Quarterly Highlights
High Temperature Materials Laboratory
Oak Ridge National Laboratory
October – December 2007

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Diffraction User Center (DUC)

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University of Florida studies photovoltaic films by *in situ* x-ray diffraction

Research problem: To develop efficient and inexpensive processing methods for producing photovoltaic cells with polycrystalline thin films

Implications: Potential development of highly efficient and low cost photovoltaic cells

Description of Work: Copper indium/gallium selenide (CIGS) is used to make polycrystalline thin films photovoltaic cells. This material may be manufactured as a thin film by several different technologies, and their potential high efficiency and low cost make them an attractive alternative to crystalline silicon solar cells. However, the quaternary phase diagram for polycrystalline thin films is not well understood, and the equilibrium phases and reaction kinetics have not been fully explored. Therefore, graduate students Vaibhav Chaudhari and Rakesh Mahadevapuram as well as postdoctoral fellow Kyoung Kim from Dr. Tim Anderson's group at the University of Florida traveled to ORNL and worked with Andrew Payzant and Robbie Meisner to collect new data on the CIGS system, extending work undertaken through two previous HTML user proposals. The unique *in situ* high-temperature x-ray diffraction instruments at the HTML enabled quantitative analysis of time-resolved data collected as precursor thin-film materials reacted at high temperature to form the desired phases (Fig. 1). This project focused on understanding the selenium binaries Cu-Se, Ga-Se, In-Se, in

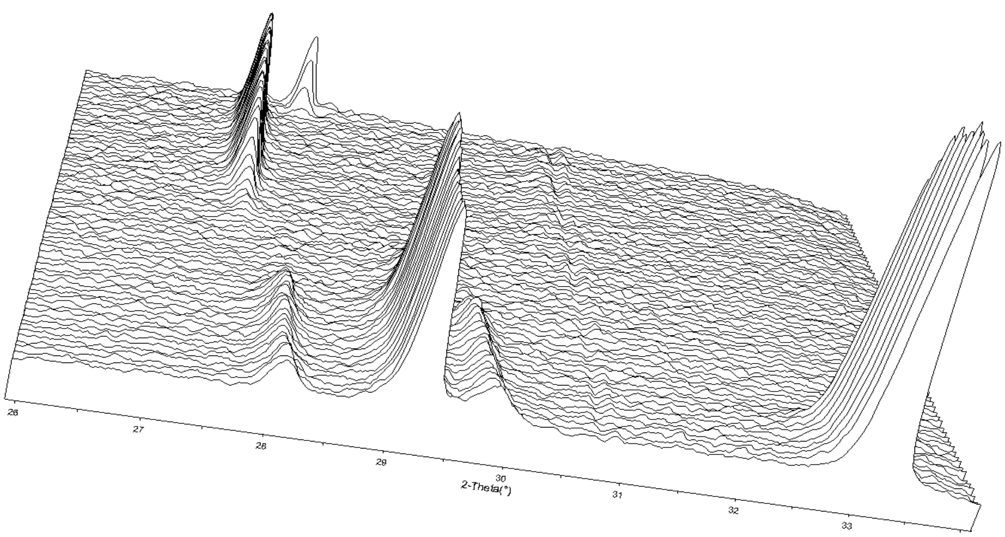

Fig. 1. A typical high-temperature x-ray diffraction data set, which displays a series of x-ray scans on an In/Se bilayer film as the temperature is increased from 27°C (front) to 720°C (back). The Indium precursor is observed to melt first, then the Selenium, and the In_2Se_3 phase forms at high temperatures.

order to determine the reaction pathways and provide quantitative values for the reaction kinetics that can be used in predictive modeling of the CIGS system. An understanding of the reaction pathways and their kinetics could enable the development of efficient and inexpensive processing methods for producing photovoltaic cells with polycrystalline thin films.

Materials Analysis User Center (MAUC)

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University of Missouri-St. Louis researchers study fuel cell catalysts using HTML's ACEM

Research problem: To investigate microstructure and chemistry of Pd/ZnO nanocatalysts

Implications: Ability to identify mechanisms for enhancing catalytic activity of nanostructured catalysts in fuel cells and other energy-production devices

Description of Work: Developing an understanding of new catalytic materials for direct methanol fuel cell applications is possible with the latest generation HTML electron microscopes. The results from this work are critical to efforts directed toward enhancing the catalytic activity and selectivity of nanostructured catalysts. Under a new HTML project, Prof. Jimmy Liu and his student Jinfeng Wang (Fig. 2) are working with ORNL researcher Larry Allard using the HTML's aberration-corrected electron microscope (ACEM) to study novel catalysts for on-board reforming of methanol to provide hydrogen for PEM fuel cells. An increase in the efficiency in such devices is needed to make it a favorable route for advanced fuel cell vehicles. The present study involves a new catalyst based on palladium on zinc oxide supports, where the zinc oxide is a synthesized material with a "nanobelt" microstructure. The support is an ideal model catalyst system for this study, as the nanobelts are single crystal of uniform thickness that provide an optimum structure for high resolution studies. The images in Fig. 3 (next page) show the overall structure of the material (left), where the bright contrast features are the Pd particles. The crystal lattice of the Pd relative to the ZnO nanobelt is shown in the high resolution image (right), indicating the kind of information yielded by the ACEM on the catalyst microstructure. Ultimately, this research can have a significant impact on understanding and predicting the general behavior of metal-oxide interactions and how these interactions affect the performance of nanostructured catalysts for fuel cell and other energy-production applications.

Fig. 2. Prof. Jimmy Liu (foreground) and graduate student Jinfeng Wang from the University of Missouri, St. Louis.

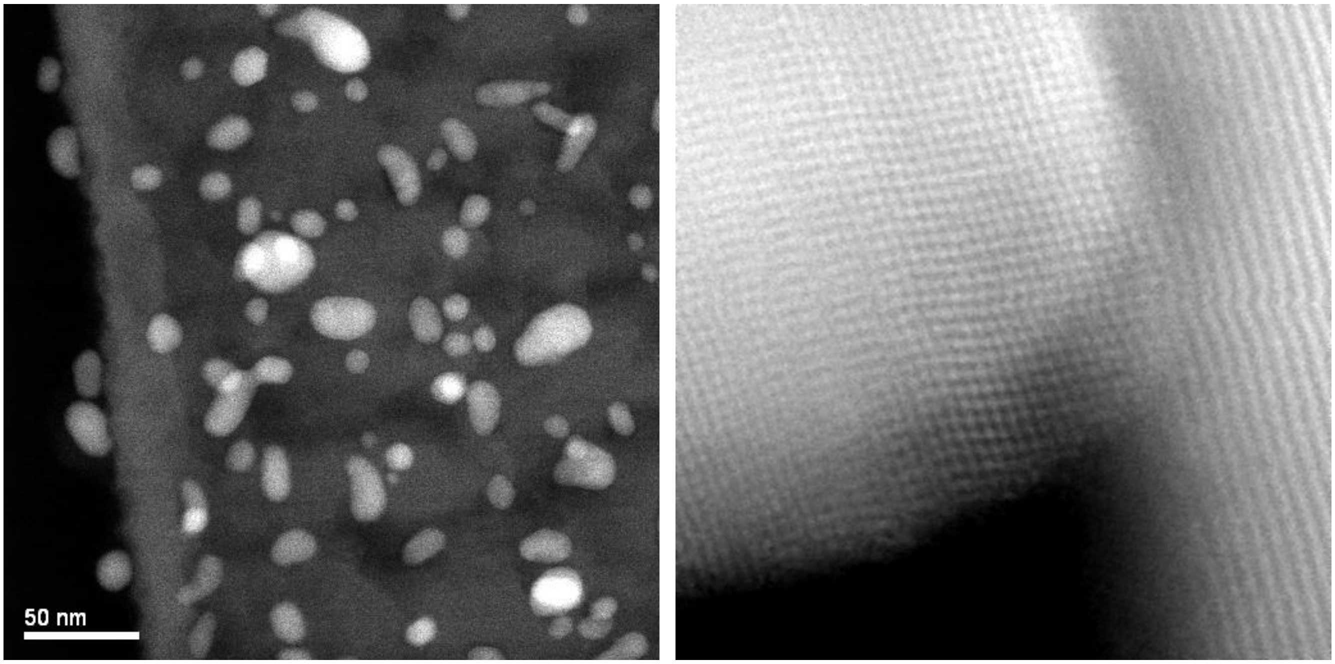


Fig. 3. (left) Overall structure of new catalyst material based on palladium on zinc oxide support, where the bright contrast features are the Pd particles. Zinc oxide is a synthesized material with a "nanobelt" microstructure, as shown in the image at right of the crystal lattice of the Pd relative to the ZnO nanobelt.

University of Maine project analyzes structures of bimetallic fuel-cell catalysts

Research problem: To analyze the atomic-level structure of a series of zeolite-based catalysts designed for fuel cells

Implications: To develop improved techniques for removing impurities from hydrogen prior to its use in PEM fuel cells

Description of Work: Prof. Howard Patterson from University of Maine and his graduate student Robert Gomez are studying the atomic structure of a series of zeolite-based monometallic and bimetallic catalysts designed to remove carbon impurities such as carbon monoxide from hydrogen that is supplied to fuel cells. Such fuel cells are being considering for powering electric vehicles in the future. Zeolites are aluminosilicate materials with a crystal structure that exhibits large "cages," in which heavy metals such as cobalt and molybdenum can sit. The question was to try to determine if the different atoms actually infiltrated into the crystal structure, or simply agglomerated into larger particles sitting on the surface of the zeolite. ORNL researcher Larry Allard worked with Robert Gomez (Fig. 4) using the HTML's aberration-corrected electron microscope (ACEM) to obtain dark-field images (in which high atomic number atoms appear in bright contrast) to characterize the catalyst microstructure and to identify the location of cobalt and molybdenum compounds. An example of the structure of a Mo/zeolite sample is shown in the dark-field image (Fig. 5 next page), which shows Mo(III) atoms in ZSM5 zeolite. The individual Mo(III) atoms (seen in bright contrast) appear to occupy the negatively-charged β-chambers of the zeolite, which are ordered in a regular grid-like

Fig. 4. Robert Gomez (foreground) works at the ACEM with the HTML's Larry Allard.

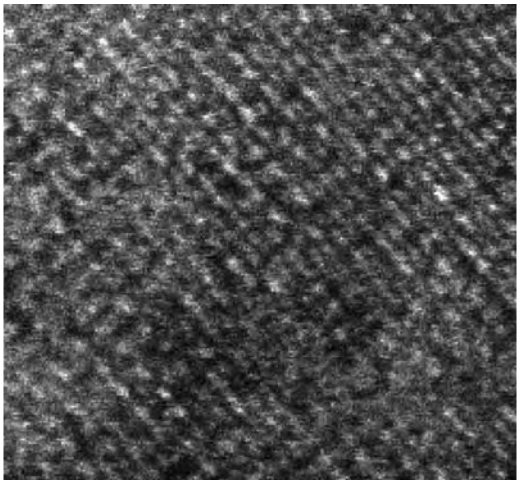


Fig. 5. Structure of a Mo/zeolite sample in a dark-field image shows Mo(III) atoms, in bright contrast, in ZSM5 zeolite.

pattern. An improvement in fuel cell efficiency with significant energy savings is expected as these catalysts are improved and effectively utilized to remove carbon impurities from hydrogen.

Capstone Turbine assesses effects of water ingestion on C60/C65 MicroTurbine™ components

Research Problem: Determine the detrimental effect of operating a microturbine with 0.05 gpm of water ingested with inlet air

Implications: Reduced microturbine operating life due to premature failure of various components due to water ingestion

Description of Work: Capstone has thousands of C60 & C65 MicroTurbines installed around the world. In hot, dry climates, the introduction of water into the inlet air of a turbine increases inlet air density and thereby increases turbine power, accompanied by ~30% more water vapor in combustion by-products at levels of .05 gpm of water entering the turbine. However, gas turbine research has established that the introduction of water along with the inlet air, while increasing power, tends to reduce component life. To assess the effects of water ingestion on the various microturbine components, a C60 engine that operated for ~13,000 hours with water ingestion of 0.05 gpm was decommissioned, torn down, and the hot section components sectioned for analysis at the HTML. Capstone Turbine's Wendy Matthews utilized the electron microprobe (JEOL 8200 EPMA) and scanning electron microscope (Hitachi S-3400 Environmental SEM) available at HTML to examine samples taken from the compressor impeller, combustor, fuel injectors, turbine nozzle, turbine wheel, and recuperator. Components analysis revealed accelerated corrosion rates for both the combustor and recuperator materials, and the presence of pitting corrosion on the compressor impeller. HTML staff Dr. Karren More and Larry Walker worked with Ms. Matthews to identify the contaminants present in the pitting corrosion on the stainless steel compressor impeller. Elemental mapping at the location shown in Fig. 6 (next page, left image) confirmed the presence of sulfur, calcium, sodium, chlorine and potassium throughout the corrosion product (Fig. 6, center and right images). The presence of these contaminants seems to indicate that the pitting corrosion on the compressor impeller occurred as a result of the increased content of the water and the contaminants within the water.

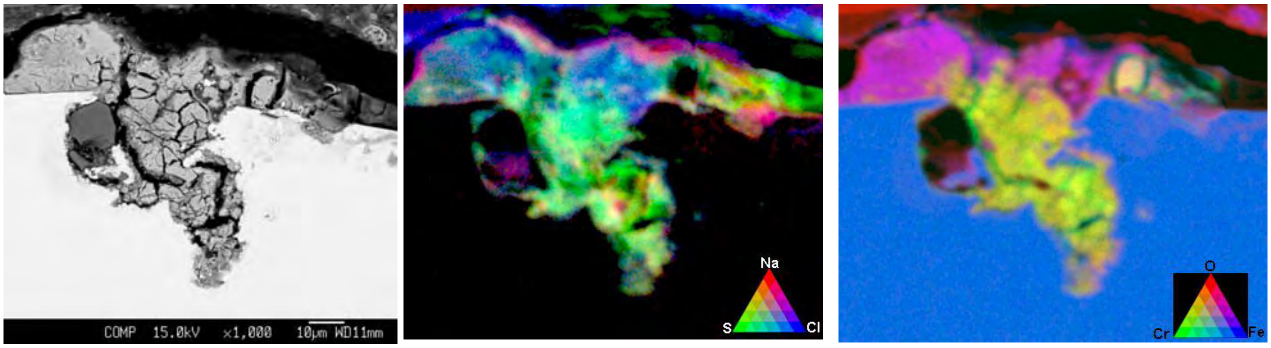


Fig. 6. Environmental SEM images of gas turbine compressor impeller. Left: pitting corrosion; center and right: elemental mapping showing presence of sulfur, calcium, sodium, chlorine and potassium.

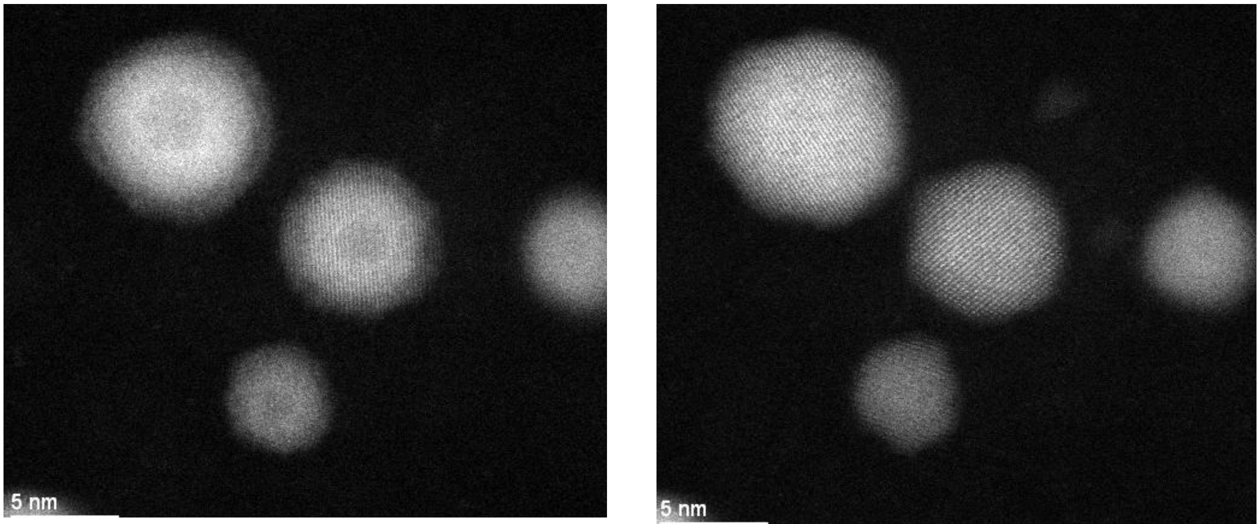
In situ heating of Au-Pd nanoparticles reveals interesting phenomena in University of Texas-Austin catalyst user project

Research problem: To better understand behavior of catalyst bimetallic nanoparticles during heating and gas reactions

Implications: Develop catalyst materials for automotive applications

Description of Work: University of Texas-Austin researcher Prof. Miguel Jose-Yacaman (below) and the HTML's Dr. Larry Allard continued studies of catalyst nanoparticle structure and morphology on the HTML's aberration-corrected electron microscope (ACEM), which allows the atomic imaging at sub-Ångström

resolution that is imperative for such research. The work has been extended into the area of *in situ* heating to better understand both changes in particle structure and also the sintering behavior of particles at elevated temperatures. A unique heating capability that provides the ability to record ultra-high-resolution images at high temperature (see Protochips Co. highlight, page 26) was applied to imaging the behavior of Au-Pd particles with a novel 3-layer structure (core with two surrounding shells). The images in Fig. 7 (next page) show the homogenization of the 3-layer particle (Pd-rich core, Au-rich first shell and Pd-rich outer shell) with time at temperature. Interestingly, it was observed that adjacent particles would sometimes not sinter together, or agglomerate, but instead one particle would gradually reduce in size while the grain in a closely adjacent particle concomitantly grew. This phenomenon is shown in the second and third images of Fig. 7. As a more complete understanding of *in situ* heating system is developed, systematic experiments will be conducted to further clarify behavior of catalyst materials to be used in fuel cells and other vehicle-related applications.



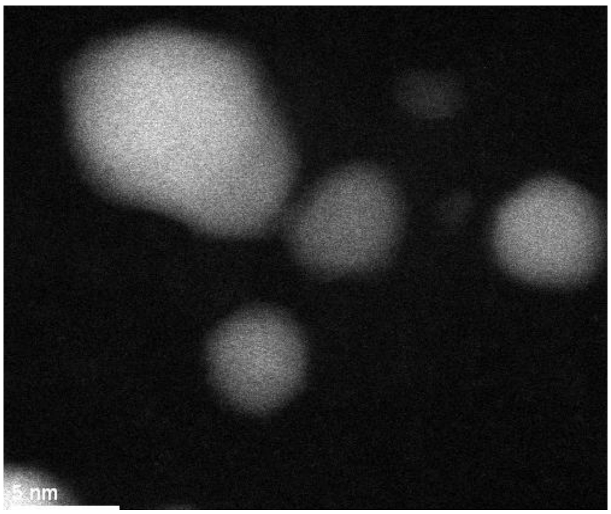


Fig. 7. Homogenization of the 3-layer particle (Pd-rich core, Au-rich first shell and Pd-rich outer shell) with time at temperature. Upper left image of three layers; upper right and lower left images illustrate the observed behavior in which one particle gradually reduces in size while the grain in a closely adjacent particle concomitantly grows.

Florida State analyzes Ru/Sr_2RuO_4 metal/superconductor interface

Research problem: To understand the structure of superconductors and metals at the interface of oxide materials

Implications: Insights into behavior of superconductor microstructure under high-temperature conditions

Description of Work: Dr. Larry Allard has been collaborating with Dr. Yan Xin from the National High Magnetic Field Laboratory at Florida State University to characterize the microstructure of super-conductors using advanced high resolution STEM techniques, e.g., atomic resolution high-angle annular dark-field (HA-ADF) imaging and atomic resolution EELS. During a recent visit they studied the interfaces between Ru metal and an unconventional superconductor, strontium ruthenate (Sr_2RuO_4). This layered superconductor is the first perovskite superconductor that shares the same crystal structure as high critical temperature (high-Tc) superconductors of copper oxides, but without Cu. Its Tc is 1.5K. A surprising discovery was a remarkable enhancement of Tc by a about a factor 2 in a two-phase composite eutectic system made of a single-crystalline matrix of Sr_2RuO_4, in which microdomains of ruthenium metal are embedded. Figure 8 (next page) shows a Ru metal lamella in bright contrast (left), and a spectacular atomic resolution HA-ADF image of the Ru/Sr_2RuO_4 interface (middle). It is immediately clear from the image that the terminating plane for the

Sr_2RuO_4 is parallel to the c axis, i.e. a or b plane terminates at the Sr atoms, leaving the RuO_2 octahedron intact (as indicated by the schematic at right). Preliminary EELS data of the oxygen core loss spectra from the interface compared with the bulk shows some difference, indicating the distortion/rotation of the RuO_2 octahedron at the interface. Further experiments using the HTML's ACEM will confirm these results. Possible insights into the 3K superconducting phase will be obtained through our continuing investigation.

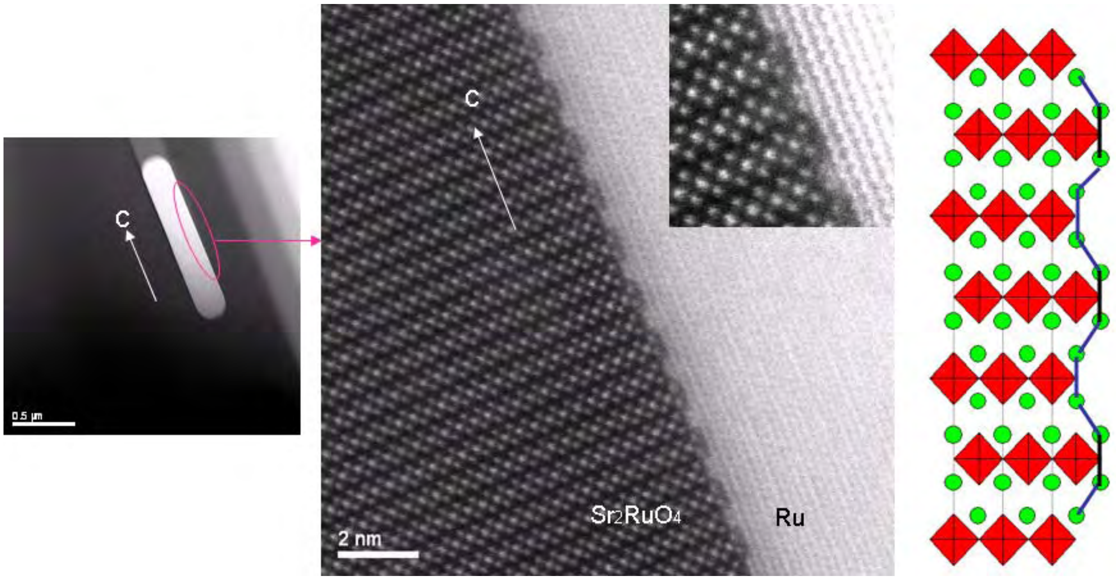


Fig. 8. (left) ACEM image of Ru metal lamella in bright contrast; (middle) atomic resolution HA-ADF image of the Ru/Sr_2RuO_4 interface; (right) Ru/Sr_2RuO_4 schematic.

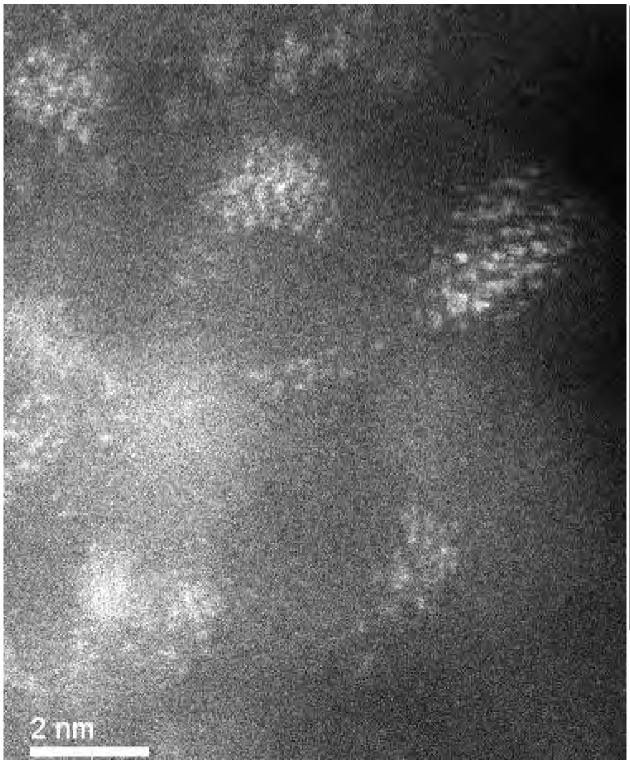
PNNL continues collaborative electron microscopy studies of NOx trap catalysts

Research problem: To develop a thorough understanding of the properties and behavior of lean NOx trap catalyst systems using ultra-high-resolution electron microscopy techniques

Implications: Improvements in diesel engine emission reduction may ultimately allow the highly fuel-efficient diesel engine to be more fully incorporated into the U.S fleet of passenger vehicles

Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

Description of Work: Using the HTML's JEOL 2200FS-AC aberration-corrected electron microscope (ACEM), both Ba-alumina and Pt-alumina compositions of lean NOx trap catalysts are being studied by PNNL's Dr. Chuck Peden and HTML researcher Dr. Larry Allard. In recent follow-up work to a user visit in 2007, Allard imaged Pt/alumina samples calcined at both 300°C and 600°C, to determine the structure and morphology of the Pt species at atomic resolution. Because of a relatively high loading (10% Pt by weight), the sample at 300°C showed some discrete crystals in the 3-5nm size range, but also a uniform dispersion of single atoms, small clusters, and "rafts" of atoms with irregular periodicity. The 600°C sample, however, showed a bimodal distribution of species, with some large perfect Pt crystals 10nm or larger, although with some remaining clusters and rafts of atoms. The latter features are illustrated in Fig. 9 (left, next page), while the perfect crystalline structure appears in Fig. 9 (right, next page). Work is continuing with new samples having significantly lower Pt loadings.



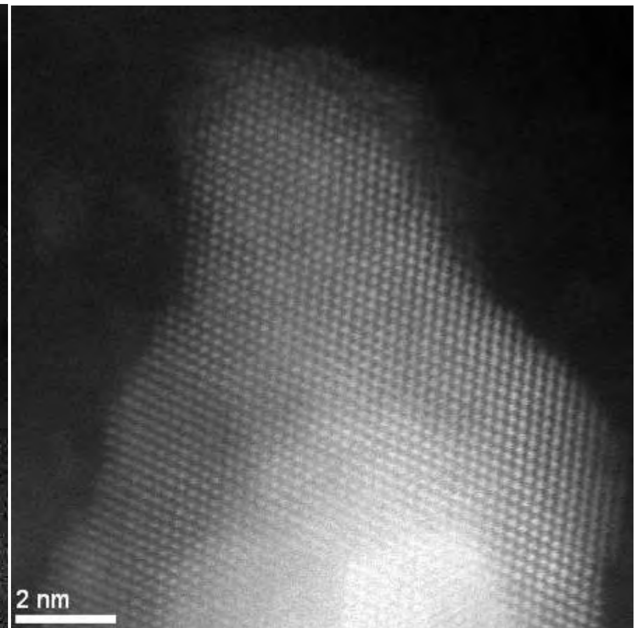


Fig. 9. (left) Pt-alumina trap catalyst material showing clusters and "rafts" of atoms; (right) crystalline structure of Pt-alumina trap catalyst material.

Mechanical Characterization and Analysis User Center (MCAUC)

contact: Edgar Lara-Curzio, Leader, laracurzioe@ornl.gov, 865/574-1749

Michigan State evaluates thermal cycling effect on elastic modulus of thermoelectric materials

Research problem: To characterize the physical and mechanical properties of silver and antimony doped PbTe thermoelectric materials

Implications: Energy recovery through thermoelectric materials could significantly increase overall engine efficiency.

Description of Work: Thermoelectric materials are being considered for applications in waste heat recovery, which would improve the efficiency of transportation systems. New compositions, such as lead-antimony-silver-tellurium (LAST) and lead-antimony-silver-tellurium-tin (LAST-T) are being developed by the department of Chemical Engineering and Materials Science at Michigan State University (MSU). Professor Eldon Case and graduate students Fei Ren, Jennifer Ni, and Brad Hall visited the HTML to work with MCAUC researchers Rosa Trejo, Amit Shyam, and Edgar Lara-Curzio to assess the effect of temperature and thermal cycling on the elastic properties of these materials. They used the HTML's infrared (IR) lamp and IR camera systems to thermal cycle the samples (Fig. 10, next page). After each thermal cycle, the resonant ultrasound spectrometer (RUS) was used to determine the resonant frequencies of the material, which are subsequently used to determine the elastic constants of the material. In addition to thermal cycling, the MSU group also continued using RUS to determine the elastic constants as a function of temperature of test specimens obtained from new ingots of cast and hot-pressed thermoelectrics. It was found that the Young's modulus decreases linearly as a function of temperature, and no hysteresis or kinks were found (Fig. 11). The results of this investigation will provide necessary information for design purposes and for the calculation of thermal stresses present during operation of waste heat recovery devices

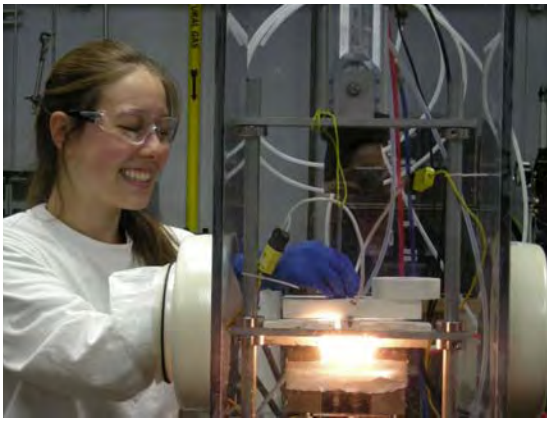


Fig. 10. MSU student Jennifer Ni using the infrared (IR) set-up to thermal cycle thermoelectric materials.

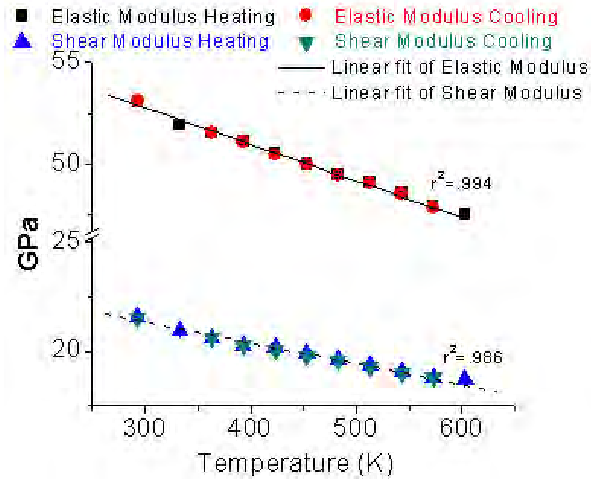


Fig. 11. Elastic and shear modulus as a function of temperature for a hot pressed n-type thermoelectric.

Army Corps of Engineers R&D Center conducts heat-of-deformation study

Research problem: To investigate the effects of strain-rate and heat-of-deformation on energy absorption processes during dynamic progressive crush deformation

Implications: Ability to develop accurate predictive computational material models and obtain advanced material solutions for protective structures applications

US Army Corps of Engineers ®
Engineer Research and Development Center

Description of Work: Ductile, metal alloy structures that rely on controlled, progressive plastic deformation along specific paths or "traveling fold-lines" have the potential of achieving extremely high energy absorption capability to provide advanced levels of impact and blast load protection. However, under these conditions the material response is very complex because it experiences a severe transient, nonlinear, multi-axial state of stress. In addition, the maximum load and the amount of energy absorption capability are influenced by inertia, strain rate, temperature and heat-of-deformation. Researchers at the Army Corps of Engineers are participating in a multi-scale modeling and experimental research program to obtain critical knowledge and physically-based material models for the development of advanced material solutions for the design or defeat of structures subjected to extreme impact and blast loading conditions. Dr. Beverly DiPaolo of the Army Corps of Engineers R&D Center visited the HTML to work with MCAUC researchers Don Erdman, Mike Starbuck and Ralph Dinwiddie (TTPUC) to study material deformation mechanisms, and to develop physically-based constitutive models for structural components that utilize severe plastic deformation to absorb high levels of energy. As part of this HTML user project dynamic axial crush tests of thin-wall tubular structures were carried out using ORNL's unique Test Machine for Automotive Crashworthiness (TMAC) at a constant speed of 6 m/s. The tube specimens had groove collapse initiators and were quasi-statically pre-crushed. Four different steel materials were evaluated: A36 structural steel, A513 mechanical steel, 316SS stainless steel and 304SS stainless steel. In addition to high speed video, the tests were recorded using a high-speed infrared camera (Fig. 12, next page) to determine the temperature distribution of the tubular components during the test (Fig. 13). The results of this research are expected to lead to better protective structures, e.g., improved vehicle crash-worthiness. Partial support for the operation of TMAC was provided by DOE's Automotive Lightweighting Materials Program.

Fig. 12. High-speed and thermography cameras ready for high-rate testing of steel crush tubes.

Fig. 13. Beverly DiPaolo (ERDC) discusses thermography results from axial-crush testing with the HTML's Don Erdman (left) and Ralph Dinwiddie.

Pratt & Whitney investigates long-term residual properties of ceramic matrix composite materials

Research problem: To predict durability and life of melt infiltrated SiC/SiC based on interfacial shear strength

Implications: Development of CMC systems that could significantly improve fuel efficiency and lower emissions of gas turbines.

Description of Work: Insertions of ceramic matrix composite (CMC) materials are key to more efficient internal combustion engines. Currently melt infiltrated SiC/SiC CMCs are leading candidate material systems for the manufacture of hot section components in gas turbine engines. Pratt & Whitney's Dr. Greg Ojard worked with HTML staff Rosa Trejo, Laura Riester, and Dr. Edgar Lara-Curzio to characterize the fiber/coating/matrix interfacial sliding in this composite system. Using the HTML's nanoindenter, many independent fibers were pushed out from CMC materials in the as-received state and also from test specimens that had been subjected to creep or fatigue testing for thousands of hours at 1204^{o}C. Figure 14 (next page) shows force-displacement curves obtained from single-fiber push-out tests in test specimens in the as-received state or after being subjected to 1204^{o}C for 1508 hours under a constant tensile stress of 165 MPa. It was found that the interfacial shear strength decreases from an average of 30MPa for materials in the as-received state to 18MPa for comosites that had been subjected to creep testing. The decline in the interfacial shear strength after exposure is an indication that the interface deteriorates due to a combination of stress and environment (i.e. the boron nitride interface oxidized). Knowing how the interfacial shear strength of these materials is affected by stress, temperature, environment and time will aid in the prediction of durability and life of the CMC for power generation systems.

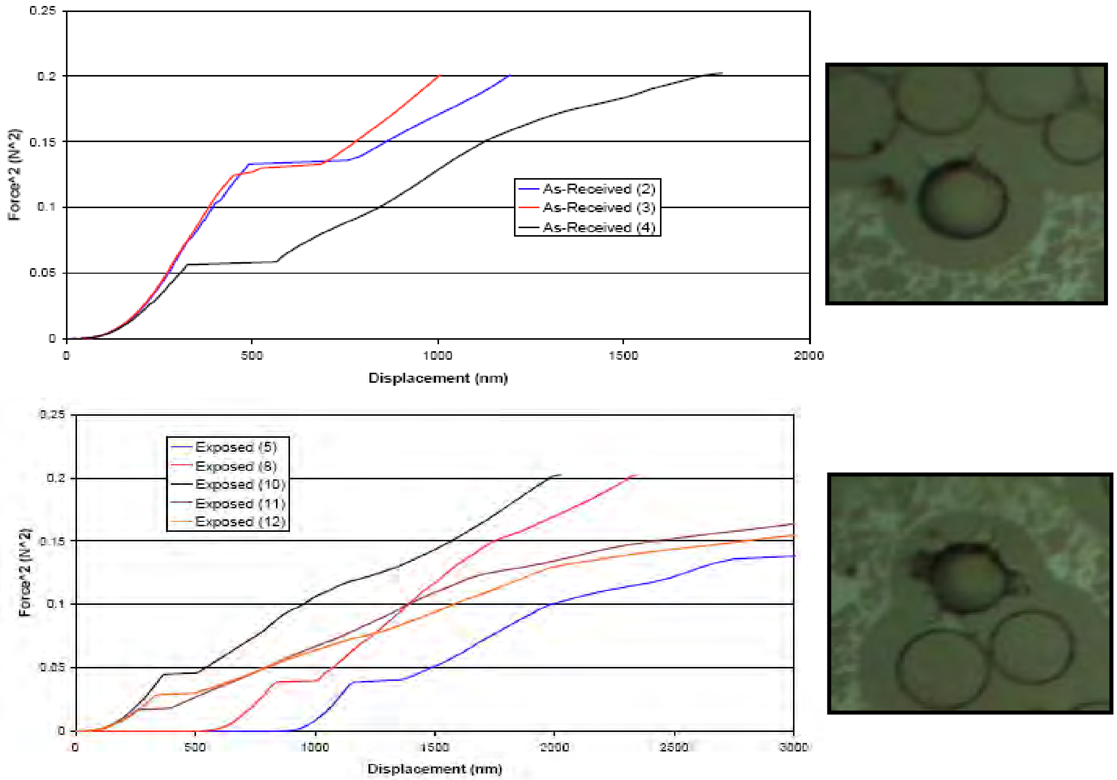


Fig. 14. Fiber push-out curves and pushed out fibers on test specimens in the as-received state (top) and after exposure to 1204°C for 1508 hours at 165 MPa (bottom). It was found that the interfacial shear strength decreases in the exposed composites.

L&L Products conducts exploratory high-rate testing of hybrid box beams

Research problem: To collect test data on crash resistance of composite/steel vehicle structural components

Implications: Knowledge of the mechanical performance of automotive structural components at high rates is needed to ensure vehicle safety.

L&L Products

Description of Work: L & L Products manufactures structures known as Composite Body Solutions®, which are macro-composites made of a metal, thermoplastic or thermoset carrier and heat-activated expanding adhesive. These structures can be used at key locations to reinforce vehicles and improve their crashworthiness. Ken Takahashi and Dean Schneider from L & L Products collaborated with an HTML research team of Don Erdman, Mike Starbuck, Rick Battiste, and Barbara Frame (Fig. 15, next page) to perform preliminary bend crush tests to establish baseline data on the suitability of L & L's custom three point bend test fixture and to verify the reliability of the test configuration for collecting the desired high-rate data. All preliminary tests were successfully completed (Fig. 16, next page), and future testing has been scheduled in early FY2009 to complete the test matrix, which consists of over 100 specimens at various rates up to 8 m/sec. Partial support for the operation of TMAC was provided by DOE's Automotive Lightweighting Materials Program.

Fig. 15. The research team reviews test results. (back to front): Dean Schneider (L&L), Ken Takahashi (L&L), Mike Starbuck and Barbara Frame (ORNL Polymer Matrix Composites Group), and Rick Battiste

Fig. 16. 3-point bend test equipment (left) and crushed bend specimens after successful testing (right).

Mississippi State Tests Combined Creep-Fatigue Model for Composites

Research problem: To develop and evaluate a constitutive spectrum-based model for composite materials used for lightweighting vehicles

Implications: Models are needed to optimize the design of composite components and to predict their serviceability and lifetimes.

Description of Work: The primary benefit of using lightweight advanced materials in transportation applications is the increase in fuel economy, but high performance is also realized from the use of these materials. Mississippi State University researchers Dr. Rani Sullivan and student Jutima Simsiriwong (Fig. 17) have developed a predictive model of creep and fatigue behavior of an E-glass/vinyl ester composite, and they visited the HTML to acquire the necessary material creep and fatigue input parameters

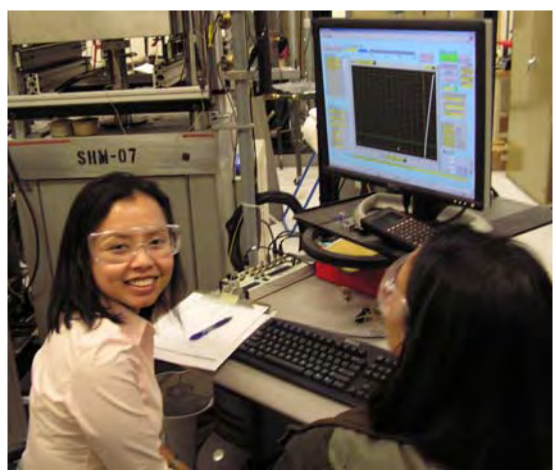


Fig. 17. (left) Dr. Rani Sullivan (at left) and Jutima Simsiriwong review data from the servohydraulic testing machine. (above) J. Simsiriwong (left) and R. Sullivan continue mechanical testing at the MCAUC.

for this modeling endeavor. Working with ORNL researcher Don Erdman, the MSU researchers initiated the first phase of their user project by conducting monotonic tensile tests at ambient and slightly elevated temperatures (90°C) to determine strength and stiffness values that can be used to select creep load levels. All monotonic tensile testing was completed, and creep tests were initiated to determine creep rupture times as a function of applied stress. Additional testing to determine rupture parameters at higher temperatures and lower stress levels will also be conducted. The response from this model will be used to define a damage function that describes the degradation in stiffness due to cyclic loading.

Colorado School of Mines Conducts *In Situ* Raman Analysis of Beta-Eucryptite Pressure-Induced Transformation

Research problem: To understand the fundamentals of phase transformations in β-eucryptite

Implications: Possible development and manufacture of tough, durable catalyst supports and diesel particulate filters

Description of Work: To address the stringent diesel engine emissions regulations adopted in the United States, attention has focused on developing improved high-temperature-resistant, high-thermal-shock-resistant, low cost honeycomb soot filter compatible with advanced emissions control catalyst technologies that can replace current high-cost and/or uncatalyzed particulate filters. Among the catalyst-compatible ceramic particulate filter designs being developed for this application are the low-expansion alkali aluminosilicates such as β-eucryptite.

Professor Ivar Reimanis and his research group at the Colorado School of Mines recently reported a unique phenomenon in which particles are ejected from the surface of a β-eucryptite subsequent to indentation. It is hypothesized that a reversible phase transformation to orthorhombic ε-eucryptite occurs under compressive loads, and that the reverse transformation to β-eucryptite leads to particle ejection. Since this transformation may occur during material processing, it opens the possibility of a novel transformation-toughened material that could lead to significantly reduced cost of materials for applications in catalyst support and filtration of diesel particulates. To test this hypothesis, graduate student Tim Jochum (Fig. 18) visited the HTML to work with MCAUC researcher Michael Lance to quantify the effect of stress on the phase transformations of β-eucryptite *in situ* under a diamond indenter using Raman spectroscopy.

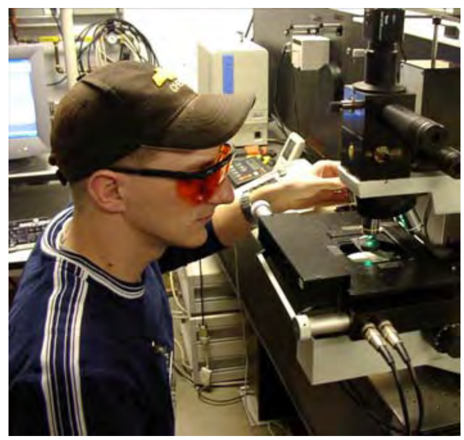


Fig. 18. Tim Jochum collecting Raman spectra on his β-eucryptite sample.

The incident laser light was focused through a ~100 μm diameter flat diamond punch which was loaded from 1 to 6.5 kg. Figure 19 (next page) shows the spectra acquired over the applied load range. The β-eucryptite peak occurs at 490 cm^{-1} and a background peak from the diamond indenter occurs at 650 cm^{-1}. At an applied load of ~3 kg, a shoulder appears on the 490 cm^{-1} peak that doesn't shift away gradually from the initial peak, but rather it grows independently. This strongly suggests that it is produced by a phase transformation occurring at high loads as opposed to a high compressive stress region under the indenter, which would produce a peak shift to higher frequencies with more load. This peak may be the sought-after high pressure ε phase which is the hypothesized cause of the ejection phenomenon being investigated. Additional work is underway to verify these results, which could lead to the development of diesel particulate filters with improved resistance to high exhaust temperatures encountered during filter regeneration cycles, as well as to the thermal shock conditions arising during the course of startup and regeneration.

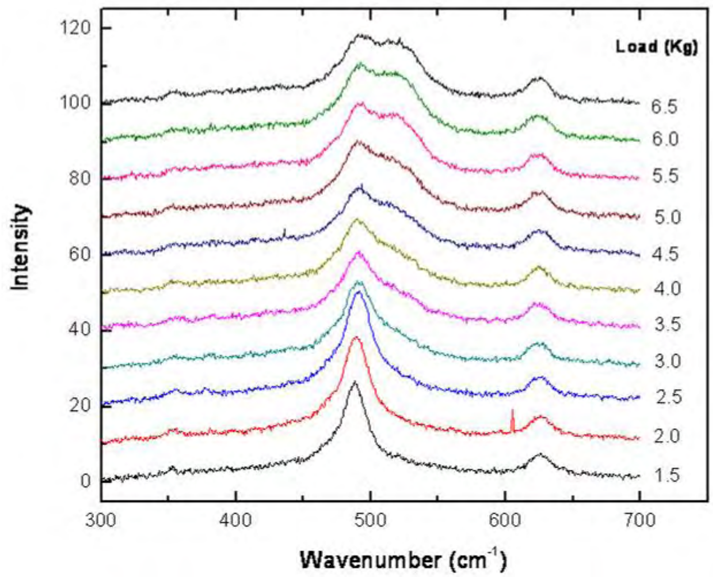


Fig. 19. β-eucryptite exhibits a Raman peak near 490 cm^{-1}. The peak at around 625 cm^{-1} is from the diamond punch. At roughly 3 kg of applied load, a shoulder emerges at ~525 cm^{-1} that may be due to ε-eucryptite formation.

Mattson Technology characterizes properties of high-dose implant strip (HDIS) residues

Research problem: To characterize residues created by plasma stripping process during integrated circuit manufacturing

Implications: Efficient residue removal is a major step toward accomplishing the goal of reducing energy usage while increasing reliability of microelectronics.

Description of Work: Mattson Technology, Inc. manufactures integrated circuits via ion-implantation methods. Application of reactive plasma is a very efficient process to strip photoresists on silicon wafers, but it leaves unfavorable residues on the strips that cause yield problems. High-Dose Implant Strip (HDIS) residues are typically removed through chemical etching and/or physical force, but this produces hazardous chemical waste and mechanical damage to the underlying substrate. A plasma stripping process used to remove an implantation photoresist mask normally leaves behind a residue that causes yield problems and hence waste and excess energy usage in the manufacturing process.

Dr. Andreas Kadanavich (Fig. 20, next page) from Mattson Technology collaborated with research staff in the MCAUC as well as in the MAUC to identify location and composition of residue material left behind following stripping of the photoresist. MCAUC staff member Sanghoon Shim and Dr. Kadanavich used nanoindentation to examine representative samples of wafers that were previously stripped by the user to determine the strength of the residue itself and also the adhesion strength of the residues on a wafer. Based on scratch testing data (Fig. 21, next page), the residues appeared to be much stronger than expected, and their adhesion strength is even greater than that of the strips. In addition, nanoindentation revealed the presence of lengthwise shallow grooves along the strip edges. The grooves are believed to be produced during the fabrication process, but have not been observed previously, since this is the first time nano-indentation scratch tests have been performed. Mattson will investigate further to determine if these grooves must be taken into account during the fabrication process. Figure 22 (next page) shows an SEM micrograph image of residue and an image of a post-scratch sample.

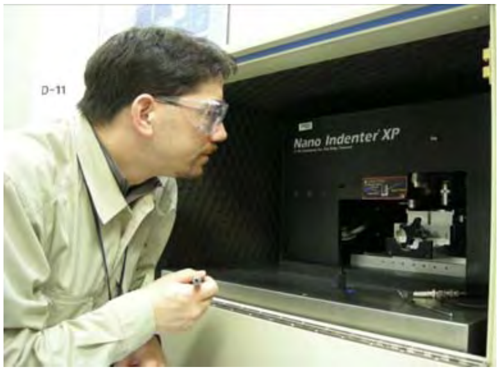


Fig. 20. Dr. Andreas Kadanavich places sample in nanoindenter.

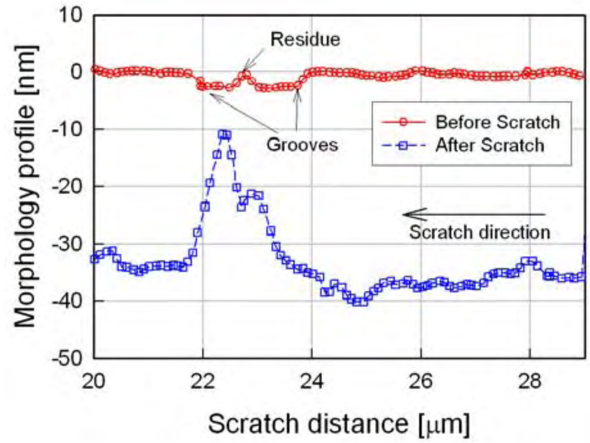


Fig. 21. Morphology profile showing grooves revealed during nanoindentation testing.

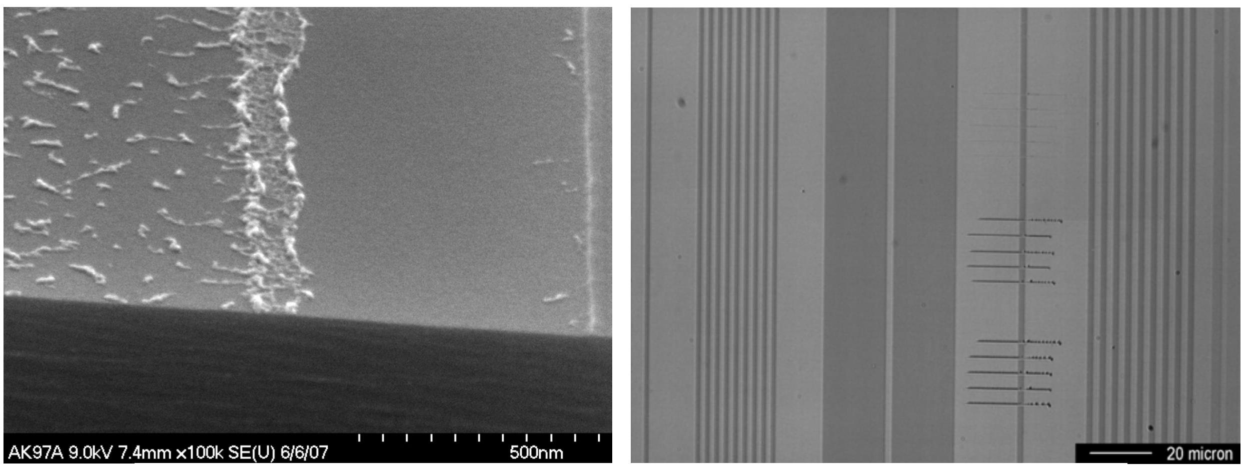


Fig. 22. SEM micrograph of residue (left) and post-scratch sample image (right).

In Mattson's characterization work conducted with Dr. Harry Meyer of the MAUC, the Scanning Auger Nanoprobe was used to characterize the structure of the residue and its chemical composition. Fig. 23 shows a typical area; the white diamond points to "lines" containing residue. Fig. 24 is a micrograph obtained in the Auger system for a 2-micron line feature. Line scans were made in two areas to determine atomic composition of the surface and the residue seen in the center. In Fig. 25 (next page), the line scan identified the residue as silicon oxide, not

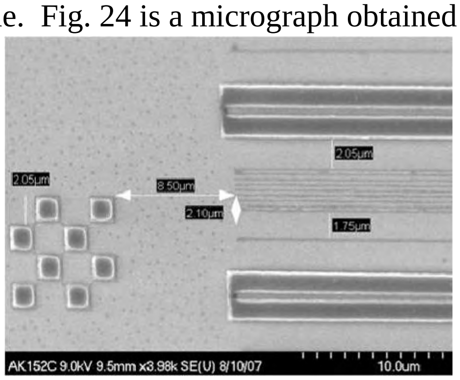


Fig. 23. SEM of typical analysis area.

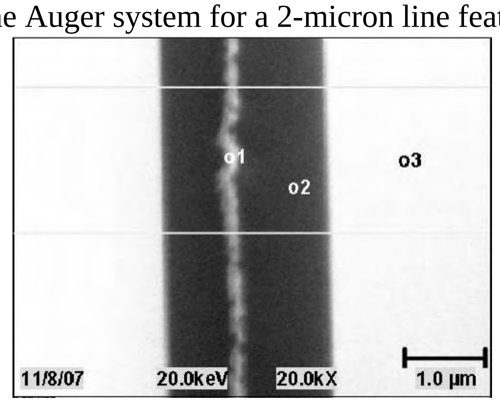


Fig. 24. SEM of 2.0 micron line containing photoresist residue. Horizontal white lines indicate where elemental scans were taken to identify the composition as a function of position.

containing any arsenic (arsenic was one of the species being ion-implanted). Low levels of arsenic were detected in the substrate outside the etched line. This was clear indication that the photoresist stripping process was working as designed.

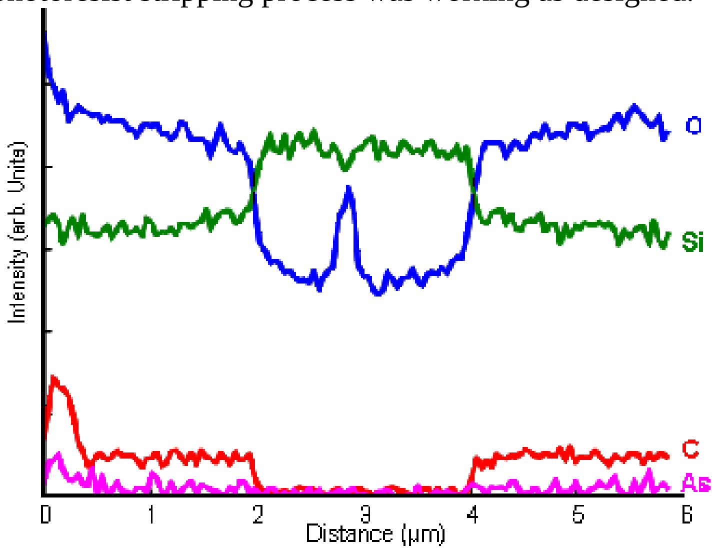


Fig. 25. Line scan for carbon, arsenic, oxygen, and silicon as a function of position along line 1 in Fig. 24. Residue in the center is clearly identified as a silicon oxide material.

Mattson Technology hopes to use the microscopy and nanoindentation results to improve both the fabrication of silicon wafers as well as the removal of the residues created as byproducts during manufacturing, thereby leading to higher performance of these key components of vehicle system microelectronics.

University of Akron (OH) investigates the effect of processing on high-temperature ceramics

Research problem: Processing influences on microstructure, hardness and mechanical response of fine-grained ceramic materials

Implications: Develop durable and reliable fine-grained ceramic materials through better processing

Description of Work: Fine-grained ceramic materials, such as boron carbide, are widely used for high temperature, wear and scratch resistance applications. Professor Tirumalai Srivatsan of the University of Akron (Fig. 26) worked with HTML staff Rosa Trejo, Laura Riester, Sanghoon Shim and Edgar Lara-Curzio on the mechanical characterization of boron carbide samples with different compositions and processing techniques (Reactive Sintering [RS], Hot Press [HP], and Plasma Pressure Compaction [P2C]). One finding from nanoindentation measurements at room temperature and at 400°C suggests that the hardness of boron carbide samples is not affected by the processing routes examined in this project (Fig. 27, next page). The results from this project have provided important information to identify the most effective processing technique to synthesize boron carbide and other fine-grained ceramics.

Fig. 26. Professor Tirumalai Srivatsan uses an HTML nanoindenter to measure elastic modulus and hardness of boron carbide samples.

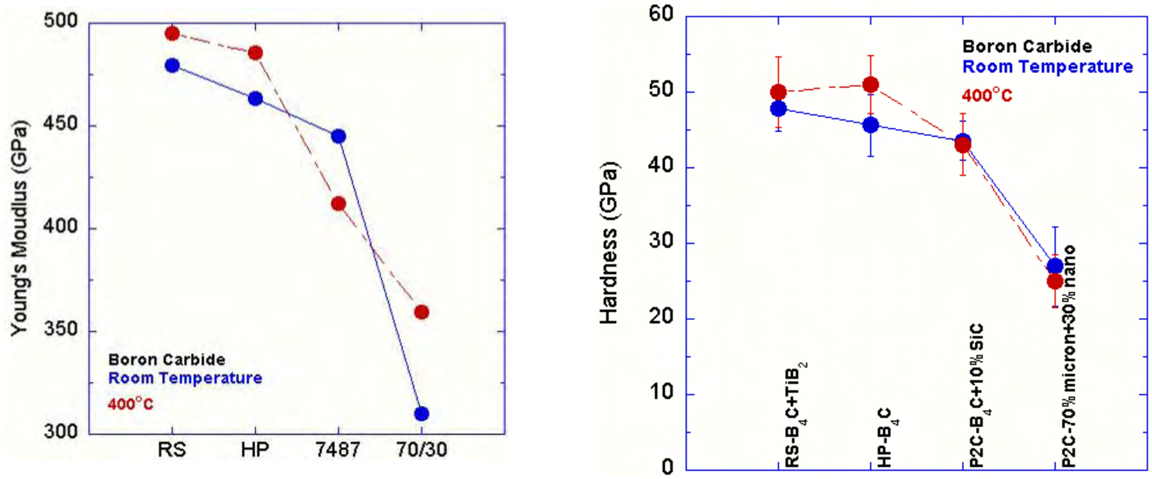


Fig. 27. Hardness vs. processing technique for boron carbide samples.

Brown University investigates ceramic-like geologic materials by nanoindentation

Research problem: To investigate fundamental theories of rock friction and their contribution to rate and state friction effects

Implications: Better understanding of creep behavior and friction effects of ceramic-like geological materials could contribute to a better understanding of the time-dependent mechanical and tribological behavior of hard materials.

Description of Work: A hallmark observation from laboratory experiments on rocks and other geological materials is that the coefficient of friction increases linearly with the log of the time of stationary contact of the sliding surfaces. The magnitude of this "log time strengthening" determines the conditions for unstable, "stick slip" sliding, which is thought to be the primary mechanism responsible for earthquakes. The increase in strength is usually attributed to an increase in the real area of contact between the nanoscale asperities on the contacting rough surfaces of a geological fault in the earth's crust. Curiously, then, the mechanism governing the "megascale" event of an earthquake is inherently controlled by "nanoscale" mechanical phenomena. In an effort to understand the important rate that controls the physical processes involved, Brown University's Dr. David Goldsby (Fig. 28) and HTML's Dr. Sanghoon Shim used nanoindentation techniques to study the time-dependent deformation and the friction processes that occur during small contacts in rocks and minerals. The techniques were applied to quartz (Fig. 29, next page) and olivine, important rock-forming minerals of the earth's crust. The newly developed dynamic techniques allow measurement of the changes in contact area. Measurements revealed that the fractional indentation area increases linearly with the log of the creep time for times up to 15 hours (Fig. 30, next page), congruent with the increase in fractional real area of contact inferred from rock friction experiments over

Fig. 28. David Goldsby monitors nanoindentation measurement of quartz creep.

similar time scales. Knowledge obtained from this investigation could be used to design better systems that are subjected to frictional loading at high homologous temperatures.

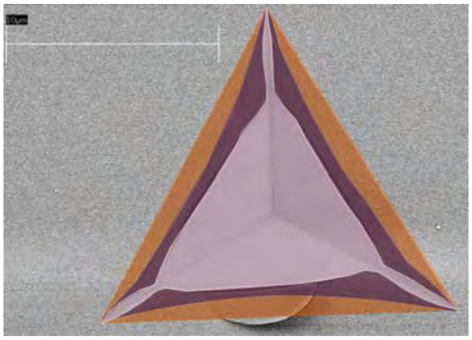


Fig. 29. SEM micrograph of an indent on quartz (scale = 10μm).

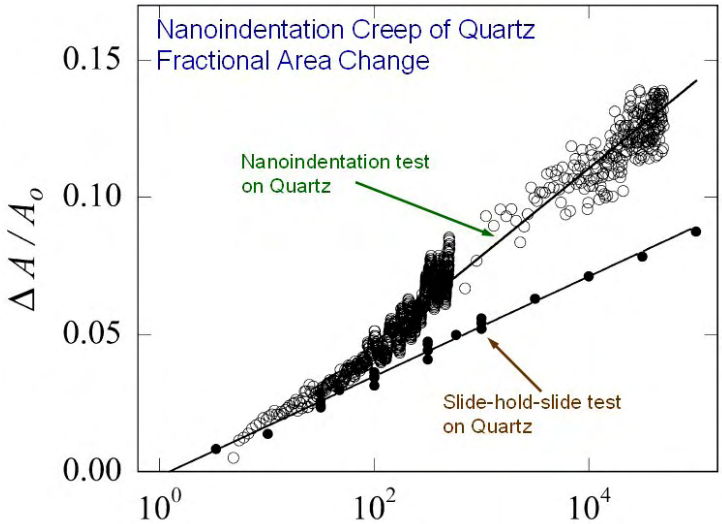


Fig. 30. Increase of fractional indentation area with the log of the creep time and a comparison with a rock friction experiments.

Residual Stress User Center (RSUC)

contact: Camden Hubbard, Leader, hubbardcr@ornl.gov, 865/574-4472

Georgia Tech investigates residual stresses and mechanical properties of white layer on Inconel 718

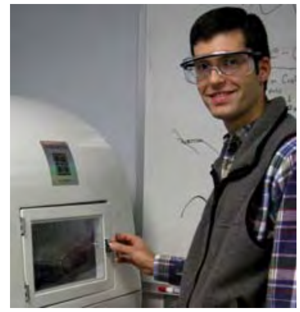
Research problem: To measure residual stress and characterize the Inconel 718 white layer after wire-electrical discharge machining (EDM)

Implications: Potential to apply advantageous EDM to a nickel alloy that improves performance and efficiency of combustion systems

Description of Work: Due to the high temperature properties of nickel alloy materials such as Inconel 718, they are widely used in vehicles, turbines, pumps, and tools. Electric discharge machining (EDM) is a common process for manufacturing Inconel 718 parts because of its many advantages over traditional machining. During EDM, a pulsed voltage difference between a wire electrode and a conductive workpiece initiates sparks that erode workpiece material, forming a recast ("white") layer on the surface of the part. This layer contains an altered microstructure, tensile stresses, microcracks, impurities, and other undesirable features that have a detrimental impact on the surface integrity of machined surfaces, potentially leading to premature part failure. Therefore, this layer is removed to restore the mechanical properties of the material.

Four Inconel 718 samples were examined by Georgia Tech graduate student Tommy Newton using x-ray diffraction (XRD) with Dr. Thomas Watkins, an RSUC researcher, and nanoindentation with MCAUC staff Rosa Trejo, Laura Riester and Edgar Lara-Curzio. The RSUC research focused on determining the impact of EDM wire diameter and "energy/spark" on the resulting residual stresses in the white layer. While little difference was found regarding wire diameter, the tensile residual stress decreased with increasing energy/spark. Careful layer removal revealed a large stress gradient as shown below in Fig. 31.

Newton also used the HTML's Hysitron nanoindenter (right) to compare the mechanical properties of the material after being machined with different EDM parameters. It was found that there is an effect on the hardness and modulus with respect to wire diameter. Even though XRD indicates a relationship between the residual stress and the energy/sparks and nanoindentation shows a correlation between wire diameter and material hardness and modulus, the hardness can also be related to the residual stress in the material. The hardness decreases as the residual stress in the recast layer increases (Fig. 32).



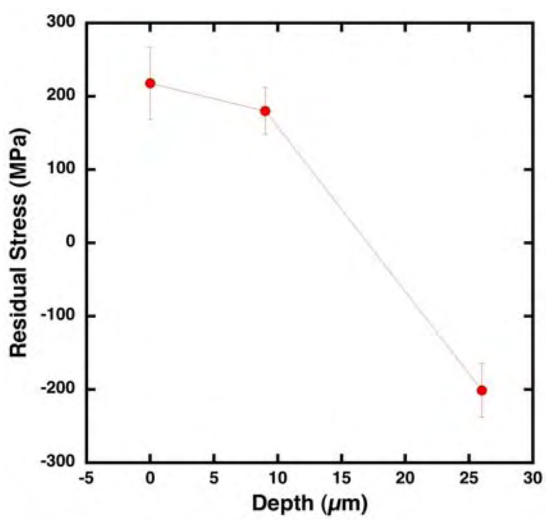


Fig. 31. Residual stress transitions from a tensile recast surface to a compressive substrate in less than 25 microns.

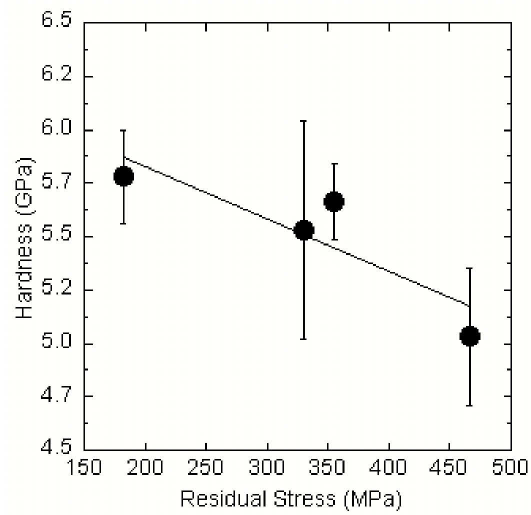


Fig. 32. Hardness vs. residual stress in the recast layer.

The results of this study will aid in a better understanding of recast, or white, layer formation in wire-EDM of Inconel 718, and it will lead to better design of parts to help lower manufacturing costs. Also, for applications where the recast layer must be removed through post-processing, in-process minimization of the recast layer thickness and a thorough understanding of the heat-affected zone could significantly reduce the associated post-processing time and costs.

University of Tennessee studies *in situ* crack closure after overload during fatigue crack growth in alloy

Research problem: To use neutron residual stress mappings of Haynes alloy to develop new fundamental understanding of the overload effect and crack closure mechanisms during fatigue

Implications: Enhance lifetime prediction capabilities and safety models as well as improve design of transportation structural components that are subjected to fatigue and random overloads

THE UNIVERSITY of TENNESSEE UT KNOXVILLE

Description of Work: The deep penetration depth of neutrons enables bulk crack closure measurements as compared to surface crack closure measurements made with a strain gage. Using the RSUC engineering neutron diffractometer NRSF2 at ORNL's High Flux Isotope Reactor (HFIR), the changes in internal crack opening strains in compact tension (CT) test specimens were measured by NRSF2's load frame capabilities as a function of the distance from the crack tip.

Graduate student Sooyeol Lee (Fig. 33) from the University of Tennessee (UT) and Prof. Hahn Choo worked with RSUC team members Cam Hubbard, Tom Watkins and Ke An to conduct the *in situ* loading and strain mapping experiments using Hastelloy C-2000 alloy. CT test specimens with pre cracks were fatigued and overloaded at UT and then subsequently evaluated at the NRSF2, where the strains as a function of applied load and location relative to the crack tip were mapped. The changes in crack opening lattice strain with changes in applied load in a sample subjected to an overload event are shown below in Fig. 34. The *in situ* data enable determination of the crack opening strain level at different stages, resulting in a clearer understanding of the relationship between effective crack tip driving force and crack growth rate. This information will enable the design of more durable and reliable structural components.

Fig. 33. Sooyeol Lee sets up *in situ* loading for neutron diffraction measurements.

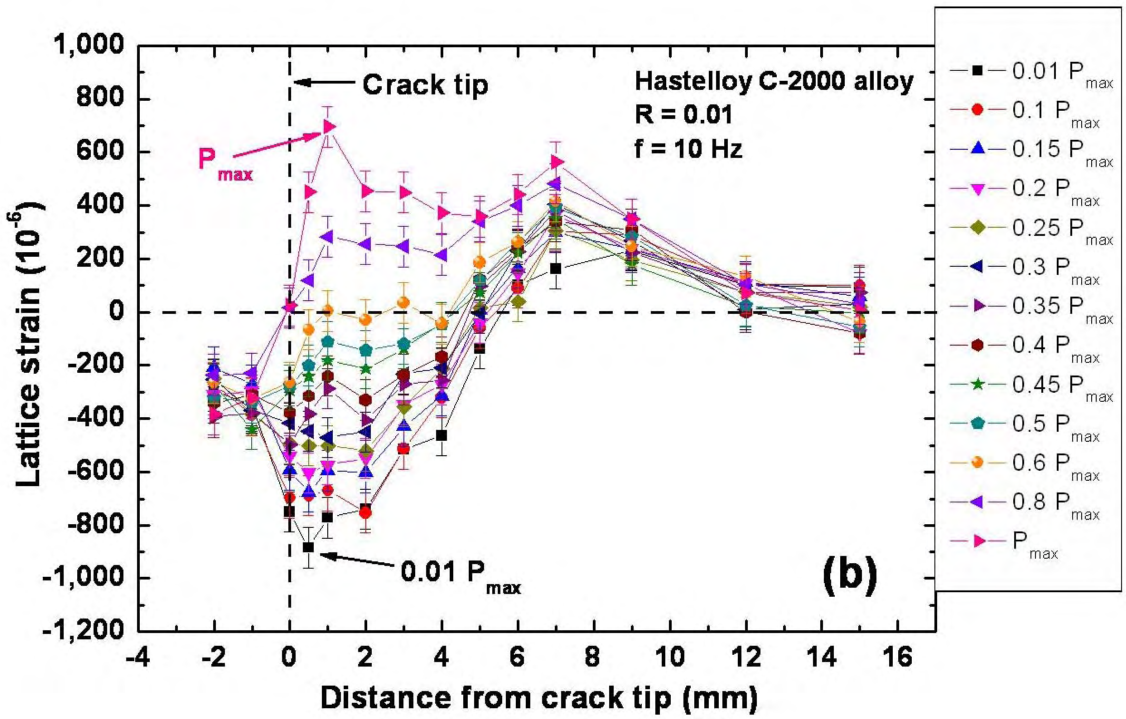


Fig. 34. Evolution of lattice strains with increasing the applied load right after overload during fatigue crack growth.

Tribology Research User Center (TRUC)

contact: Peter Blau, Leader, blaupj@ornl.gov, 865/574-5377

MSU researchers collect wear data for improved forging die models

Research problem: To produce wear rate data for a series of candidate die steels in support of powder process modeling efforts

Implications: Enable the development of improved models for forging die wear and material selection in the production of lightweight powder metallurgy parts for automobiles

Description of Work: Professor Seong J. Park and his new Ph.D. student Wei Li (Fig. 35), from the Mississippi State University's Center for Advanced Vehicle Systems (CAVS), worked with TRUC staff members Dr. Peter Blau and Dr. Jun Qu to develop a new test method to assess the wear of hardened steel forging die materials against lightweight alloy powders. It is important both from the standpoint of economics and also die material selection to have dependable die life models, and the MSU researchers sought help through the HTML user program to obtain data that would support their model development efforts.

A series of aluminum-based alloys and three candidate steels were brought to the HTML for the first phase of the effort, namely, to determine the proper wear testing methodology. Initially, steel-pins were oscillated against workpiece material that consisted of cold-compressed aluminum alloys to simulate the die conditions, but this approach was not successful due to fracture of the compressed powder specimens. Then the alloys were sintered and retested. This time there was too much transfer of the aluminum onto the steel pin to measure its wear (Fig. 36). A third method is being developed in which loose powders in a small cavity will abrade the pin tip. The new approach will be investigated during a follow-on visit. During the MSU visit, a secondary assessment of the relative wear characteristics of the steel pin materials, the ASTM G174 continuous loop abrasion test, was used. Results from that test showed clear differences between the three candidate steels, but the test uses a 30 μm grit-size alumina abrasive belt rather than the alloy powders themselves as the counterface. While loop abrasion data were helpful, development work on a steel-against-powder wear test method continues.

Fig. 35. Prof. Park (right) and Mr. Li adjust a specimenon the reciprocating wear test system.

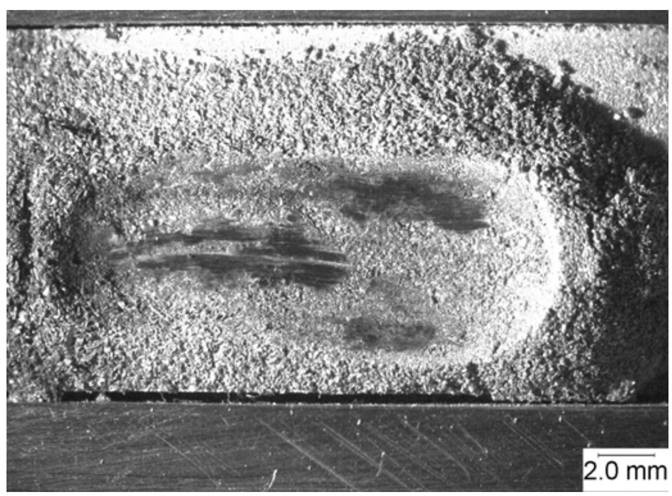


Fig. 36. Wear debris accumulated on a pressed-powder specimen and transferred aluminum to the steel pin. Therefore, the wear of the steel pin could not be measured and a new test method is being developed.

Case Western project studies effects of mating material on wear of carburized stainless steel

Research problem: To investigate the friction and wear compatibility of various counterface materials against a novel, low-temperature-carburized stainless steel

Implications: Expand applications for a new, award-winning surface hardening process for stainless steels by identifying which of several mating materials is most suitable for bearing applications

Description of Work: The Department of Energy's Industrial Technologies Program previously sponsored a research partnership that included the Swagelok Company, Case Western Reserve University (CWRU), and the Oak Ridge National Laboratory. A novel, low-temperature colossal super-saturation process for hardening austenitic stainless steels (known as LTCSS) was developed. Its combination of outstanding corrosion resistance and low wear earned the team members ASM International's prestigious Engineering Materials Achievement Award for 2006. The current user project follows up on that work to expand the potential application of the surface treatment to other bearing material systems.

Lucas O'Donnell (Fig. 37), a master's student with Prof. Arthur Heuer of CWRU, worked with the HTML's Dr. Peter Blau to conduct sliding friction and wear experiments on the LTCSS against three materials: polycrystalline ceramic (alumina), tool steel used in bearing applications (M-50), and cobalt-bonded carbide (WC) composite commonly used in tooling. A series of both unidirectional and reciprocating sliding tests were performed under non-lubricated conditions. Results indicated excellent low-wear when the LTCSS stainless steel was paired with alumina. M-50 (Fig. 38) and WC exhibited higher wear rates, both on the treated surface and on themselves. Friction data showed that while the wear rates were favorable, steady-state friction coefficients were relatively high, suggesting that future applications for the material would require lubrication. O'Donnell will continue his studies of the wear surfaces at CWRU, and a joint paper is planned.

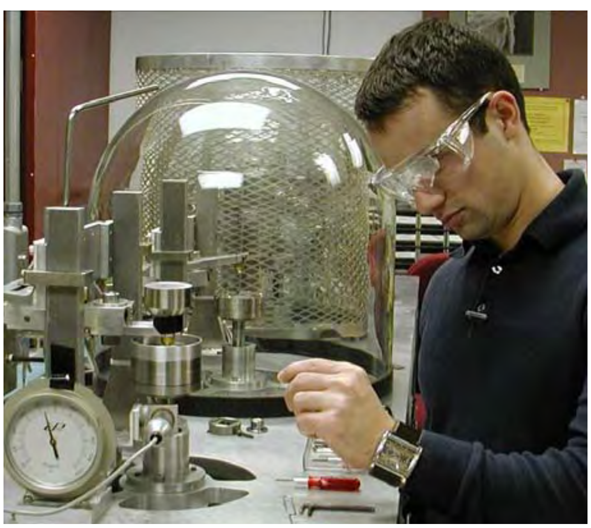


Fig. 37. Lucas O'Donnell prepares to start a new pin-on-disk friction and wear test.

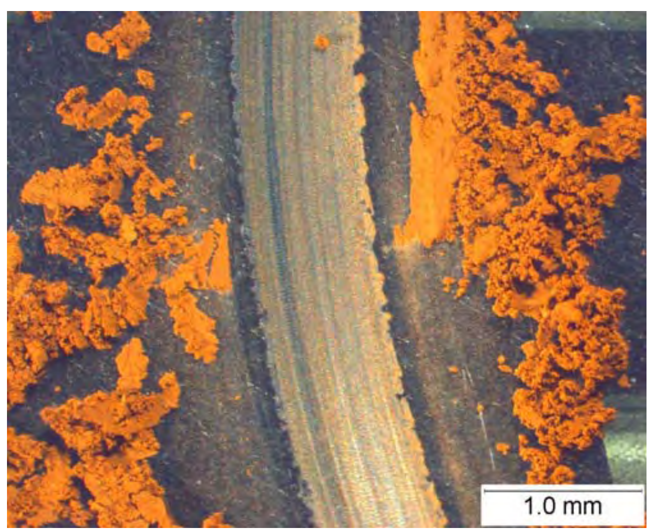


Fig. 38. Sub-micron oxidized wear particles surround the sliding path on an LTCSS treated disk that was slid against M-50 tool steel.

University of Tennessee researchers study friction and wear characteristics of bulk metallic glasses

Research problem: To evaluate tribological properties of Zr-based bulk metallic glass (BMG) of as-cast, annealed, crept, and deformed states

Implications: Evaluate potential usage of Zr-based BMG as bearing materials for transportation applications

Description of Work: Ph.D. student Feng Jiang from University of Tennessee (Fig. 39), worked with TRUC staff member Dr. Jun Qu to evaluate the tribological properties of Zr-based bulk metallic glass (BMG). The mechanical properties of Zr-based BMG have been well-studied; however, publications on their tribological characteristics are rare. An investigation was conducted on the friction and wear behavior of a bulk-metallic glass (BMG) known as Vit 105, $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10.0}Ti_{5.0}$. BMGs in as-cast, annealed, crept, and deformed states were rubbed against AISI 52100 bearing steel using a ball-on-flat reciprocating sliding tribo-tester. Tests were conducted at room temperature and no lubricant was applied. The wear resistance in ascending order was deformed, crept, annealed, and as-cast. Examination of the worn surfaces using optical and electron microscopy and energy-dispersive spectroscopy (EDS) indicated both abrasion and oxidation effects contributed to the wear process. The wear resistance of BMG is influenced by the hardness, toughness, and the amount of free volume associated with the thermo-mechanical stress history. Results of this work were accepted for presentation at the TMS Annual Meeting in New Orleans, in March 2008.

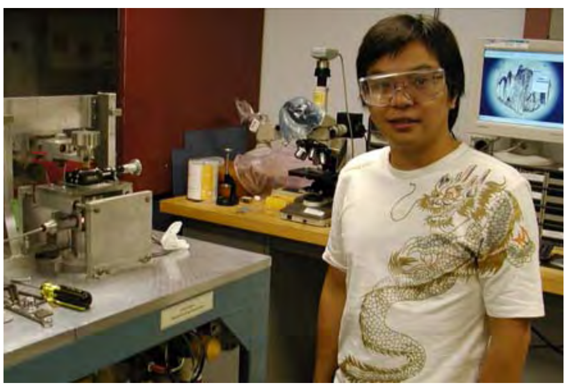


Fig. 39. Feng Jiang prepares to conduct a ball-on-flat friction and wear test.

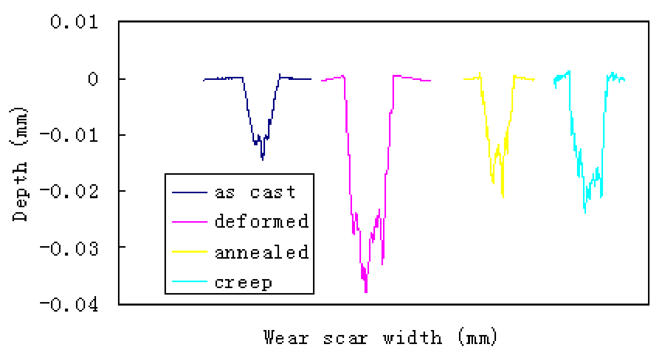


Fig. 40. Comparison of wear scar cross-section profiles.

IAP researchers investigate titanium composites for high temperature bearings

Research problem: To determine whether additions of TiC hard particles will reduce the wear of titanium sufficiently to enable its use in elevated temperature bearings

Implications: Reduce the weight of bearing races by substituting a titanium-based composite for a steel bearing material

Description of Work: This TRUC user project focused on an experimental titanium-based composite as a promising candidate material for high temperature bearing races. Professor Ratnasingham Sooryakumar, representing IAP Research, Inc., a Dayton, Ohio firm, worked with TRUC staff members Peter Blau and Jun Qu to conduct room temperature and elevated temperature sliding friction and wear measurements on three titanium-based metal matrix composites (TiMMCs) containing varying amounts of titanium carbide (TiC) as hard, reinforcing particles. In a past HTML project, it was shown that the elastic modulus, a measure of the stiffness of the material, was increased by over 50% when 25% by volume of TiC was incorporated into the Ti. TiMMCs containing 6, 12, and 25% TiC were tested in sliding wear at both room temperature and 450°C using the TRUC's high-temperature pin-on-rotating disk apparatus. The pins were made from the TiMMCs and the disk specimens were composed of M-50 tool steel, a common ferrous bearing alloy.

In selecting mating pairs of bearing materials, it is important that the total wear of the system – both counterfaces – is kept to a minimum; therefore, both the pin and disk wear behavior was of interest. In a series of tests at high loads and 450°C, it was determined that additions of 25% TiC reduced the steel counterface wear by a factor of about two compared with the 6% TiC material (see Fig. 41). Because the adherent debris on the tip of the pin specimens prevented accurate measurement, only in the case of 25% TiC was there measurable pin wear. The friction coefficients were not low enough to enable the use of the material without a lubricant of some kind, but several kinds of solid lubricants can perform in the 450°C range. The selection of the most tribologically compatible solid lubricant with the TiMMC materials remains a subject for further investigation.

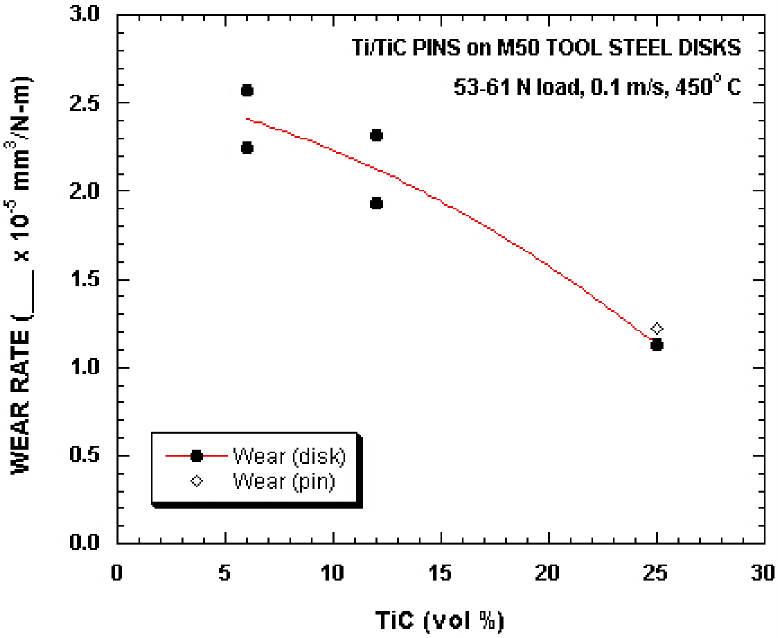


Fig. 41. Effects of TiC particle content on the wear of M-50 tool steel sliding against three TiMMCs.

Thermography and Thermophysical Properties User Center (TTPUC)

contact: Ralph Dinwiddie, Leader, dinwiddierb@ornl.gov, 865/574-7599

Purdue University studies thermal interface materials to determine optimal particle size

Research problem: To understand thermal boundary resistance of nanoparticles in an epoxy matrix

Implications: Improved thermal management of materials for automotive applications

Description of Work: Purdue graduate student Rushabh Kothari (Fig. 42, next page) and HTML research Dr. Hsin Wang evaluated thermal boundary resistance of an epoxy/silica particulate composite. The thermal conductivity of the neat epoxy and the composite sample was determined using the Hot Disk System.

PURDUE UNIVERSITY

The thermal boundary resistance between particles and matrix was back-calculated using the Hasselman et al. equation. a_k is considered as the Kapitza radius. If a_k exists ($R_c \neq$ zero), that means that there is a finite boundary thermal resistance of value R_c acting through the radius a_k between particles and matrix. The higher the value is of boundary resistance, the lower the effective conductivity of composite system will be. The following diagram (Fig. 43) represents the physical significance of non-zero a_k.

The measured average values of the conductivities for epoxy/silica particulate composite are as follows:

Matrix (Epoxy): 0.248 W/m-K
6% 20-nm Silica-Epoxy Composite: 0.260 W/m-K
6% 45-nm Silica-Epoxy Composite: 0.262 W/m-K

The boundary resistances for 20nm and 45nm particles were found to be:

Boundary resistance for 20nm particles: 3.02 10^{-9} m^2/K-W ; a_k = 0.75 nm
Boundary resistance for 45nm particles: 2.27 10^{-9} m^2/K-W ; a_k = 0.57 nm

These values can be used to predict effective conductivity of the particulate composite system for 20nm and 45nm silica particles at many different volume fractions. In Fig. 44, it can be seen that thermal properties of the composite reinforced with 20-nm silica particles is lower than that of the composite reinforced with 45-nm particles because there are more particle-matrix boundaries in the composite with the same concentration of smaller particles that will scatter phonons. Unless the particle-matrix bond can be improved at the interface (e.g., by chemical treatment) the use of small-sized particles will result in lower thermal properties of the composite.

Fig. 42. Purdue graduate student Rushabh Kothari preparing to analyze silica composite.

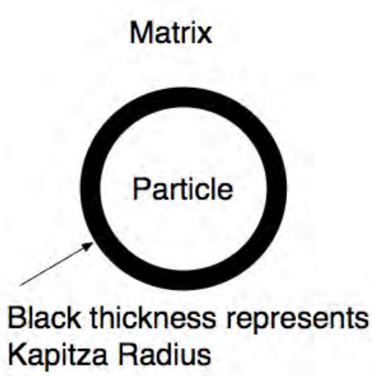


Fig. 43. Diagram of physical significance of non-zero a_k.

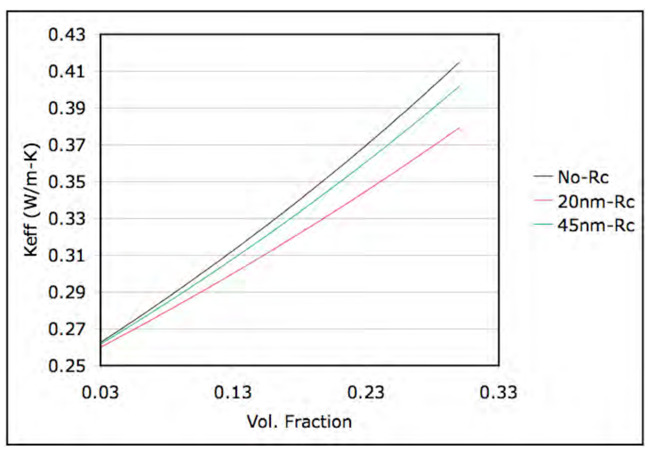


Fig. 44. Prediction of effective conductivity for 20nm and 45nm silica composite.

Materials and Analysis User Center

Hitachi's next-generation field-emission TEM installed in HTML

The HTML announces the successful installation of a new electron microscope, recently purchased from Hitachi High-Technologies America. The HF-3300 TEM is a next-generation cold field emission instrument, which replaces the venerable HF-2000 field emission TEM that serviced our research programs for more than 15 years. The new instrument offers a number of advanced capabilities beyond those of the HF-2000, including 300kV (vs 200kV) operation which enables higher resolution imaging, much better energy-dispersive x-ray analysis sensitivity, and a state-of-the-art electron holography capability employing two electron biprism devices. In addition, images are recorded on a digital camera system providing 4 megapixel resolution (vs 1 megapixel) with 4 times the dynamic range of our old camera. Another very useful aspect is the ability to operate the microscope effectively at only 100kV, which is a requirement for beam-sensitive materials such as carbon nanotubes. Because all of the microscope functions are computer-controlled (e.g., aperture drives), the instrument will ultimately be accessible from anywhere in the world.

Fig. 45. Dr. Jane Howe at the controls of the Hitachi HF-3300.

The microscope was purchased by pooling funds from five different programs: HTML User Program (Vehicle Technologies Program), two Office of Electricity Delivery and Energy Reliability programs (Distributed Energy and Superconductivity), EERE's Hydrogen, Fuel Cells, and Infrastructure Technologies Program, and the BES-SHaRE program. This is the first HF-3300 to be installed in the United States (and only the second one worldwide). After arriving at ORNL in late October 2007, installation was completed in a scant five weeks, with the performance of the HF-3300 meeting all specifications and surpassing most of them. The microscope has supported ORNL research projects since early December 2007, and it has been available to outside users via the HTML, SHaRE and CNMS user programs. HTML research staff member Dr. Jane Howe (Fig. 45) has principal responsibility for the operation of the new instrument. Figure 46 is an example image from the 4 megapixel CCD camera.

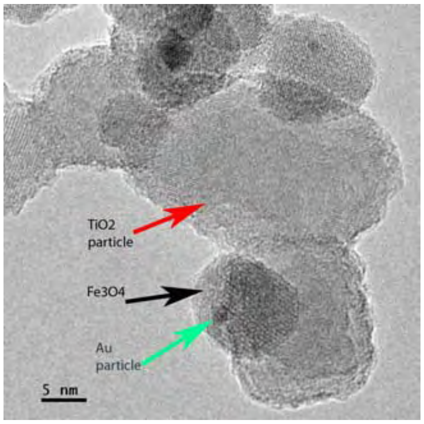


Fig. 46. HF-3300 image of catalyst with 2nm Au particles sitting on larger iron oxide particles supported by titanium dioxide.

Protochips Co. initiates project to develop novel **in situ** *heating capability for the ACEM*

Protochips Co. (Raleigh, NC) provides novel TEM heater "chips" that offer great promise for ultra-high resolution in-situ heating experiments in the aberration-corrected electron microscope. This work forms the foundation for a new HTML thrust into *in situ* studies of microstructural changes with temperature (and ultimately gas environment) to support programs in catalyst development, as well as thermoelectrics, photovoltaics, and battery materials.

Drs. David Nackashi and John Damiano from Protochips visited the HTML on a new project, partially funded by an SBIR grant, to collaborate with Dr. Larry Allard on developing techniques for *in situ* microscopy at ultra-high resolution. Protochips' heater "chip" is designed to be heated to temperatures well in excess of 1000°C, while remaining ultra-stable at the atomic level. The heater area is a thin ceramic membrane 500 microns square, mounted on a 3-mm diameter silicon chip that mimics a standard TEM specimen geometry. The chip is mounted in a special holder fabricated by HTML consultant Dr. Wilbur Bigelow (professor emeritus, University of Michgan) to enable electrical connections to a programmable power supply. The ceramic membrane can be heated to 1100°C in 1 millisecond and cooled essentially just as rapidly. Because of the low mass and symmetrical design, there is virtually no thermal drift, after just a few seconds at temperature. This allows images such as the platinum particle, shown below on an alumina support, to be recorded at an original magnification of 10Mx and 1000°C. <u>This is the first-ever recorded image that shows such detail at high temperature.</u> The images in Fig. 47 display an ordered alloy structure (not pure Pt) that is still being analyzed. Several HTML users involved with catalyst studies, such as UOP Co., University of Texas-Austin, Vanderbilt, and the Pacific Northwest National Laboratory, have had initial success with the Protochips *in situ* heating capability being developed.

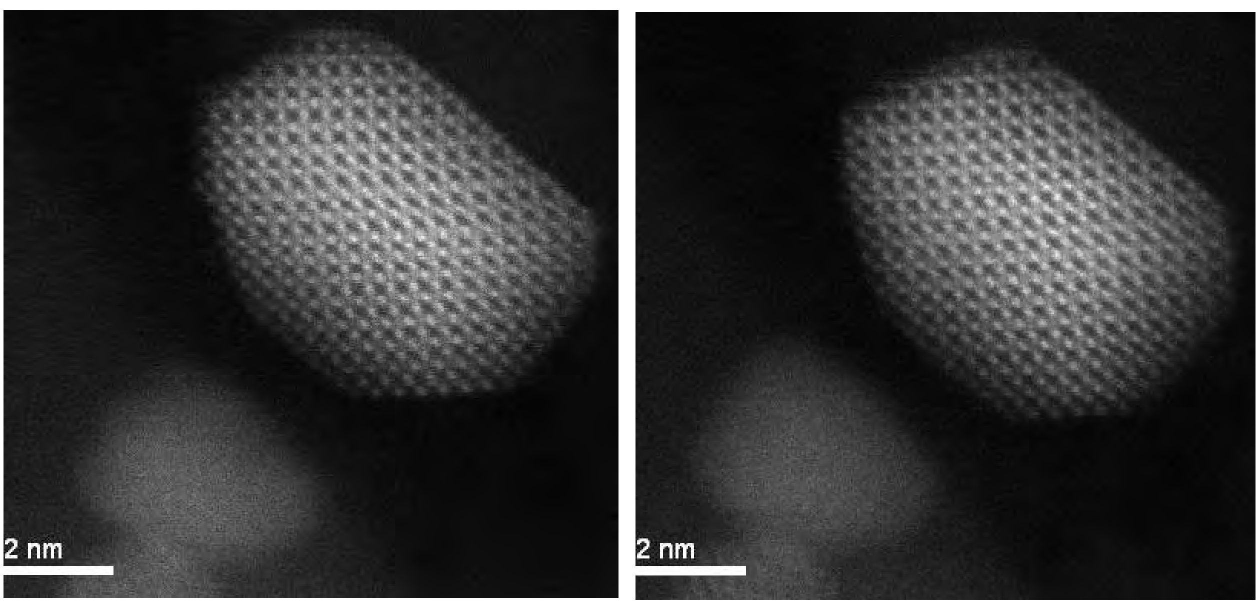


Fig. 47. Platinum particle on alumina support, at nominal 1000°C, showing ordered structure suggesting compound formation (oxide?). Two HA-ADF images from a 20-image exposure series.

Dr. Larry Allard visits BASF Company, Woodbridge, New Jersey

Following his invited talk on catalyst characterization via electron microscopy at the June 2007 meeting of the North American Catalyst Society in Houston, TX, Dr. Larry Allard was invited in December to visit the BASF Company research laboratory in New Jersey. The primary purpose of the visit, hosted by Dr. Rostam Madon, was to present a seminar to more than 30 scientists and managers, entitled “Looking at Atoms with the Aberration-Corrected Electron Microscope.” Dr. Allard also toured the well-equipped BASF laboratories. Until recently this research facility was owned by Englehard Catalyst Company. As a result of the visit, Dr. Allard is working with BASF scientists Drs. Junmei Wei and Xingtao Gao to develop suitable non-proprietary and proprietary HTML User project proposals.

Residual Stress User Center

Raymond Orbach, Under Secretary for Science at the U.S. Department of Energy, Visits HTML’s NRSF2 and observes University of Tennessee-Martin faculty/student team map stresses in loaded gears

As previously reported in FY2007, the recommissioned neutron residual stress mapping instrument (NRSF2) at the High Flux Isotope Reactor has been the site of world-class research in support of DOE’s goal of transforming science to energy via the HTML user program. During Under Secretary Orbach’s visit on January 4, 2008 (Fig. 48), RSUC leader Cam Hubbard and Prof. Robert LeMaster and his student Jon Kolwyck from the University of Tennessee-Martin explained how the NRSF2 is uniquely helping expand the fundamental knowledge of how stresses are distributed in loaded gears, which will aid industry to increase power density while simultaneously reducing weight and extending gear life. Sponsored by the American Gear Manufacturers Association, the UT-Martin team designed and constructed the static load frame that put a pair of gears under applied load while enabling the neutron strain measurements at NRSF2. The January research completed mapping of the strain components at the critical bend radius (the site of likely gear tooth failure). Figure 49 (next page) shows the results of low and high loading. Further work will measure d-spacing in two orthogonal directions and determine the stress free d-spacing as a function of depth.

Fig. 48. Prof. Robert LeMaster of Univ. of Tennessee-Martin (left) and Dr. Camden Hubbard, Leader of HTML’s Residual Stress User Center (center), discuss with Dr. Raymond Orbach the capabilities of the neutron residual stress mapping instrument (NRSF2) as well as the study of statically loaded gears.

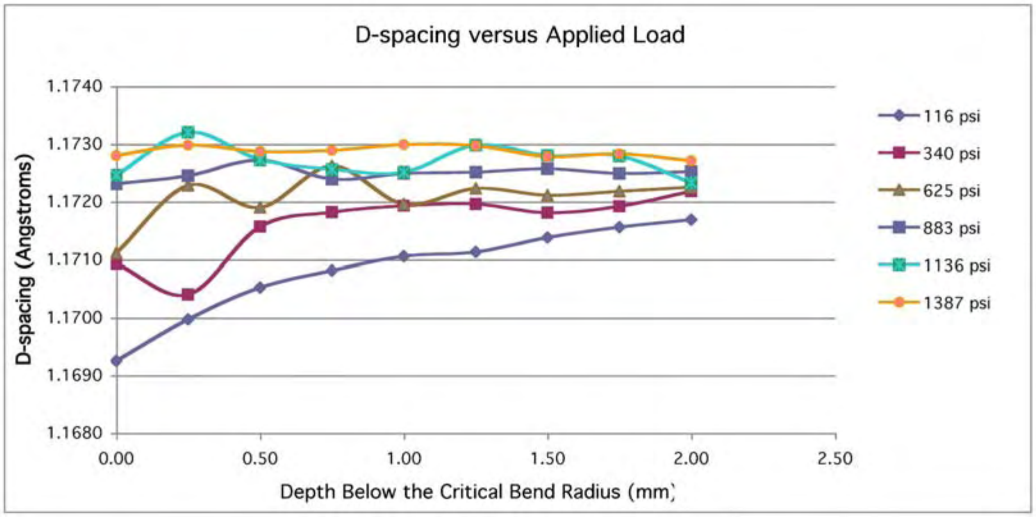


Fig. 49. D-spacings versus applied load and depth below the critical bend radius. The low load (116 psi) case shows the effect of compressive stresses due to carburization and grinding while the high load case (1387 psi) shows that these beneficial compressive stresses are balanced by this load.

Diffraction User Center

Workshop on x-ray diffraction data analysis hosted by the HTML

In October 2007 the HTML hosted a workshop at ORNL on collection and analysis of x-ray diffraction data. The workshop was co-organized by PANalytical, Inc., a leading manufacturer of laboratory x-ray diffraction systems, and the instructors included HTML staff members Drs. Tom Watkins and Andrew Payzant, former HTML postdoc Scott Speakman (now at MIT), and Drs. Jeff Nicolich and Thomas Degen from PANalytical. Approximately 20 attendees (Fig. 50 photo below) from industry, academia, and ORNL received training on advanced methods for analysis of x-ray and neutron diffraction data.

Fig. 50. Participants in x-ray diffraction data analysis workshop. HTML instructors were Dr. Tom Watkins (blue jacket, 4th from left) and Dr. Andrew Payzant (4th from right).

Mechanical Characterization & Analysis User Center

Georgia Tech graduate student completes Ph.D. dissertation research at the HTML

Dr. Christopher Green, an HTML user from Georgia Tech, successfully defended his Ph.D. dissertation in October 2007 (Fig. 51, next page). His characterization work on mica compressive seals for solid oxide fuel cells earned him invitations to present papers at international conferences in Europe. The majority of his research was conducted under an HTML user project in the MCAUC in collaboration with Dr. Edgar Lara-Curzio and Rosa Trejo.

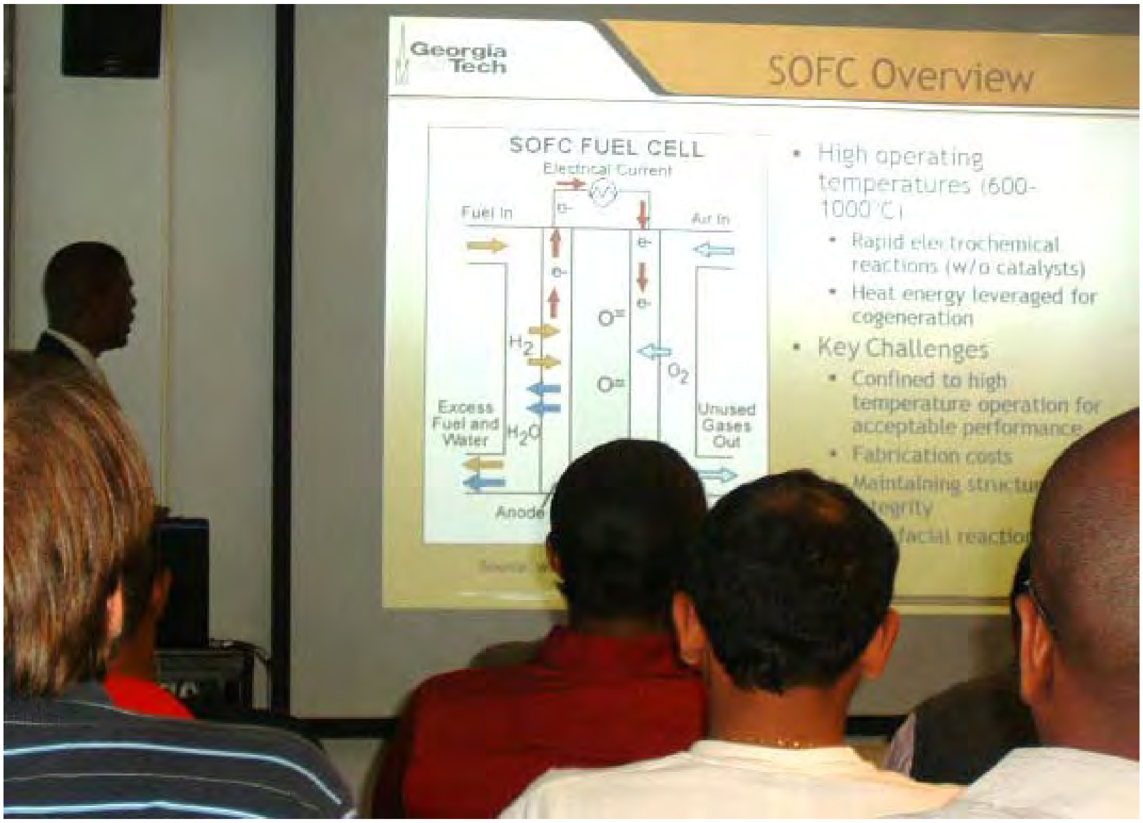


Fig. 51. Georgia Tech graduate student Chris Green (standing at left background) explains a point during his successful defense of his Ph.D. dissertation seals in solid oxide fuel cells.