M. Singh. The paper documents the micromechanical and thermochemical mechanisms responsible for the interlaminar shear failure of ceramic matrix composites.



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4. DIFFRACTION USER CENTER (DUC)

Group Members

Camden Hubbard, Group Leader
Joy Kilroy, Secretary

Jianming Bai
Burl Cavin
Andrew Payzant
Robbie Peascoe-Meisner
Claudia Rawn

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

In addition to supporting academic, industrial, and U.S. Department of Energy (DOE) laboratory users' diffraction needs, the diffraction facilities are also extensively used by qualified staff members in the Metals and Ceramics (M&C) Division, who are conducting a wide variety of ceramic and alloy research and development (R&D) efforts sponsored by DOE-Energy Efficiency and Renewable Energy (EE/RE). The DUC staff also provide technical expertise in diffraction and materials science in support of a number of DOE and Laboratory-Directed Research and Development- (LDRD-) funded projects.

DUC Instruments Include

- Two fully automated room-temperature x-ray diffractometers
- An automated high-temperature, controlled-environment x-ray diffractometer with Buehler furnace
- Neutron powder diffractometer: furnaces for 1600°C in vacuum and 1200°C in air
- Synchrotron high-flux beam line: capillary mount, Buehler furnace

In addition, an agreement has been reached to install in November 2000 a state-of-the-art Philips X'Pert Pro diffractometer with an Anton Paar ceramic furnace. This unit will have user-selectable options in detectors and x-ray optics including multilayer x-ray mirrors. The ceramic furnace will provide a uniform hot zone and sample rotation, which will complement the higher temperature and rapid heating and cooling capabilities of the Buehler strip heaters.

Selected Highlights

- phase transitions in the NH_4NO_3-KNO_3 solid solution using high-temperature x-ray diffraction (HTXRD) and differential scanning calorimetry
- thermal decomposition of zircon refractories
- thermal expansion anisotropy in ternary molybdenum silicide intermetallics
- crystal structure and thermal properties of $AgGaTe_2$

- barium titanate crystallization featured on cover of *Journal of Materials Science*
- kinetics of the reaction between fly ash and $Ca(OH)_2$ studied at the X14A beamline
- demonstration of the ability to follow complex changes in real time during superalloy heat treatment by in situ neutron diffraction methods

In addition to the user project work, DUC staff have been awarded several LDRD and Seed Money projects involving real-time, in situ studies using x-ray, synchrotron, and (particularly) neutron facilities. These projects will lead to new facilities and will lend to the unique nature and specific expertise at HTML as well as lay a foundation for cooperative use of the upgraded instruments at the High Flux Isotope Reactor (to be completed in late 2001) and the revolutionary Spallation Neutron Source (starting in 2006). Two highlights from completed work are provided. For FY01, the new LDRD projects include Claudia Rawn's role as co-investigator on "Physical and Thermodynamic Properties of Gas Clathrate Hydrates Determined by In Situ Neutron Scattering Techniques" and Andrew Payzant's role as principal investigator on "In Situ Neutron Diffraction for Characterization of Materials."

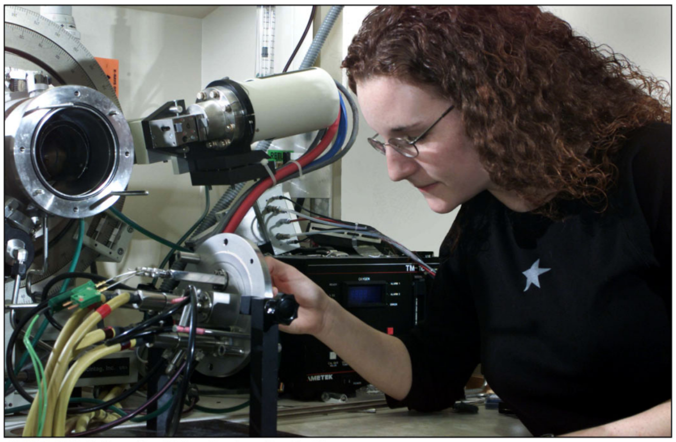
The following are selected DUC highlights for FY00.

Phase Transitions in the NH_4NO_3-KNO_3 Solid Solution Using High-Temperature X-ray Diffraction (HTXRD) and Differential Scanning Calorimetry (DSC)—University of Nevada and TRW, Inc.

University of Nevada, Reno and TRW, Inc.: D. Chandra, J. Smith
HTML: C. Rawn, W. Porter

Professor Dhanesh Chandra and his group are studying the phase equilibria of nitrate solid solutions used in automotive air-bag generators in collaboration with the Occupant Safety Systems Division of TRW Automotive. Several compositions in the NH_4NO_3-KNO_3 solid solution were examined using a combination of HTXRD and DSC. Pure ammonium nitrate (NH_4NO_3) is of great interest for use in gas generators for automobile air bag systems; however, its use is limited because of the intrinsic phase transitions that lead to macroscopic dimensional instability. In the temperature range of importance, -100˚C to +120˚C, three phase transitions cause abrupt changes in specific volume, leading to irreversible growth of cast charges. In particular, a 32˚C-phase transition causes a great deal of concern because of day-night temperature cycling in storage. Further, this phase change is associated with the hygroscopic nature of the material. In very dry NH_4NO_3, this 32˚C transition is elevated to 52˚C; thus there are issues on the role of moisture content in NH_4NO_3.

Additions of KNO_3 were tested as a likely means by which to raise the phase transition temperature. The HTXRD and DSC results show that for compositions between 6 and 50 wt % KNO_3, there are no solid-state phase transitions between 22 to 80˚C. These results suggest that $(NH_4,K)NO_3$ (ammonium potassium nitrate) solid solutions are potential candidate gas generators for air bags.



#2000-028, University of Nevada, Jennifer Smith, C0000245-01

Fig. 4-1. University of Nevada student Jennifer Smith loads a specimen for HTXRD study.

One of the users for this project, Jennifer Smith (Fig. 4-1), a senior in the Metallurgical Engineering Department at the University of Nevada, Reno, was honored at the Senior Scholar Fall 2000 Awards Banquet for her scholastic achievement at UNR. Professor Chandra, her mentor, was cited for his support allowing her to conduct HTXRD experiments at the HTML.

Thermal Decomposition of Zircon Refractories—Corning, Inc.

Corning, Inc.: H. Holland
HTML: A. Payzant

Zircon can be used to make refractory bricks for the glass industry. At elevated temperatures, it is known to dissociate into silica and zirconia, and one of the factors that can adversely affect the quality of the glass melted in zircon-lined tanks is the degree of dissociation. The thermal dissociation of purified and unpurified zircon refractory was studied by ambient and high-temperature x-ray diffraction using the unique diffraction facilities at the HTML to gain a better understanding of the high-temperature performance of zircon bricks. Diffraction data were collected on powders and sintered samples at temperatures up to 2000°C. The degree of dissociation, monitored using the intensity of the zirconia peaks, was shown to be dependent on purity, particle size, peak temperature, and time at temperature. High-temperature mass spectrometry (HTMS) revealed that the silica vaporized at elevated temperatures, leaving zirconia and, in some cases, nondissociated zircon.

Thermal Expansion Anisotropy in Ternary Molybdenum Silicide Intermetallics

ORNL: J. Schneibel
HTML: C. Rawn

Silicides based on, or containing, intermetallic compounds such as Mo_5Si_3, Mo_5SiB_2, and Mo_3Si are of great interest due to their high oxidation resistance, high creep strength, and reasonable fracture toughness. However, Mo_5Si_3 exhibits a very high thermal-expansion anisotropy, and alloys that contain Mo_5Si_3 are prone to microcracking when cooling from high temperature. Zr_5Si_3 and Ti_5Si_3 have the same crystal structure and also exhibit pronounced thermal-expansion anisotropy; however, thermal expansion studies of $Zr_3Ti_2Si_3$ reveal a very low thermal-expansion anisotropy.

To determine whether substituted Mo_5Si_3 exhibits similarly low thermal-expansion anisotropy, Dr. J. Schneibel of ORNL is studying these alloys under DOE Basic Energy Sciences (BES) funding. Substitution of 37.5 at. % of the Mo in Mo_5Si_3 by W, Ta, Nb, or Ti resulted in single-phase ternary alloys with the Mo_5Si_3-type structure. Although all alloys exhibited microcracking, the Nb-containing alloy showed a distinctly lower density of cracks than the other alloys. HTXRD has been used to study the thermal expansion of $(Mo,Nb)_5Si_3$, and the results clearly indicate that the thermal-expansion anisotropy is decreased.

Crystal Structure and Thermal Properties of AgGaTe_2 Studied—Fisk University

Fisk University: A. Burger
HTML: C. Rawn

$AgGaTe_2$ shows promise as a nonlinear optical (NLO) material for use in high-power, broadly tunable, solid-state, infrared laser systems. A reliable knowledge of the thermal properties of $AgGaTe_2$ is important not just for understanding and improving the crystal growth process but also for high-power applications as an NLO material. HTXRD data revealed the formation of at least one unidentified phase at temperatures greater than 500˚C. This result supports the theory of the formation of a Ga-rich film creating a "crust" and helps to explain processing issues such as phase instability and polyphasic growth occurring in the last-to-freeze section of ingots. The thermal expansion results were obtained from the lattice parameters at high temperatures. These results aid in designing processing procedures where the growth direction is considered to minimize mechanical stresses from expansion of the crystal along the growth direction and against the walls of the sample boat. This research was carried out in collaboration with users from the Center for Photonic Materials and Devices at Fisk University and the Air Force Research Laboratory.

Study of Barium Titanate Crystallization Featured on the Cover of *Journal of Materials Science*—Colorado School of Mines

Colorado School of Mines: G. Miller
ORNL (BES): M. Hu
HTML: C. Rawn, A. Payzant

The June 2000 cover of *Journal of Materials Science* (Volume 35) featured a micrograph from a paper describing research undertaken at HTML by Dr. Michael Hu (ORNL) and Mr. Grant Miller (Colorado School of Mines). The paper, "Homogeneous (co)precipitation of inorganic salts for synthesis of monodispersed barium titanate particles," was coauthored by HTML staff members Andrew Payzant and Claudia Rawn. Characterization of the formation of these perovskite oxides was accomplished in part using the HTML HTXRD facilities.

Kinetics of the Reaction Between Fly Ash and $Ca(OH)_2$ Studied at the X14A Beamline—Tennessee Technological University

Tennessee Technological University: J. Biernacki
HTML: J. Bai, C. Rawn

Tennessee Technological University is studying the kinetics of the alkali-activated reaction between a Class F coal fly ash and $Ca(OH)_2$ at the X14A beamline. By using synchrotron radiation, small amounts of crystalline material in an amorphous environment can be detected. The use of capillary diffraction techniques provides bulk analysis, reduces the risk of surface effects that can appear when preparing wet samples for conventional analysis, and decreases the possibility of side reactions such as carbonation of $Ca(OH)_2$ due to exposure to air. In situ investigation of a single sample over a long period was performed. The $Ca(OH)_2$ consumption was determined quantitatively by calibration against an inert TiO_2 standard. These data are being used to estimate activation energy and to develop a kinetic model for the reaction. The kinetic model will in turn be used to update existing computer codes that simulate this reaction and the similar reactions that occur when fly ash is added to portland cement.

Demonstration of the Ability to Follow Complex Changes in Real Time During Heat Treatment by In Situ Neutron Diffraction Methods

HTML: A Payzant, D. Wang
ORNL: S. Babu

Demonstration of in situ characterization of nickel-based superalloy heat treatment using neutron diffraction was the subject of an ORNL Seed Money project developed by HTML's David Wang and Andrew Payzant along with ORNL Metals and Ceramics Division's Suresh Babu. Babu and Wang collected data at the new GEM diffractometer at ISIS, Didcot, England, presently the most advanced pulsed-neutron source in the world. Diffraction spectra from an array of 3820 detectors were collected at 1-min intervals to follow the kinetic changes that occur in the alloy during heat treatment. In one experiment, the sample was heated to 1000°C and held for 5 h before being cooled to room temperature. In another experiment, the sample was heated to 1200°C to solutionize the alloy, cooled to 1000°C, held for 5 h, and then cooled to room temperature. The large quantity of collected data (500+ scans in each experiment) were subsequently analyzed using a specially developed Rietveld (whole-pattern fitting) refinement approach to determine the relative amounts of gamma-phase-matrix and gamma-prime-phase precipitates, as well as the lattice mismatch between the two phases.

The two samples behaved very differently during the 5-h, 1000°C-heat treatments (Fig. 4-2). For the first "nonequilibrium" sample (i.e., previously quenched sample reheated to 1000°C), the lattice mismatch (blue) increased with time, but the volume fractions (red) obtained from peak intensities stayed constant. However, for the "equilibrated" sample (i.e., cooled from solutionizing at 1200°C), the lattice mismatch (blue) and relative intensity of the gamma-prime peaks (red) increased as the temperature dropped from 1200 to 1000°C but then remained constant with time at 1000°C.

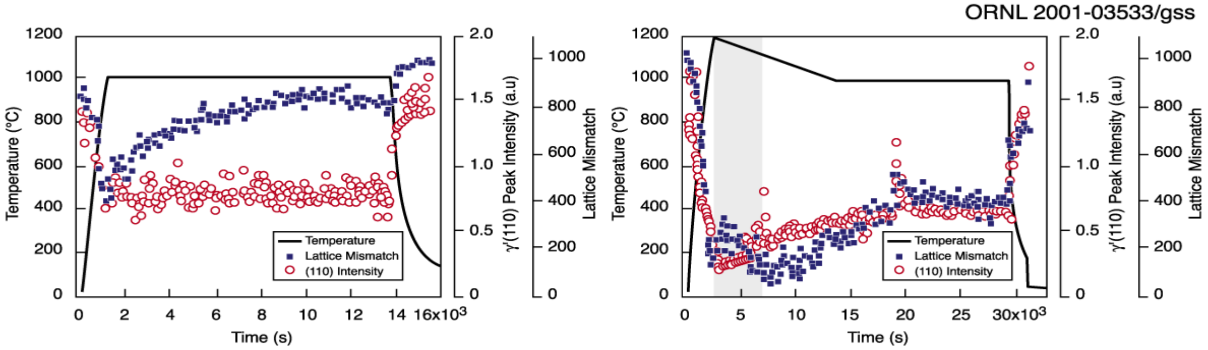

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Fig. 4-2a (left). Measured lattice mismatch between γ and γ' phases, γ' (110) peak intensity, and temperature with time for "nonequilibrium" water-quenched sample. The mismatch continues to change with time while γ' content is constant. Fig. 4-2b (right), measured lattice mismatch between γ and γ' phases, γ' (110) peak intensity, and temperature with time from as-received samples subjected to solutionizing and cooled to 1000°C ("equilibrated"). The values in the shaded area are less reliable due to limitation of sensitivity to low levels of γ' phase in that portion of the study. During aging at 1000°C, both mismatch and γ' remain constant.

These results have great significance for improved control of microstructure in advanced nickel-base superalloys subjected to different thermal processing. The ability to monitor the microstructure changes in situ as a function of heat treatment will enable the linking of diffusion processes and structural changes in these complex multicomponent alloys.

DUC Researchers Demonstrate Time-Resolved In Situ Neutron Diffraction

The neutron diffraction residual stress facility at the HFIR HB-2 spectrometer was used to demonstrate time-resolved, in situ neutron diffraction of two functional ceramics. A position-sensitive detector for rapid data acquisition was mounted inside the detector drum. A horizontal tube furnace (Fig. 4-3) was mounted on the sample axis so that the incident neutron beam passed through the furnace tube. The diffracted beams pass through the furnace tube and wall. Two materials systems were examined as "proof of principle" experiments.

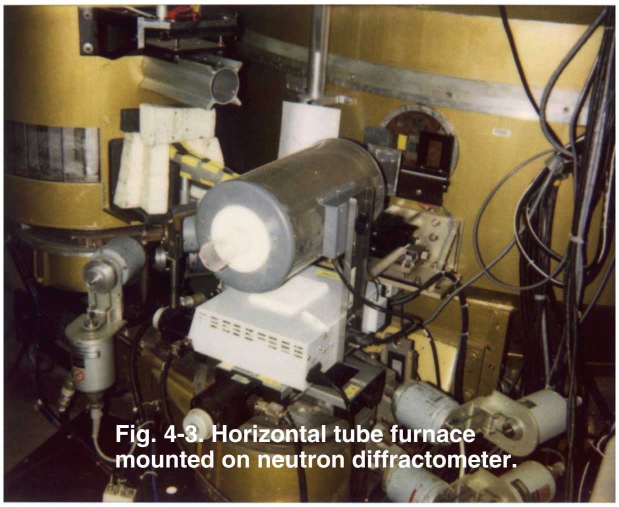

Fig. 4-3. Horizontal tube furnace mounted on neutron diffractometer.

Calcia-doped bismuth oxide, (Bi_2O_3)-10.75%(CaO), has high-oxygen ion conductivity, especially in the moderate temperature range (500–750°C); therefore it has potential applications in gas separation and fuel cell technologies. It is evident that the transformation from the single-phase high temperature (HT) structure to the two low temperature (LT) phases occurred during the 620°C data collection and was completed prior to the 570°C data collection (Fig. 4-4).

$BaCeO_3$ is an oxygen-defect perovskite that is a protonic conductor, although the mechanism of conductivity is not fully understood at this time. It is evident that a transformation from the LT structure occurred between 200°C and 840°C on heating and that the transformation was reversible, with the LT phase evident on cooling to 420°C.

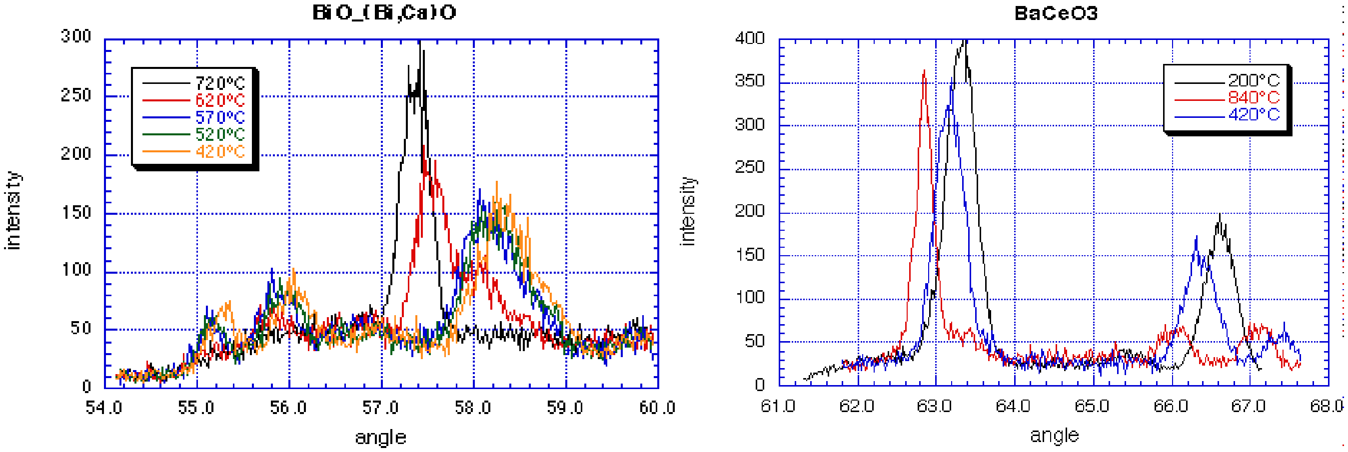

Fig. 4-4. At left: (Bi_2O_3)-10.75%(CaO) at T = 720–420°C; at right, $BaCeO_3$ at T = 850°–200°C.

5. RESIDUAL STRESS USER CENTER (RSUC)

Group Members

Camden Hubbard, Leader
Joy Kilroy, Secretary

Jianming Bai
Tom Ely
Andrew Payzant
Steve Spooner
David Wang
Thomas Watkins

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

The RSUC includes three principle measurement capabilities in three locations: the HTML x-ray residual-stress facilities, the synchrotron beamline at the National Synchrotron Light Source, and the neutron residual stress facility at the High Flux Isotope Reactor (HFIR). Unique in themselves, together, the three facilities make RSUC an unparalleled resource offering a wide range of measurement capabilities to both industry and academia. These diffraction facilities are used to measure both macro (long-range) and micro (short-range) residual stresses in polycrystalline materials.

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

RSUC Instruments Include

- 4-axis powder-texture-stress (PTS) goniometer with rotating-anode x-ray generator
- 4-axis PTS goniometer with 2-kW x-ray tubes
- Large-specimen x-ray diffraction stress analyzer
- Neutron diffraction strain-mapping facility, including remote access
- Neutron powder diffraction facility with high-temperature furnaces
- Synchrotron high-flux, highly parallel x-ray beamline with XRD furnace and cryostat

New Instruments

The Technology for Energy Corp. (TEC) large-specimen x-ray diffraction stress analyzer recently transferred from the Y-12 National Security Complex was made fully operational and now supports a number of user and DOE Energy Efficiency and Renewable Energy projects. Goals are to further increase reliability and ultimately to fully automate the system to enable stress mapping of industrial-sized specimens.

An OSMIC multilayer x-ray mirror was added to the PTS/rotating-anode system and has significantly extended the capabilities, accuracy, and throughput of this instrument. Parallel beam optics are used to enable stress measurements on highly curved surfaces (e.g. valve stems), which are impossible with conventional divergent-beam optics available in most all x-ray stress instruments. These optics also greatly improve the quality and rate of data collection during grazing-incidence x-ray stress or texture studies of thin films.

The Neutron Residual Stress Facility (NRSF) began a major upgrade in July 2000 coinciding with the beryllium reflector and beam-tube replacements at HFIR. For approximately 12 months the HFIR and NRSF will be off-line while major upgrades are under way. When completed, the NRSF should have approximately a tenfold improvement in the measurement capability it had only two years ago. Upgrades to NRSF include a new monochromator system, multiple detectors, new goniometers with expanded capacity for large and small specimens, and enhanced automation, all of which will be supported with remote collaboration tools.

Selected Highlights

The RSUC continues to address critical industrial and academic problems, typically using a combination of our facilities. Growth in use of the unique flux, energy tunability, and parallel beam characteristics of synchrotron radiation continues, as well as a strong demand for both the laboratory x-ray and neutron strain-mapping facilities. The highlights below were selected to display the scope of activities conducted in this user center and the growing use of combinations of RSUC instruments to obtain a comprehensive mapping of stress:

- Residual stresses as a function of depth in diesel engine components using all three probes—laboratory x-rays, synchrotron x-rays, and neutrons
- Texture in copper processed by the equal-channel angular-extrusion process
- Characterization of stresses in bone and hydroxyapatite coatings
- Neutron and x-ray stress mapping around journals of as-cast and heat-treated aluminum engine blocks

Residual Stresses as a Function of Depth in Diesel Components Using Neutron, Laboratory X-ray, and Synchrotron X-ray Diffraction—Cummins Engine Corp.

Cummins Engine: R. D. England (HTML Fellow)
HTML: T. R. Watkins, J. Bai

The sign, magnitude, and gradients of residual stresses are major factors affecting the fatigue life of components subjected to cyclic loading. In the characterization of two diesel-engine components, a section was removed from a connecting rod and from a finished crankshaft. These were measured nondestructively using neutron, laboratory x-ray, and synchrotron x-ray diffraction techniques. All three techniques were required to fully describe the stress gradients through thickness.

The synchrotron source identified the near-surface residual stresses, and the neutron diffraction technique measured the residual stresses from 1 mm below the surface into the interior of the samples. The strains from the synchrotron measurements were corrected for the effect of the exponentially weighted averaging over the irradiated depth using a numerical linear inversion method. The neutron measurements did not require these corrections. After the determination of the residual stress profiles, the same locations on the components were measured using the common x-ray diffraction etch (layer removal) technique. The measured data from this iterative etch technique were corrected for the effect of the removal of material on the remaining stress field using equations from the Society of Automotive Engineers (SAE) J784a standard. The corrected data from this destructive technique are compared in Fig. 5.1 with the residual stress profile data determined nondestructively, showing respectable agreement. The technique of layer removal was more accurate than expected, with the errors being within the normal variation of stress in most microstructures.

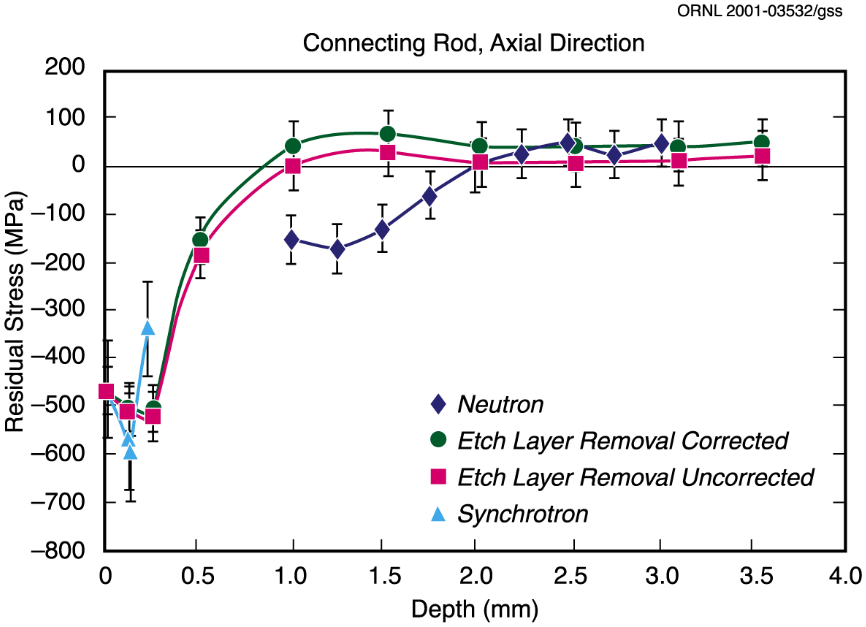

Fig. 5-1. Residual stress as a function of depth in a connecting rod.

This research was the basis for a master's thesis by Roger England, "Measurement of Residual Stresses in Diesel Components Using X-ray, Synchrotron, and Neutron Diffraction" (University of Cincinnati, Cincinnati, Ohio, August 2000).

Orientation Textures in Copper Processed by Equal-Channel Angular Extrusion (ECAE) —Texas A&M University

Texas A&M: K. Hartwig, H. Mohammed
HTML: E. A. Payzant, T. R. Watkins

Texas A&M University continued research on texture in extruded materials using the specialized facilities of RSUC. Previously tested materials included body-centered cubic iron and iron-silicon; these studies had lead to understanding of the deformation mechanisms, summarized in several presentations and publications. The latest study was on pure copper processed by ECAE with and without intermediate heat treatments, resulting in unique orientation distributions. The enhanced capabilities of the multilayer x-ray optic recently coupled to the rotating anode x-ray powder-texture-stress (PTS) diffractometer were also shown to greatly increase the rate and quality of data collected for pole figure determination.

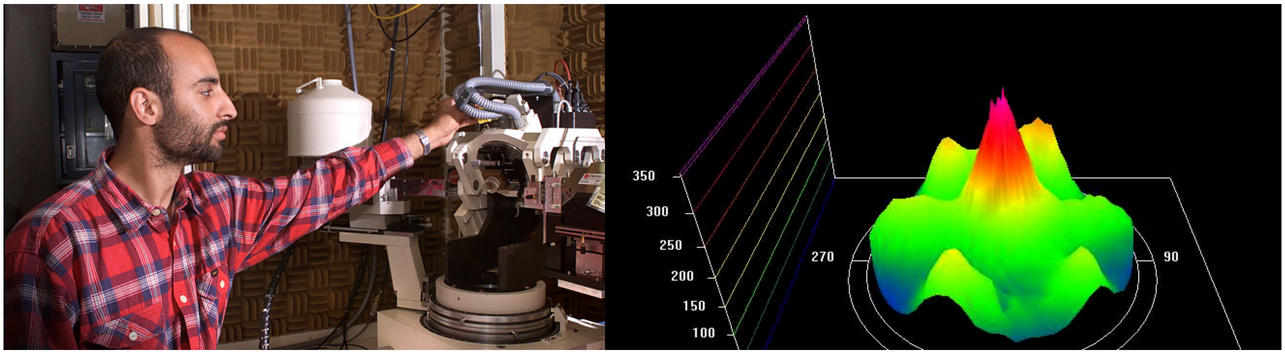

Fig. 5-2. Texas A&M's Mohammed used the PTS goniometer to measure texture in their ECAE copper specimens.

Biomaterials Characterization: Bone and Hydroxyapatite Coatings—University of Alabama

University of Alabama: A. Eberhardt, J. Haman
HTML: T. R. Watkins

Biomaterials represent an exploding field of materials research. Two researchers from the Department of Biomedical Engineering at the University of Alabama–Birmingham used RSUC facilities to investigate their biomaterials. In one project, material characterization techniques are being applied to human bone to elucidate how the living bone responds to applied loads and as a function of densification, thickening, etc. In this study, a bar taken from a human femur bone was mounted on the goniometer in four-point flexure, and the elastic response of the hydroxyapatite (HA) phase was examined using x-ray diffraction. Bone is a composite material with a complex hierarchical structure having two main components, collagen and HA. The collagen forms a fibrous matrix around discontinuous HA platelets. Surprisingly, the x-ray results indicated that the HA phase was in compression on the tensile face of the bar. A more detailed examination of the microstructure is planned to develop a model consistent with the x-ray results. This research has spawned an ORNL seed money project to develop an understanding of load transfer between the organic and inorganic phases in abalone shell, a simpler model system.

In another project, plasma-sprayed HA on titanium metal substrates was examined. This materials system is part of a larger study investigating the dissolution of materials in vivo for drug release/delivery. The residual stress, crystallite size, and preferred orientation were characterized to determine their impact on dissolution of the coatings. The magnitude of the residual stresses in the coatings is small with some in tension and others in compression.

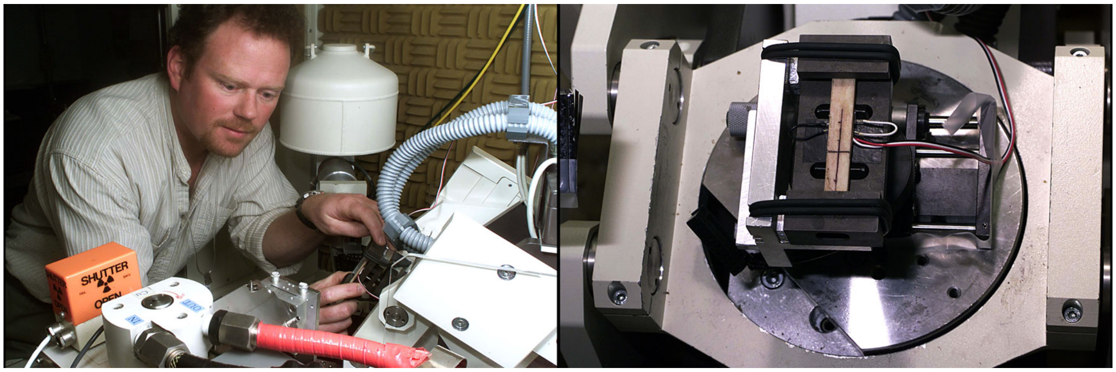

Fig. 5-3. Professor Alan Eberhardt works on a human bone specimen in four-point flexure; at right, the bone specimen is shown in flexure with a strain gage monitoring the macrostrain.

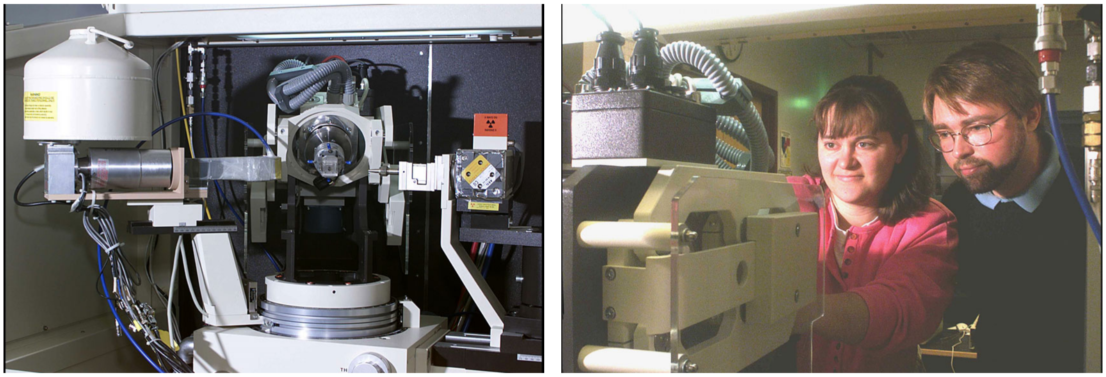

Fig. 5-4. A plasma-sprayed hydroxyapatite coating on titanium metal substrate was examined for residual stresses. Jeannie Haman and HTML's Thomas Watkins mount the next sample.

X-ray and Neutron Stress Mapping Around Journals of Die-cast Aluminum Engine Blocks—Ohio State University

Ohio State University: J. Williams, S. Raman
HTML: T. Watkins, T. Ely, S. Spooner

Two researchers from Ohio State and three from Honda of America (who sponsored the work at Ohio State and HTML) visited the RSUC to take part in discussions and experiments on characterization of residual stresses in Honda of America's engine blocks. In general, die-cast materials have residual stresses due to differential cooling.

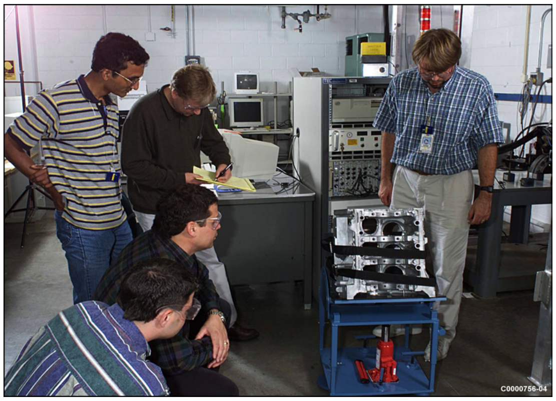

Fig. 5-5. Researchers from Ohio State, Honda of America, and ORNL examine an as-cast aluminum engine block being prepared for x-ray residual stress measurement.

Understanding these residual stresses and the impact of subsequent heat treatments is of immense interest due to distortion during machining of the journals. Here, the residual stresses within as-cast and heat-treated engine blocks were measured and mapped with both neutrons and x-rays using the neutron residual stress facility and the large specimen x-ray diffraction stress analyzer, respectively. The neutron stress mapping clearly showed a major change of one strain component around the journal portion of the engine block from tensile to compressive due to heat treating. The x-ray work showed a slight reduction of the magnitude of the compressive surface residual stress after heat treatment.

6. THERMOPHYSICAL PROPERTIES USER CENTER (TPUC)

Group Members
Camden Hubbard, Group Leader
Joy Kilroy, Secretary

Ralph Dinwiddie
William Elliott
Gerry Ludtka
Wally Porter
Hsin Wang

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

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

TPUC Instruments Include

- Differential scanning calorimeter (DSC)
- Dilatometers (bilayer, dual push rod, high temperature)
- High-mass thermogravimetry with concurrent differential thermal analysis (TG/DTA)
- IR point detectors, IR fiber optics, and IR light pipes
- Pycnometer
- Quench and deformation dilatometer
- Simultaneous thermal analysis: DTA, TG, and mass spectrometer
- Thermal conductivity 3-omega system
- Thermal conductivity lock-in IR camera
- Thermal constants hot-disk system
- Thermal diffusivity laser flash and xenon flash systems
- Thermography (IR camera)

New Instruments and Expertise

A new high-temperature DSC capable of operating to 1650°C was ordered and will be put into productive operation early in FY 2001. The system will extend the temperature range and will improve the precision of measuring heats of formation and specific heat. This new unit will also provide modern automation and data analysis tools to further improve the value to users.

The TPUC gained a Materials Measuring Corp. (MMC) quench dilatometer with high-speed gas-quench and deformation capabilities along with staff who are expert in its operation and application. This added capability is primarily used for quantitative measurement of phase-transformation properties of steels and other alloys. These quantitative data are critical to modeling heat-treat phenomena or any process involving a transient thermal cycle. Several current projects sponsored by the Metals-Processing Laboratory Users Facility (MPLUS) involve industries such as Cummins Engine Co.; Ispat Inland Steel, Inc.; and Deformation Control Technologies. The Colorado School of Mines and Ohio State University are also working collaboratively with Oak Ridge National Laboratory (ORNL) in using this capability.

Selected Highlights

The TPUC has experienced increasing demand for use of the IR camera and IR point detectors to characterize temperatures in real time in a variety of systems and as nondestructive real-time imaging devices. As we anticipate this area to continue to grow, several highlights are presented here, including the first-ever reported use of IR point detectors and IR fiber optics to measure the temperature distribution on aircraft rotating-disk brakes during simulated braking. In another project, the IR camera was used to characterize temperature rise during low-cycle fatigue studies. The areas of thermal property and thermal transport continue to be of great interest.

The DUC portion of this report presents an example of a multi-user center study of the thermal stability of NH_4NO_3-KNO_3 solid solutions. The TPUC team is known for its expertise in characterizing thermal transport properties of thermal barrier coatings. Recent work in this important area is summarized in one of the highlights that follow.

Temperature Tests on Aircraft Brakes—Advanced Friction Study Center at Southern Illinois University

Southern Illinois University: D. T. Marx
HTML: R. B. Dinwiddie, H. Wang

ORNL 2001-03536/gss

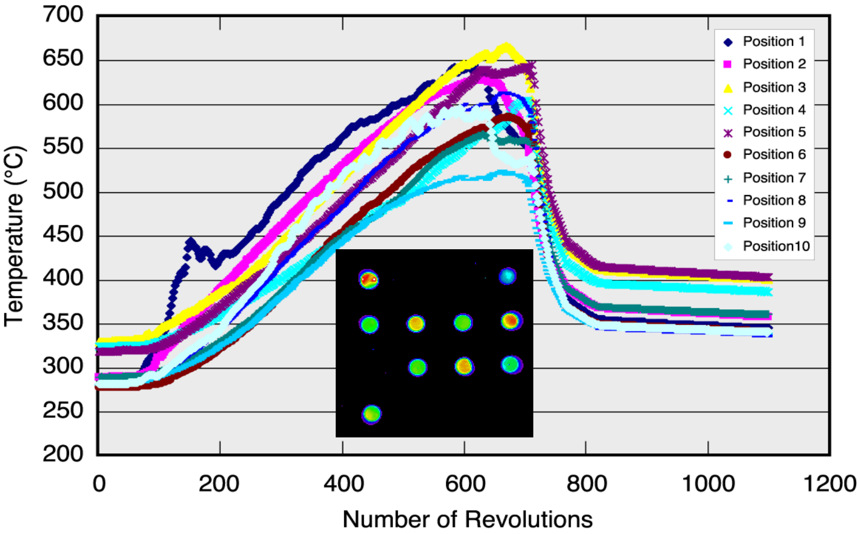

Fig. 6-1. Temperatures recorded during a simulated normal land test.

The temperatures of carbon-carbon aircraft composite brake surfaces have been calculated to reach from several hundred degrees to over 1000°C under various braking conditions. However, because the contact surfaces of the rotating and stationary disks are inaccessible, experimentally measuring the temperature is difficult. To solve this problem, TPUC worked with Amorphous Materials, Inc. to develop an IR fiber bundle containing 16 individual fibers. Ten fibers are connected through small holes drilled at different radii in the stationary disc; the other six are reserved as backups. The IR camera is synchronized with the brake dynamometer; the camera is used to monitor changes in temperature during simulated braking. Normal landing, service landing, taxi, and rejected takeoff conditions were studied. Figure 6-1 shows the temperatures recorded during a simulated normal land test. The IR imaging results matched reasonably well with the calculated temperatures and are being used to improve the models being developed at Southern Illinois University.

Publication: D. T. Marx, T. Policandriotes, J. Scott, R. B. Dinwiddie, and H. Wang, "Measurement of Interfacial Temperatures During Testing of a Subscale Aircraft Brake," *Journal of Physics D: Applied Physics*, May 2001.

Spot-Weld Qualities of Motorola Battery Packs—Motorola Co.

Motorola: H. Maleki, B. Byer
HTML: H. Wang and R. B. Dinwiddie

Motorola researchers conducted a study of spot-weld qualities of battery packs at TPUC using the IR imaging system. The Motorola battery cells are connected by metallic tabs to form battery packs. Two spot welds are usually made on each electrode. Currently, the weld quality can be checked only by using a time-consuming pull test. The IR imaging system was used to take 20 sequential images following a heat pulse provided by a xenon flash lamp. The camera was operating at 120 Hz. The properly made spot welds act as heat sinks and should appear cold in the IR images, as confirmed in Fig. 6-2, where the black areas indicate lower temperatures. This fast, nondestructive IR imaging technique was proven to identify good and bad welds within seconds. Pull tests conducted at Motorola after IR imaging studies of several battery packs matched very well with the ORNL results.

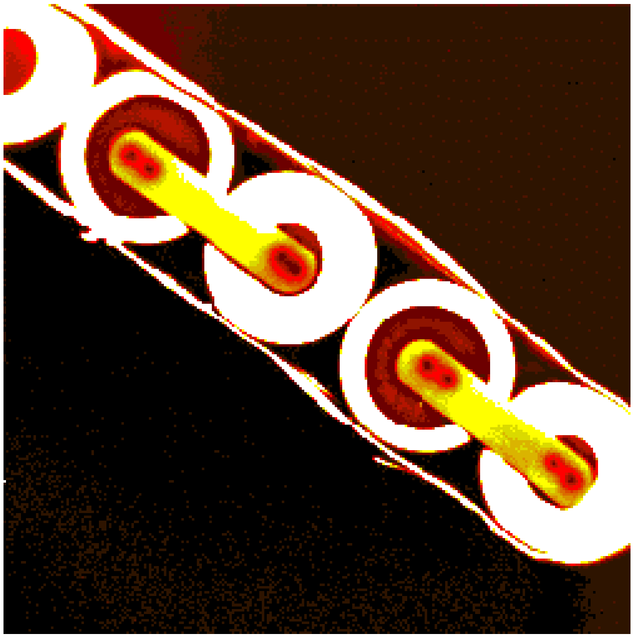

Fig. 6-2 . IR image of a battery pack after an external heat pulse. The black and red areas indicate temperatures lower than those in the yellow areas.

IR Thermography During Low-Cycle Fatigue Tests of Haynes HR-120 Alloy—University of Tennessee, Knoxville

University of Tennessee: Y. He, L. J. Chen, L. Jiang, P. K. Liaw
HTML: H. Wang

Following the success of applying IR thermography during stress-controlled, high-cycle fatigue tests, TPUC has expanded the research with the University of Tennessee to studies of strain-controlled, low-cycle fatigue tests. Because a temperature rise was expected during fatigue testing and because of the temperature dependence of mechanical properties, this project set out to quantitatively monitor the temperature during low-cycle fatigue testing. Haynes HR-120 alloy was tested. Temperature/time profiles of the alloy were recorded for different total strain ranges varying from 0.6% to 2.3% (Figs. 6-3 a and b). The temperature rise at 0.6% total strain range was only 1.5°C but rapidly increased to 120°C when the total strain range was changed to 2.3%.

A sharp temperature drop (2°C) was observed at 1.5% total strain level after 120 cycles. The effect was confirmed using high-speed imaging and tests at 1.7% total strain level. Cyclic hardening and softening of the HR-120 alloy may play an important role in this observed temperature drop.

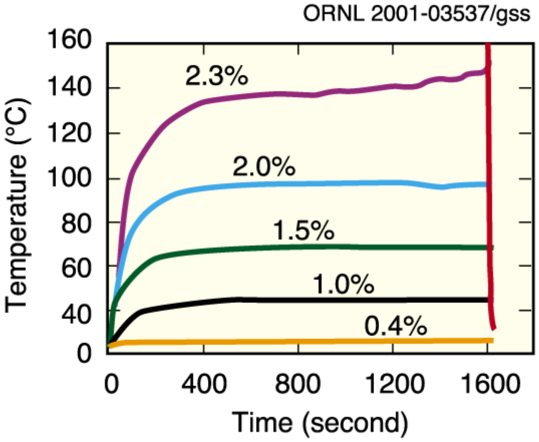

Fig. 6-3a. Temperature evolution of HR-120 specimens during five strain-controlled tests.

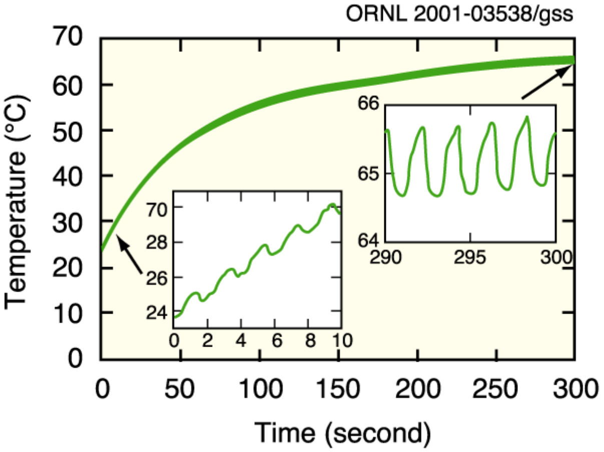

Fig. 6-3b. Temperature oscillation of an HR-120 specimen during 1.5% total-strain-level test with inserts for cycles 1–5 (left) and cycles 145–150 (right).

Thermal Properties of Zirconia Co-doped with Trivalent and Pentavalent Oxides—Pennsylvania State University

Penn State: S. Raghavan and M. J. Mayo
HTML: W. D. Porter and H. Wang

Zirconia doped with 6–8 wt % (3.2–4.2 mol %) yttria [6–8 yttria-stabilized zirconia (YSZ)], the most common thermal barrier coating material, relies mostly on oxygen vacancies to provide the phonon scattering necessary for low thermal conductivity. This study examined whether specific substitutional defects (Table 6-1) —in addition to, or instead of, oxygen vacancies—can provide similar or greater reductions in conductivity. To this end, a series of zirconia samples co-doped with varying levels of yttrium (trivalent) and tantalum/niobium (pentavalent) oxides were synthesized, thereby allowing oxygen vacancy and substitutional atom concentration to be varied independently. The Nb-Y and Ta-Y co-doped zirconia samples, containing only substitutional defects, were stable, single-phase, tetragonal materials with thermal conductivities very close to those of the conventional 6–8YSZ.

Table 6-1. Choice of Compositions

Id.	Mol% Ta_2O_5 (T)	Mol% Nb_2O_5 (N)	Mol% Y_2O_3 (Y)	Y-(N+T) (Excess Yttria)
N4	—	4.5	9	4.5
N5	—	10.0	10	0
N6	—	7.5	12	4.5
T9	4.4	—	9	4.6
T10	5.4	—	9	3.6
T13	10.0	—	10	0
T15	7.5	—	12	4.5

Figures 6-4a–6-4d show the specific heat, thermal diffusivity, and thermal conductivity of the test samples. In these samples, Nb^{+5} and Ta^{+5} are similarly effective in lowering thermal conductivity, in contradiction to phonon-scattering theories that consider primarily mass effects and thereby predict significantly greater conductivity reduction from Ta^{+5} doping than from Nb^{+5} doping. Finally, samples doped with Nb^{+5}/Ta^{+5}-Y^{+3}, which contain both oxygen vacancies and substitutional defects, were found to be unstable in single-phase form; however, the thermal conductivities of the two-phase tetragonal + cubic mixtures were as low as conventional 6–8YSZ.
Publication: S. Raghavan, H. Wang, W. D. Porter, R. B. Dinwiddie, and M. J. Mayo, "Thermal Properties of Zirconia Co-doped with Trivalent and Pentavalent Oxides," *Acta Materialia*, Vol. 49, 169–179, January 2001.

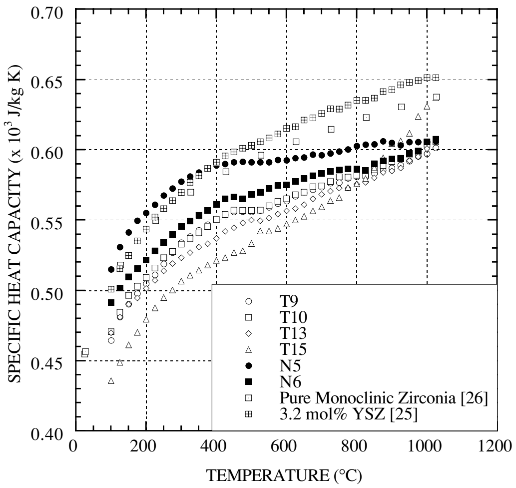

Fig. 6-4a. Specific-heat capacities of doped, co-doped, and undoped zirconias as determined using differential scanning calorimetry.

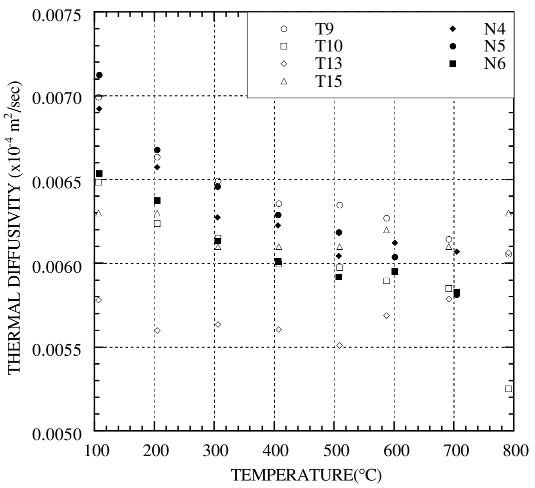

Fig. 6-4b. Thermal diffusivity vs. temperature for the co-doped zirconias. Average of three measurements on each of three samples.

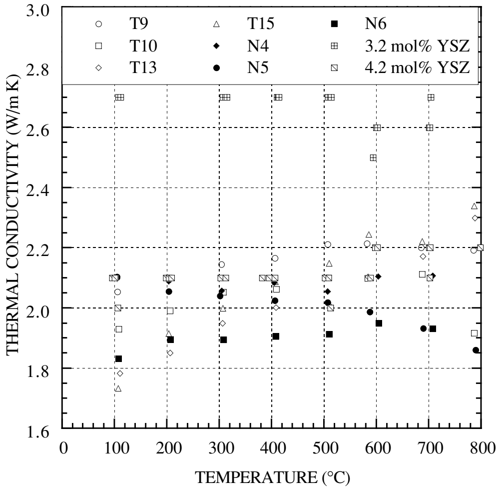

Fig. 6-4c. Thermal conductivity versus temperature for the YSZ and co-doped zirconias.

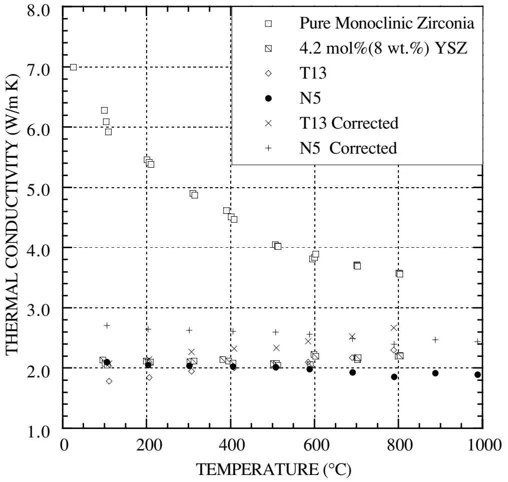

Fig. 6-4d. Comparison of the thermal conductivities of pure and 8 wt % yttria-doped zirconia with that of the co-doped zirconias containing no oxygen vacancies. Corrected data are for porosity effects.

Effect of Austenitization Hold Time and Temperature on Matrix Carbon Content and Martensite Kinetics in a 52100 Alloy Steel—Colorado School of Mines, The Torrington Company, MPLUS

Colorado School of Mines: M. T. Lusk, G. Grach, H.-J. Jou
The Torrington Company: H. Walton, D. Shick, G. Dicastanzo
TPUC: G. Ludtka, W. H. Elliott

Size change during heat treatment of hardenable steel is related in large part to the steel's phase transformation characteristics, which in turn are determined by the carbon and alloy content of the austenite before the quenching to martensite. To accurately predict the transformation behavior of the resulting austenite and related carbide size changes, it is necessary to understand, quantify, and model the kinetics of the carbide dissolution. In this work, a series of quenching dilatometer experiments was performed using a 52100 alloy steel in which the austenitizing time and temperature were varied to dissolve different amounts of carbides. Subsequent size change (dilation) upon quenching was monitored to determine the phase evolution rate and the martensite start temperature.

The influence of hold time at several austenitization temperatures is shown in Fig. 6-5. The effect of longer times is observed to move the transformation curves down and left, indicating a reduction in the martensite start and finish temperatures until all of the carbon is in solution in the parent austenite phase prior to the quenching process.

ORNL 2001-03543/gss

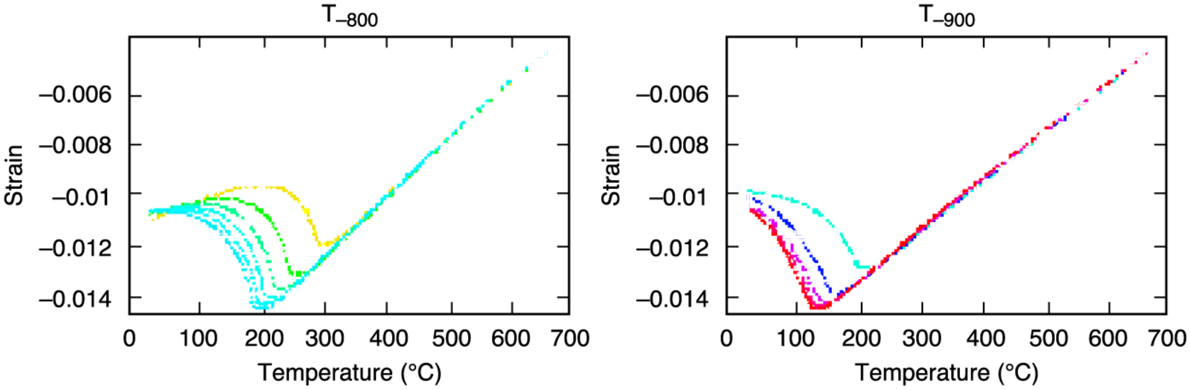

Fig. 6-5. Influence on transformation temperature and strain of hold time at several austenitization temperatures. The right-most yellow line in the 800°C plot corresponds to a 1-min hold time. The left-most blue curve corresponds to a 60-min hold time. The progression from right to left is 1, 5, 10, 15, 30, and 60 min, respectively. In the 900°C curve, the two red curves overlap. The right-most green curve corresponds to a 1-min hold time. Moving to the left, the blue curve is 5 min; the purple, 10. The remaining two overlapping red curves on the right represent 15- and 30-min hold times.

From these data, a model was developed to predict matrix carbon content as a function of austenitization hold time and temperature. This specific model was subsequently incorporated into a more generalized differential phase evolution model in the commercially available predictive heat-treatment simulation code DANTE that now predicts martensite volume fraction as a function of temperature for 52100 alloy steel during heat treatment and quenching. This effort has resulted in significantly streamlined production-heat-treatment operations, resulting in substantial heat-treatment energy and cost savings for The Torrington Company.

The experimental work was conducted on the MMC high-speed quenching dilatometer in the TPUC as part of an MPLUS User Agreement. The analysis and modeling of the dilatometer data were conducted at the Colorado School of Mines as part of this collaborative research project.

Note: Research partially sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Materials Program, under contract DE-AC05-00OR22725 with UT–Battelle, LLC, for the U.S. Department of Energy.

Standard Nonproprietary User Agreements

The next several pages comprise a listing of universities and industries that have entered into standard nonproprietary user agreements with the HTML. These include 316 industries and 157 universities from across the United States.

Standard Nonproprietary User Agreements

U. S. Industry — 316

Alabama

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

Arizona

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

California

Alzeta Corp. (Santa Clara)
Amercom Inc. (Chatsworth)
Applied Materials, Inc. (Santa Clara)
Ceradyne, Inc. (Costa Mesa)
CERCOM, Inc. (Vista)
Ensei, Inc. (Pismo Beach)
FMC Corp., (Santa Clara)
Guidance & Control Systems/ Litton Ind. (Woodland Hills)
Honeywell (AlliedSignal Inc., Ceramic Components) (Torrance)
Honeywell (AlliedSignal EMRC) (Los Angeles)
IBM Almaden Research Center (San Jose)
Lockheed Martin Skunk Works (Palmdale)
M. J. Schiff & Associates, Inc. (Upland)
Membrane Technology Research (Menlo Park)
Northrop Corp. (Pico Rivera)
Nuclear & Aerospace Materials Corp. (Poway)
Rohr Inc. (Chula Vista)
Solar Turbines, Inc. (San Diego)

California (cont.)

SRI Int'l (Menlo Park)
Sullivan Mining Corp. (San Diego)
Sundstrand Power Systems (San Diego)
Ultramet (Pacoma)
X-Ray Instrumentation Assoc. (Mountain View)

Colorado

CDM Optics, Inc. (Boulder)
Coors Ceramics Co. (Golden)
Golden Technologies Co. (Golden)
John Mansville (Littleton)
Materials Physics Research (Highlands Ranch)
Quantum Peripherals (Louisville)
Schuller Int'l Inc. (Littleton)
TDA Research Inc. (Wheat Ridge)

Connecticut

ABB C-E Services, Inc. (Windsor)
Steven Winter Associates, Inc. (Norwalk)
Torrington Co. (Torrington)
United Technologies/Pratt & Whitney (East Hartford)
U. S. Chrome Corp. (Stratford)

Delaware

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

District of Columbia

American Iron & Steel Institute
SPI/SPFD
Structural Insulated Panel Assoc.

Florida

GELTECH, Inc./LMES (Orlando)
Martin Marietta Elect. Info & Missile-LMES (Winter Garden)
National High Magnetic Field Laboratory (Tallahassee)
Pratt & Whitney (W. Palm Beach)
Siemens Westinghouse Power Corp. (Orlando)
Westinghouse Electric Corp. (W. Palm Beach)

Georgia

Advanced Engineered Materials, LLC
AMERCORD, Inc. (Lumber City)
BRH, Inc.-LMES (Lawrenceville)
Ceradyne, Inc. (Scottdale)
Ionic Atlanta, Inc. (Atlanta)
Institute of Paper Science & Tech.
ITI MOVATS-LMES (Kennesaw)
Microcoating Technologies (Chamblee)
Motorola (Lawrenceville)
RCF Seals (Vidalia)
Rolls Royce, Inc. (Atlanta)
Thermal Ceramics (Augusta)

Illinois

A. Finkl & Sons (Chicago)
Adtech Nepth, Inc. (Oak Park)
Alloy Eng. & Casting (Champagne)
Caterpillar, Inc./Tech. Ctr. (Peoria)
Honeywell (AlliedSignal) (Des Plains)
Insulating Concrete Form Association (Glenview)
Wagner Castings Co. (Decatur)

Standard Nonproprietary User Agreements (cont.)

Indiana

Allison Engine Co.
 (Indianapolis)
Cummins Engine Co.
 (Columbus)
Dana Corp. (Richmond)
Firestone Building Products
 Co. (Camel)
GM Corporation/Delco Remy
(Andersonville)
Haynes Int'l (Kokomo)
Honeywell (AlliedSignal)
 (South Bend)
Ispat Inland (East Chicago)

Kentucky

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

Louisiana

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

Maine

Surmet Corporation
 (Burlington)

Maryland

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

Massachusetts

American Superconductor
 Corp. (Westborough)
Brigham & Womens Hospital
 (Boston)
Busek Co. (Natick)
Ceramics Process Systems
 Corp. (Cambridge)
Chand Kare Tech. Ceramics
 (Worcester)
Dynamet Technology
 (Burlington)
Foster-Miller, Inc. (Waltham)
GTE Laboratories, Inc.
 (Waltham)
Hydrogen Microplasmatron
 Technologies, LLC
 (Cambridge)
JPS Elastomerics Co.
 (Holyoke)
MicroE (Natuk)
Morton Advanced Materials
 (Woburn)
Norton Co. (Northboro)
Norton/TRW Ceramics
 (Northboro)
Osram Sylvania/Univ. of
Massachusetts
 at Lowell (Lowell)
Refractory Testing Associates
 (Chestnut Hill)
Sarnafil, Inc. (Canton)
Single Ply Roofing Institute
 (SPRI) (Needham)
SSG, Inc. (Wilmington)
Textron Specialty Materials
 (Lowell)
Uniform Metal Tech. LLC
 (Watertown)

Michigan

Bosch Braking Systems
 (Farmington Hills)
Chrysler Corporation
 (Highland Park)
Detroit Diesel Corp. (Detroit)
Dow Chemical Corp. (Midland)
Dow Corning Corp. (Midland)
Duro-Last Roofing, Inc.
 (Saginaw)
Eaton Corp. (Southfield)
Energy Conversion Devices,
 Inc. (Troy)
Ford Motor Co. (Ann Arbor)

Michigan (continued)

GM AC-Rochester (Flint)
GM Powertrain Group
 (Pontiac)
GM Research & Development
 (Warren)
Hoskins Mfg. (Hamburg)
Howmet (Whitehall)
Metal Building Manufacturers
 Assoc. (Traverse City)
Metallamics Inc. (Traverse)
Modern Alloying Technologies,
 LLC (West Bloomfield)
Parker Abex NWL
 (Kalamazoo)
Thixomat (Ann Arbor)
Valenite, Inc. (Troy)

Minnesota

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

Mississippi

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

Missouri

McDonnell Douglas Corp.
 (St. Louis)
SB&TD Business Systems

Montana

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

New Hampshire

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

Standard Nonproprietary User Agreements (cont.)

New Jersey

AT&T Bell Laboratories (Murray Hill)
Ceramic Magnetics, Inc. (Fairfield)
Certech, Inc. (Wood Ridge)
Engelhard Corp. (Edison)
Exxon Research & Eng. Co. (Annadell)
Honeywell (AlliedSignal) (Morristown)
INRAD Inc. (Northvale)
Intl. Paper (Princeton)
Lucent Technologies (Murray Hill)
Materials Technology (Shrewsbury)
Mobil Technical Co. (Paulsboro)
Nanopowder Enterprises, Inc. (Piscataway)
NEC Research Inst. (Princeton)
Phone-Poulenc, Inc. (Cranbury)
Union Camp Corp. (Princeton)

New Mexico

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

New York

Advanced Refractory Tech., Inc. (Buffalo)
AKZO Nobel Chemicals, Inc. (Dobbs Ferry)
Applied Nano Metrics, Inc. (Stormville)
CDH Energy Corp. (Cazenovia)
CMP Industries, Inc. (Albany)
Carborundum (Niagara Falls)
Corning Inc. (Corning)
Eastman Kodak Co. (Rochester)
GE (Schenectady)
Monofrax, Inc. (Falcomer)
ReMaxCo Technologies, Inc. (Kenmore)

New York (cont.)

Sulzer-Metco (Westbury)
T. J. Watson Research Center (Yorktown Heights)
UK Software Services (Grand Island)
X-Ray Optical Systems (Albany)

North Carolina

Cree Research, Inc. (Durham)
MicroMet Technology, Inc. (Matthews)
Selee Corp. (Hendersonville)
Teledyne Allvac (Monroe)

Ohio

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

Pennsylvania

Advanced Technology Materials, Inc. (University Park)
AHT, Inc. (Chicora)

Pennsylvania (cont.)

Alcoa Tech. Center (Alcoa\ Center) Aluminum Co. of America (Alcoa Ctr.)
Aristech Chemical Corp. (Murrysville)
Bethlehem Steel Corp. (Bethlehem)
Calgon Corp. (Pittsburgh)
Carlisle Syntec, Inc. (Carlisle)
Certainteed Corp. (Valley Forge)
Concurrent Technologies Corp.
IBACOS (Pittsburgh)
J&L Specialty Steel (Pittsburgh)
Kennametal, Inc. (Latrobe)
Leroy A. Landers (Philadelphia)
PPG Industries, Inc. (Lancaster)
SB&TD Business Systems (Lancaster)
Thermacore, Inc. (Lancaster)
Westinghouse Science & Tech Center (Pittsburgh)

Rhode Island

Advanced Components Materials (East Greenwich)
Quadrax Corp. (Portsmouth)

Tennessee

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

Standard Nonproprietary User Agreements (cont.)

Tennessee (cont.)

Computational Mechanics Corp. (Knoxville)
Computational Systems (Knoxville)
Coors Electronic Package (Chattanooga)
CTI, Inc. (Knoxville)
DG Trim Products (Alcoa)
Eastman Kodak (Kingsport)
Eastman Chemical (Kingsport)
Environmental Engineering Group, Inc. (Knoxville)
Environment Systems Corp., LMES (Knoxville)
Forged Performance Products, Inc. (Oak Ridge)
ForMat Industries, LMES (Oak Ridge)
Gaylon’s Machine Shop (Sweetwater)
Goal Line Co. (Knoxville)
Great Lakes Research (Elizabethon)
H. R. DeSelm (Knoxville)
Herbert E. McCoy, Jr. (Clinton)
IMTech Co. (Knoxville)
IntraSpec, Inc. (Oak Ridge)
J. A. Martin (Knoxville)
Jeffrey Chain (Morristown)
Lear Corp. (Morristown)
Mahle, Inc. (Morristown)
Materials Eng., & Testing (Oak Ridge)
Microbial Insight, Inc. (Knoxville)
MINCO Acquisition Co. (Midway)
Nano Instruments, Inc. (Knoxville)
Oak Ridge Housing Authority (Oak Ridge)
Oxyrase (Knoxville)
Photogen, Inc. (Knoxville)
ReMaxCo Technologies (Kingston)
Ronald K. McConathy (Kingston)
SENES Oak Ridge, Inc. (Oak Ridge)
Smelter Service Corp. (Mt. Pleasant)
Smith & Nephew (Memphis)
Standard Aero (Maryville)
Status Technologies (Knoxville)

Tennessee (cont.)

Technology for Energy Corp. (Knoxville)
Tennessee Center for R&D (Knoxville)
Textron Specialty Materials Div.Avco (Nashville)
Third Millenium Tech., Inc. (Knoxville)
TTE Diecasting (Oak Ridge)
Vamistor Corp. (Sevierville)

Texas

Agriboard Industries (Electra)
CarboMedics, Inc. (Austin)
Dallas Optical (Decatur)
Electrospace Systems, Inc., LMES (Richardson)
Exxon Corp. (Houston)
Ludlum Measurement Inc. (Sweetwater)
POCO Graphite (Rockwall)
Robert Hageman (Austin)
Smith Intl., Inc. (Houston)
Southwest Research Institute (San Antonio)
Stone & Webster (Houston)
Texas Instruments (Dallas)
Tycom Corporation (Austin)

Utah

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

Virginia

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

Washington

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

West Virginia

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

Wisconsin

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

Other Govt. Facilities — 16

Federal Highway Administration (Virginia)
Idaho National Engineering Laboratory (Idaho Falls)
Los Alamos National Laboratory (Los Alamos)
NASA/Marshall Space Flight Center (Huntsville, Alabama)
NASA Glenn (NASA Lewis Research Ctr.) (Ohio)
NASA Langley Research Ctr. (Virginia)
National Highway Traffic Safety (DC)
Naval Post Graduate School (California)
Naval Research Lab. (DC)
NIST (Maryland)
Sandia National Laboratories (Livermore)
Space and Navel Warfare Systems Center (California)
U.S. Army Research Lab (Virginia)
U.S. Bureau of Mines (New York)
U.S. FDA (Maryland)
U.S. Naval Academy (Maryland)

UNIVERSITIES — 157

Alabama

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

Arizona

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

California

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

Colorado

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

Connecticut

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

Delaware

Univ. of Delaware (Newark)

District of Columbia

George Washington Univ.
Howard Univ.

Florida

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

Georgia

Georgia Inst. of Tech. (Atlanta)

Hawaii

Univ. of Hawaii (Honolulu)

Illinois

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

Indiana

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

Iowa

Iowa State (Ames)

Kansas

Kansas State (Manhatten)
Wichita State (Wichita)

Kentucky

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

Louisiana

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

Maine

Univ. of Maine (Orono)

Maryland

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

Massachusetts

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

Michigan

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

Standard Nonproprietary User Agreements (cont.)

Michigan (cont.)

Western Michigan Univ. (Kalamazoo)

Minnesota

Univ. of Minnesota (Minneapolis)

Mississippi

Mississippi College (Clinton)
Mississippi State (Mississippi State)

Missouri

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

Montana

Univ. of Montana (Missoula)

Nebraska

Univ. of Nebraska-Lincoln

Nevada

Univ. of Nevada, Reno

New Hampshire

Dartmouth College (Hanover)

New Jersey

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

New Mexico

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

New York

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

North Carolina

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

North Dakota

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

Ohio

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

Oklahoma

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

Oregon

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

Pennsylvania

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

Rhode Island

Brown Univ. (Providence)

South Carolina

Clemson Univ. (Clemson)
Univ. of S. Carolina (Columbia)

South Dakota

S. Dakota State (Brookings)

Tennessee

East Tennessee State Univ. (Johnson City)
Fisk Univ. (Nashville)
Jacksboro State Area Voc. School (Jacksboro)
Maryville College (Maryville)
Tennessee State (Nashville)
Tennessee Tech. Center (Knoxville)
Tennessee Tech. Univ. (Cookeville)
Univ. of Memphis (Memphis)

Standard Nonproprietary User Agreements (cont.)

Tennessee (cont.)

Univ. of Tennessee (Knoxville)
Vanderbilt Univ. (Nashville)

Texas

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

Utah

Univ. of Utah (Salt Lake)

Virginia

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

Washington

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

West Virginia

West Virginia Univ. (Morgantown)

Wisconsin

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

Publications and Presentations: October 1999–September 2000

Note: Asterisks indicate HTML staff members.

2000 Publications

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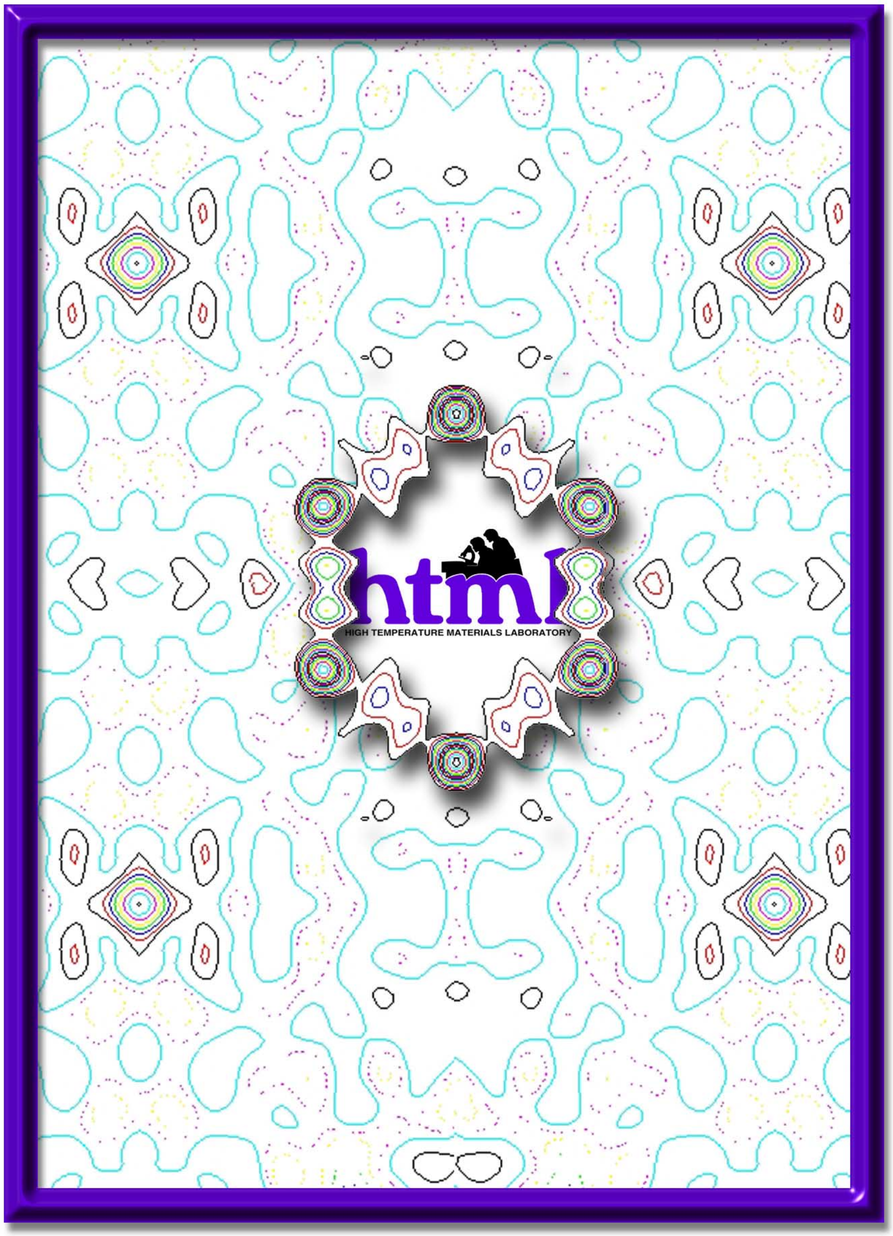
Front cover: Micrograph of a silicon carbide substrate, silicon carbide hillocks, and aluminum nitrate.

Back cover: Nuclear density map showing the locations of the oxygen and deuterium atoms in a six-member ring in the framework of a structure I gas hydrate.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