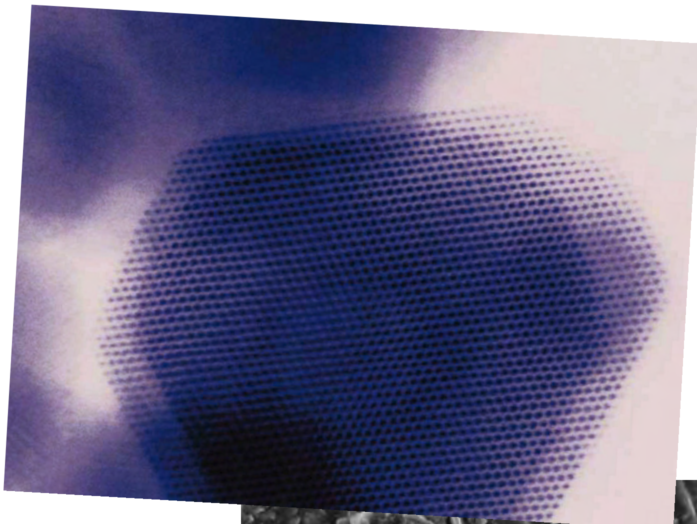
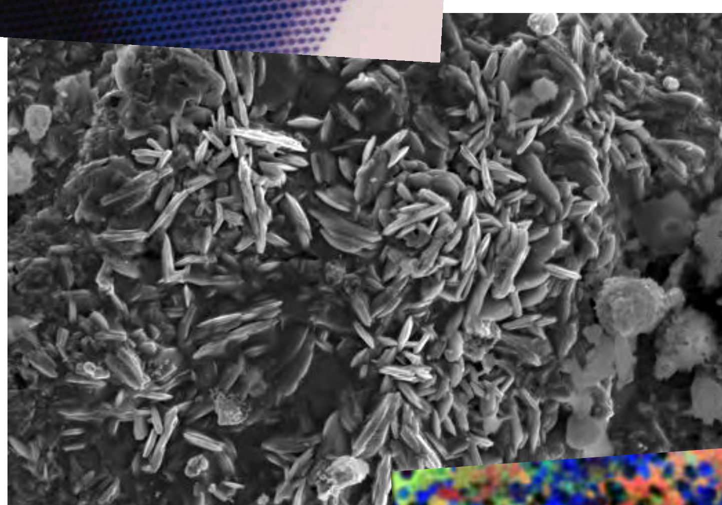
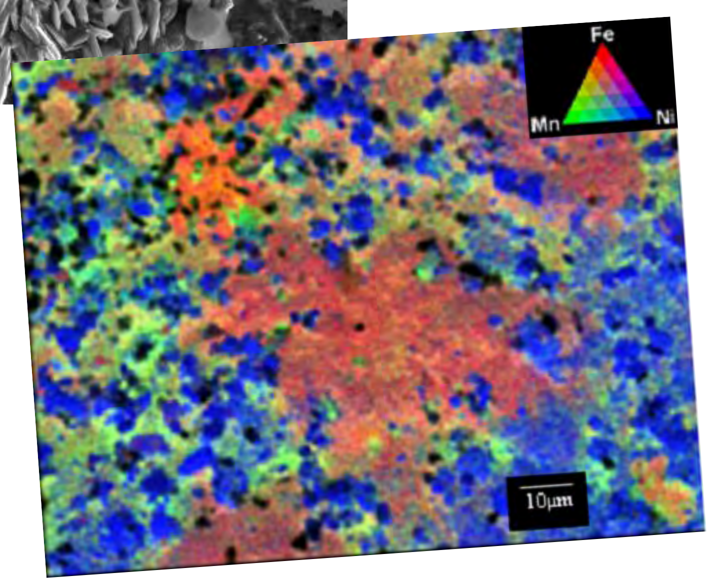
Quarterly Highlights
High Temperature Materials Laboratory
Oak Ridge National Laboratory
January – March 2008

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







html
HIGH TEMPERATURE MATERIALS LABORATORY

Diffraction User Center (DUC)

contact: Andrew Payzant, Leader, payzanta@ornl.gov, 865/574-6538

Palladium-copper alloys for hydrogen separations studied by *in situ* x-ray diffraction

Research problem: To better understand the formation of palladium-copper alloys, which have been proposed as a more sulfur-tolerant alloy for hydrogen separation from syngas

Implications: Improved palladium alloy based supported membranes may prove essential to the success of the proposed "hydrogen economy" thereby reducing carbon emissions and also our dependence on foreign oil.

Description of Work: Natalie Pomerantz, a doctoral student from Dr. Ma's group at Worcester Polytechnic Institute traveled to ORNL to work with Dr. Andrew Payzant to collect new data on the Pd-Cu system. The unique *in situ* high-temperature x-ray diffraction instruments at the HTML enabled quantitative analysis of time-resolved data collected as Cu + Pd precursor films (electroless plated on porous stainless steel substrates) were reacted at high temperature to form the hydrogen separation membrane alloy. This project focused on understanding the conversion from a Cu-plated Pd film to the fully reacted Pd-Cu alloy film. The frontmost scan in Fig.1 shows the (111) peaks of separate FCC Pd and FCC Cu phases at 27°C. In the second scan, at 500°C, these peaks are shifted to the left, primarily due to thermal expansion. The subsequent scans are all at 500°C and reveal the time-dependent evolution of the FCC Cu-Pd alloy phase. An intermediate Cu-rich BCC alloy phase is also observed, which is to be expected from the phase diagram as the alloy composition evolves from Cu-rich to Pd-rich (see Fig. 2). By understanding the kinetics of formation of Pd-Cu alloys it will be possible to optimize the design of membranes for hydrogen separation, which are essential to the success of the proposed "hydrogen economy" thereby reducing carbon emissions and also our dependence on foreign oil.

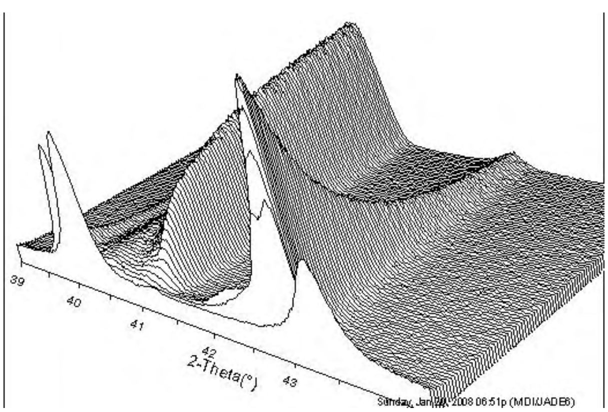


Fig. 1: A typical high-temperature x-ray diffraction data set, which displays a series of x-ray scans of a Cu-Pd bilayer film as a function of time and temperature.

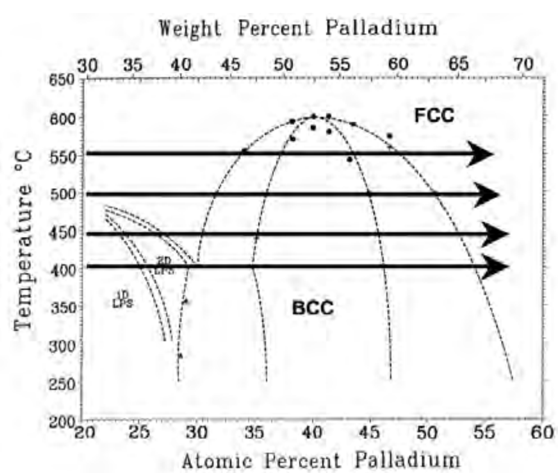


Fig. 2: The Cu-Pd phase diagram, with arrows indicating the progression from a Cu-rich surface layer to a fully evolved 60%Pd-40%Cu alloy. HTXRD experiments were performed at the four temperatures indicated on the plot.

University of Tennessee monitors carbon fiber morphology during stabilization and carbonization

Research Problem: Determine range of conversion parameters where critical morphological changes occur during various steps of carbon fiber production

Implications: Development of low-cost carbon fibers

Description: Replacement of steel and aluminum body panels and chassis components with structurally equivalent carbon fiber composites offers up to 68% weight reduction, resulting in savings of up to 40% in gasoline consumption. Although the relatively high cost of carbon fibers has constrained their use in automotive applications, the use of low-cost precursors (e.g., textile-grade PAN and lignin-based feedstocks) could result in carbon fibers with a cost of less than $5/lb and their increased use in automobiles.

Using the high-temperature x-ray diffraction (XRD) facility at the HTML, Dr. Amit Naskar of the University of Tennessee, Knoxville and the HTML's Dr. Andrew Payzant investigated the evolution of morphology of carbon fibers as a function of time, temperature and precursor composition (chemical structure). Figure 3 displays representative 2θ profiles of wide-angle diffraction data for PAN-based fibers collected at different time intervals under controlled thermal and atmospheric conditions. Ongoing work is focused on investigating the effect of applied tensile loads during conversion on the morphological evolution of polymeric carbon precursors. Results from this HTML user project will provide the knowledge base for the carbon fiber processing window, aid in exploring alternative low-cost carbon precursors such as lignin-based fibers, and enable the commercialization of low-cost carbon fibers.

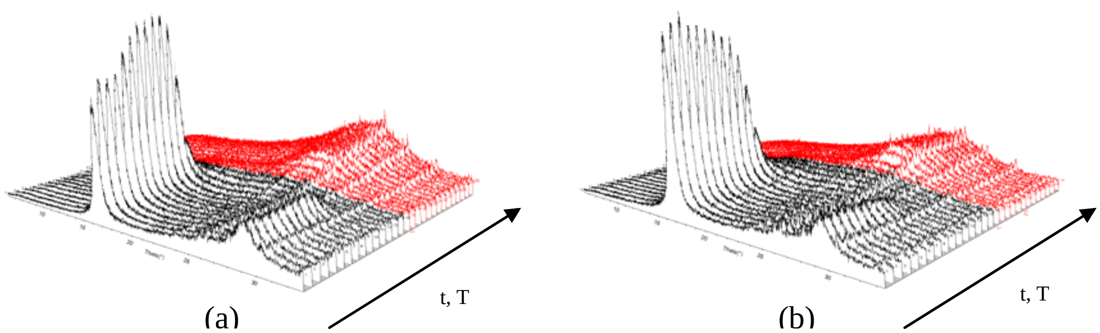


Fig. 3. Morphological evolution as observed by online XRD during heat treatment of (a) as-spun PAN and (b) radiation modified PAN fibers.

Other Diffraction User Center News

Payzant, Hubbard attend ICDD Spring Meeting

The 2007 Spring Meeting of the International Center for Diffraction Data (ICDD) was held at ICDD headquarters in Newtown Square, PA in March. The HTML was represented by Cam Hubbard, who completed his term on the Board of Directors, and by Andrew Payzant, who is continuing as chair of the Non-Ambient Diffraction Sub-Committee.

DUC staff member featured scientist on Missouri schools videoconference

On March 24, Dr. Claudia Rawn was featured on the "Meet the Scientist" videoconference broadcast to upper elementary and middle school classrooms in Missouri. Sponsored by the Missouri Research and Education Network (MORENet) at the University of Missouri, the program focused on demonstrating how ORNL scientists apply the scientific inquiry process to actual research activities. Using her research on gas hydrates as an example, Dr. Rawn discussed the experimentation process of setting objectives, executing the research, and analyzing the data gathered. There are plans to broadcast "live" from a laboratory the next time.

Materials Analysis User Center (MAUC)

contact: Larry Allard, Leader, allardlfjr@ornl.gov, 865/574-4981

University of Texas-Austin project reveals surface segregation and ordering in Pt-Co fuel cell catalyst nanoparticles

Research problem: To investigate microstructure of Pt-Co nanoparticles used in fuel cell catatalyst applications

Implications: Understanding the origins of enhanced surface activity of Pt_3Co nanoparticles vs. pure Pt may lead to further improvements in fuel cell catalysts.

Description of Work: Pt-based alloys such as Pt_3Co are promising candidates as catalysts for fuel cells because they exhibit higher catalytic activity than pure Pt and reduce the use of expensive Pt. The use of proper heat and acid treatments has produced surfaces of bulk Pt_3Co with higher activity than pristine Pt_3Co. In these bulk Pt_3Co surfaces, surface characterization has shown segregation by Pt atoms. Although enhanced activity has been reported for Pt_3Co nanoparticles relative to pure Pt nanoparticles, it is not clear if Pt segregation exists on the Pt_3Co nanoparticle surfaces, nor is the origin in activity enhancement well understood. Prof. Paulo Ferreira of the University of Texas-Austin is working with the HTML's Dr. Larry Allard to study Pt_3Co nanoparticles subjected to two different types of treatments: an acid leaching treatment and a heat treatment in vacuum at 1000K, using the aberration-corrected electron microscope (ACEM) to image particle ultrastructures in both treatments. Figure 4 (left) is a dark-field ACEM image showing a typical 2-3 nm acid-leached nanoparticle. Varying intensities in atomic columns are interpreted as resulting from both the loss of Co due to the acid treatment and also the difference in atomic number between Co and Pt. The nanoparticle in Fig. 4 (right) resulted from growth during the high-temperature treatment in vacuum. It shows an ordered structure, with an indication of segregation of Pt to the first 3 surface layers, as suggested by the schematic of the structure shown below in Fig. 5. Although

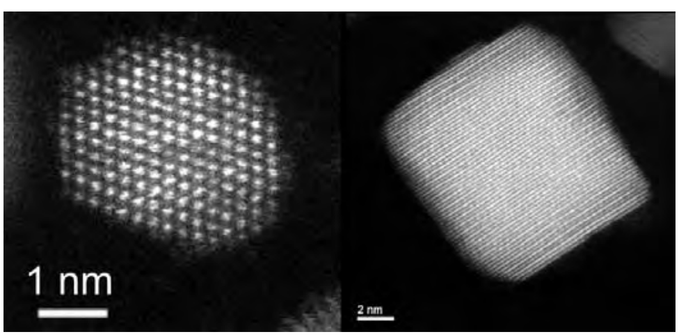


Fig. 4. (left) Dark-field ACEM image of Pt3Co acid-leached nanoparticle; (right) Dark-field ACEM image of Pt3Co nanoparticle following high-temperature treatment at 1000K in a vacuum.

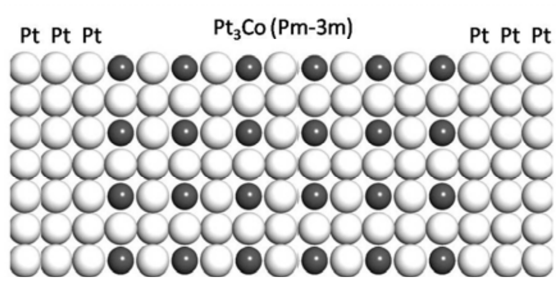


Fig. 5. Schematic of Pt_3Co nanoparticle after annealing at 1000K.

Pt segregation and the development of the "Pt-skin" surface structure is well known on Pt polycrystalline and single-crystal alloy surfaces upon high-temperature annealing, this is the first time that Pt segregation has been reported to occur on the surface of Pt_3Co nanoparticles of ~5nm upon annealing at 1000K.

Capstone Turbine characterizes microturbine hot-section materials

Research Problem: Determine the detrimental effect of microturbine operating conditions on the HR-120 alloy recuperator core

Implications: Reduced recuperator core effectiveness due to accelerated oxidation can adversely affect microturbine efficiency and power output

Description of Work: Capstone has thousands of C60 and C65 microTurbines installed around the world. In the spring of 2005, Capstone replaced the 347 stainless steel recuperator core with an HR-120 alloy recuperator core in order to increase the life of the recuperator core to ~80,000 hours. Since the implementation of this material change, Capstone has continued to test HR-120 alloy samples in a microturbine operating at ~100F° above the specified Turbine Exit Temperature (TET) set-point. Samples are removed from the engine approximately every 1,500 hours for characterization at ORNL. Capstone's Wendy Matthews utilized the electron microprobe (JEOL 8200 EPMA) and scanning electron microscope (Hitachi S-3400 Environmental SEM) expertise available at HTML to examine the latest HR-120 alloy specimens, which had accumulated ~17,500 hours of exposure. HTML staff members Karren More and Larry Walker assisted in identifying the oxide constituents present in the exposed HR-120 alloy samples. Electron microprobe elemental mapping of the mounted cross-sectional samples revealed an inner chromia layer with an outer oxide layer containing Fe, Mn, and Ni, consistent with previously characterized samples. The surface of the oxide scale was examined in the SEM (Fig. 6), and elemental mapping revealed the presence of what appears to be mixed oxides of Fe, Mn, and Ni (Fig. 7). X-ray diffraction analysis was also performed, and results are being analyzed. The availability of durable microturbine components will enable their widespread use in applications in transportation. For example, Capstone recently announced that it has received an order for 150 microturbines to be deployed in ECOSaver IV hybrid electric

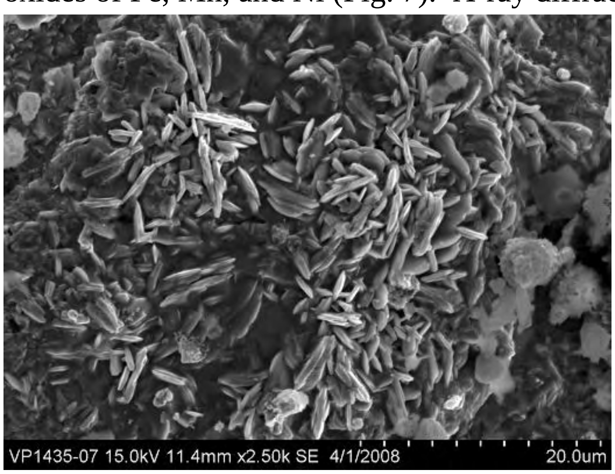


Fig. 6. SEM image of HR-120 alloy core sample from recuperator with ~17,500 hours of exposure.

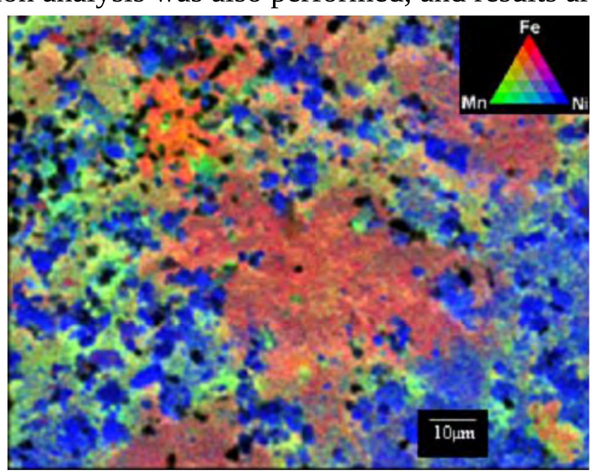


Fig. 7. Elemental mapping of HR-120 alloy recuperator core sample showing mixed oxides of Fe, Mn, and Ni.

buses manufactured by DesignLine International. DesignLine buses are constructed with advanced extruded aluminum technology. In recent product demonstrations the ECOSaver IV hybrid buses equipped with Capstone turbines have seen up to a 100% improvement in fuel economy over a traditional diesel bus, which equates to annual fuel savings of up to 6,000 gallons per vehicle.

University of Missouri-St. Louis uses novel ACEM *in situ* heating capability to study Pd/ZnO nanocatalysts for hydrogen production

Research problem: To investigate mechanisms of formation of PdZn nanoparticles in Pd-based fuel cell catalysts for hydrogen production

Implications: Understanding the synthesis-structure-performance relationship of a Pd/ZnO model nanocatalyst is necessary for improved catalysts for methanol reforming for on-board hydrogen production.

Description of work: The formation of PdZn alloy nanoparticles has been proposed to explain the catalytic performance of Pd/ZnO catalysts. The structure and the formation processes of the PdZn alloy nanoparticles, however, are not well understood, so Dr. Jimmy Liu and his graduate student Jinfeng Wang (Fig. 8) collaborated with Dr. Larry Allard in a study using the HTML's aberration-correction electron microscope (ACEM). ZnO nanoribbons were used as the support material. The HA-ADF image shown below left (Fig. 9) reveals the presence and dispersion of Pd-containing molecular species on the as-prepared ZnO surfaces; individual Pd monomers (bright dots) are seen. This material was heated *in situ* for varying times, using a novel new heating capability the HTML is developing with Protochips Co.

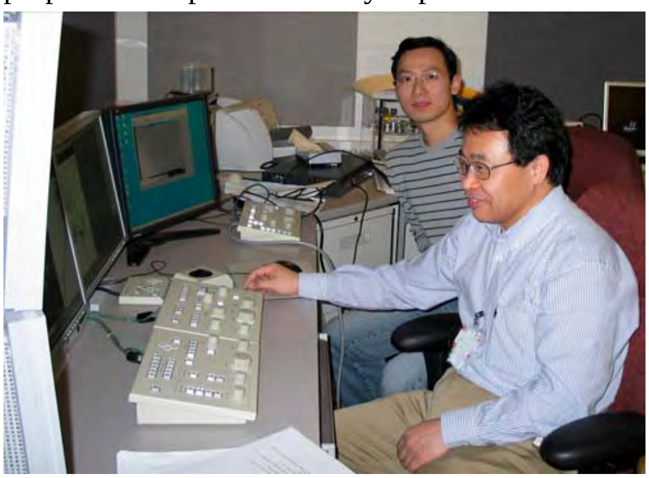


Fig. 8. Prof. Jimmy Liu (foreground) and Jinfeng Wang at the controls of the ACEM.

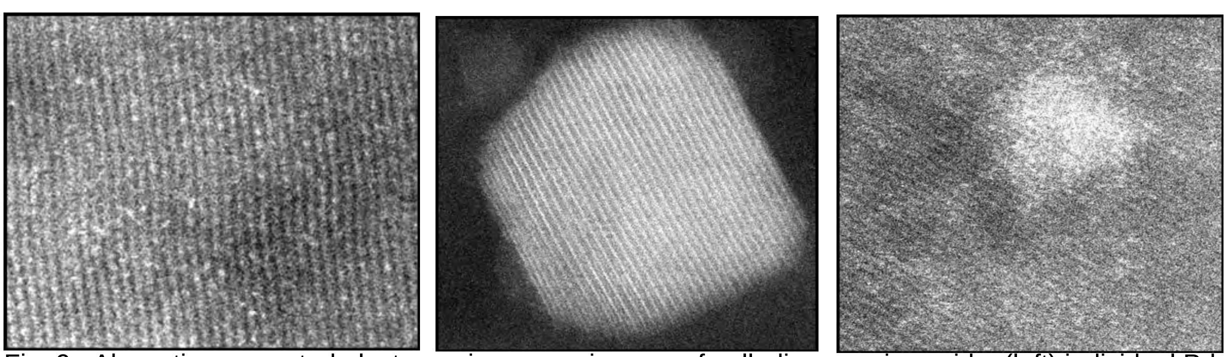


Fig. 9. Aberration-corrected electron microscope images of palladium on zinc oxide. (left) individual Pd monomers (bright dots) on as-prepared ZnO surfaces; (center) PdZn nanoparticle after heating at ±800°C for one hour, showing bright contrasting Pd layers; (right) individual monomers and small clusters of Pd in heated sample.

(Raleigh, NC). The center image in Fig. 9 shows a PdZn alloy nanoparticle after the sample was heated at ±800ºC for one hour; the PdZn alloy nanoparticle displays the Pd layers with bright contrast. Interestingly, individual monomers and small clusters of Pd were also observed, as shown in the right image. The availability of low-cost catalysts has been identified an enabler for the wide-spread commercialization of fuel cells.

The Timken Company investigates rolling contact fatigue wear modes of nanocomposite tribological thin films

Research problem: To improve the performance of wear-resistant coating based upon a clear understanding of its microstructure

Implications: Improve mechanical device energy efficiency and durability by designing mechanical component tribological surfaces with reduced friction and improved wear resistance

Description of Work: Precisely identifying interfaces in hard, thin film tribological coatings may enable the design of new coating architectures with improved adhesion to the substrate and optimized rolling contact fatigue resistance. Such coatings could be used in components that require both low friction and high wear resistance and would reduce parasitic losses in transportation systems. Dr. Ryan Evans of The Timken Company (a manufacturer of friction management and power transmission products) worked with HTML staff members Dr. Jane Howe and Dorothy Coffey to analyze WC/a-C:H coatings after being tested under thin film lubrication rolling contact conditions. Coating samples were fabricated using a plasma discharge vapor deposition process at either a Timken Technology Center or a scaled-up production facility. Transmission electron microscopy (TEM) was carried out on cross-sectional coating samples prepared by focused ion beam (FIB). Figure 10 is a micrograph of multilayers within a WC/a-C:H coating that was found to have excellent adhesion to the substrate under high contact stresses in an application testing. The TEM analysis revealed that the microstructure and architecture of coatings synthesized according to the scaled-up processes were similar to those synthesized in the laboratory. This was a very important finding to demonstrate the equivalence of laboratory- and production-scale versions of the coatings. Nanometer-scale materials characterization efforts with FIB/TEM validated new surface engineering solutions that are increasingly used to enhance the durability and performance of macroscopic tribological components.

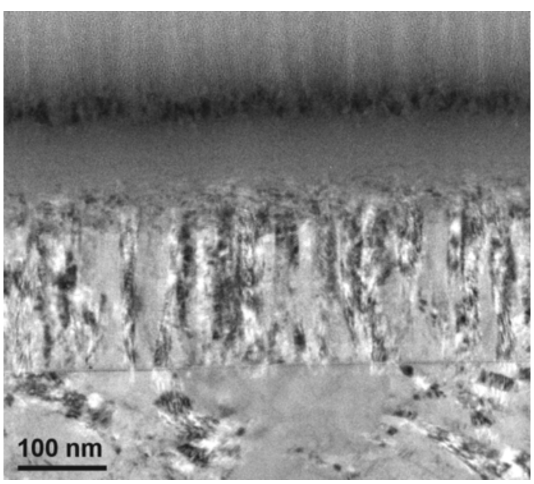


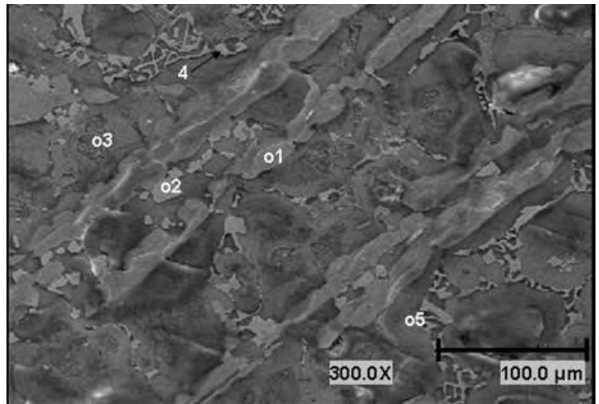
Fig. 10. A micrograph of multilayered WC/a-C:H coating on steel.

Missouri University of Science & Technology studies corrosion in Ni_3Si-based intermetallics

Research problem: To understand the relationship between alloying elements and corrosion resistance of Ni_3Si alloys

Implications: Development of optimized alloy composition for large-scale hydrogen-production technology

Description of Work: The Sulfur-Iodine cycle is a promising candidate for the thermochemical production of hydrogen. This process presents significant challenges because except for very expensive noble metals (e.g. gold or platinum) there are few materials with acceptably low corrosion rates (<20 mpy) in concentrated super heated sulfuric acid. For example, Ni_3Si with minor alloying additions has shown excellent corrosion resistance in hot sulfuric acid. Studies have indicated that a passivating oxide film that is rich in silicon imparts the superior corrosion resistance of this alloy. Unfortunately, Ni_3Si alloys have low ductility and so are difficult to fabricate into useful forms (e.g., tubes and pipes). Professor Richard Brow and graduate student Christopher Larson of the Missouri University of Science & Technology are evaluating new alloy compositions to improve ductility while retaining their corrosion resistance. They visited the HTML to work with Dr. Harry Meyer to understand the role of Ti and Nb additions on the formation of passivating films on Ni_3Si. Toward this end, they obtained detailed three-dimensional elemental and chemical information about the alloy surfaces using Auger and X-ray photoelectron spectroscopy (XPS) in combination with depth profiling (via *in situ* ion sputtering). Figure 11 shows the surface of "Alloy A" (added Nb, no Ti) after a 1-minute exposure to sulfuric acid. Auger electron spectroscopy was used to determine the composition of several different surface phases that are observed. In Fig. 12, Auger depth profiles from two of the areas shown in Fig. 11 (areas 1 and 5) reveal a silicon oxide over-layer on the base material, but with differing thickness. These results indicate the heterogeneous manner in which the oxide layer develops on the Ni_3Si alloy. Similar results



Composition (at.%)

Area	C	O	Si	Ni	Nb
1	0.0	62.7	34.2	3.1	0.0
2	0.0	67.7	27.7	2.4	2.2
3	0.0	67.0	30.4	2.5	0.0
4	0.0	66.9	28.2	3.5	1.4
5	0.0	64.6	32.1	2.0	1.3

Fig. 11. SEM of "Alloy A" after a one-minute exposure to sulfuric acid.

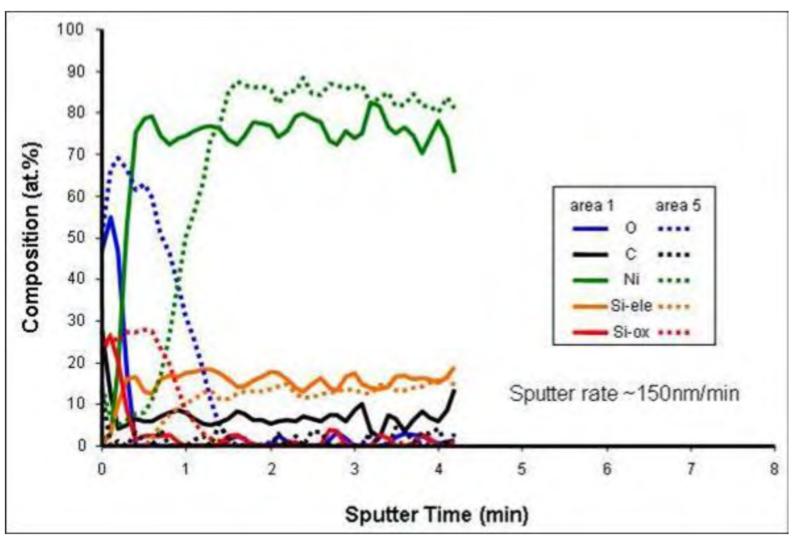


Fig. 12. Auger depth profiles of areas 1 and 5 shown in Fig. 11. Both areas had Si-oxide overlayers, but the thickness of this overlayer in area 5 was ~3 times as large.

were obtained on "Alloy J" (added Nb and Ti) and "Alloy E" (no Nb, added Ti), along with the x-ray photoelectron spectra for all surfaces. Data are currently being analyzed and future work planned. Ni_3Si-based intermetallic alloys may prove to be an enabling material for hydrogen production, and if so, the economic impact of a more efficient way to produce hydrogen for a variety of environmentally-friendly applications will be significant.

South Dakota School of Mines & Technology studies electrospun ceramic nanofibers

Research Problem: To characterize the morphology and structures of electrospun ceramic nanofibers

Implications: Ability to produce electrospun nanofibers with optimized properties for applications as reinforcements in composites and in catalysis.

Description of work: Prof. Hao Fong of South Dakota School of Mines and Technology and postdoctoral researcher Dr. Lifeng Zhang (Fig. 13) are studying the development of electrospun nanofibers for uses in structural and catalytic applications. In this process, spun jets/fibers are elongated up to 10,000 times in less than 50 milli-seconds, effectively aligning the macromolecular chains along the fiber direction. In particular, Fong and Zhang are interested in establishing the relationships between the parameters that control the electrospinning and post-electrospinning processes and the morphological, physical and mechanical properties of electrospun nanofibers. Using the HTML's newly acquired Hitachi HF-3300 TEM/STEM, Dr. Zhang worked with Dr. Jane Howe to investigate the micro-structures of TiO_2 nanofibers. Figure 14 shows that these nanofibers are composed of anatase crystallites of 8 nm in average size. The microstructural information obtained at the HTML will enable Prof. Fong and his research group to tailor the processing parameters to achieve optimized fiber properties.

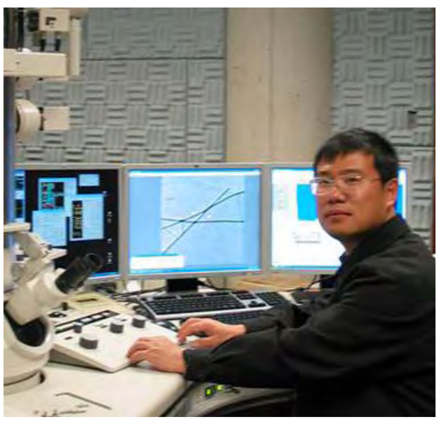


Fig. 13. Dr. Lifeng Zhang of SDSMT at the HF-3300 console.

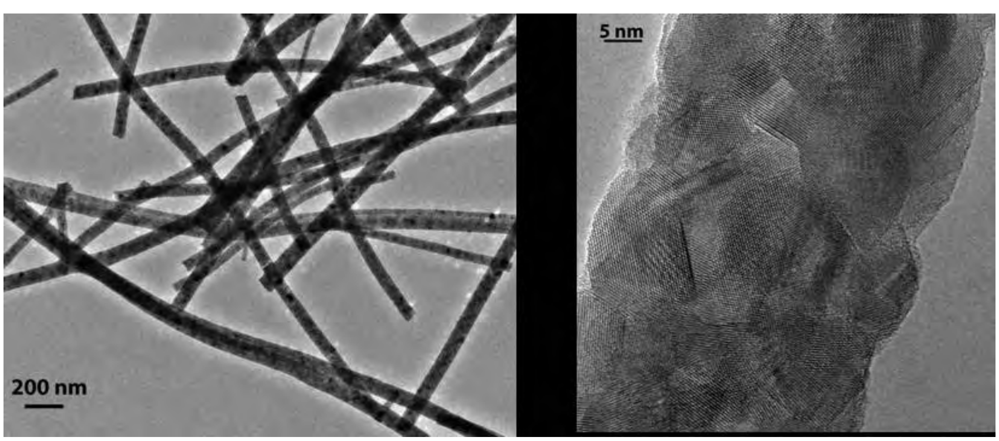


Fig. 14. TiO_2 nanofibers (left) composed of anatase crystallites (right) averaging 8nm in size.

Other Materials Analysis User Center News

ACEM images prepared during HTML user project selected for **MRS Bulletin** *cover*

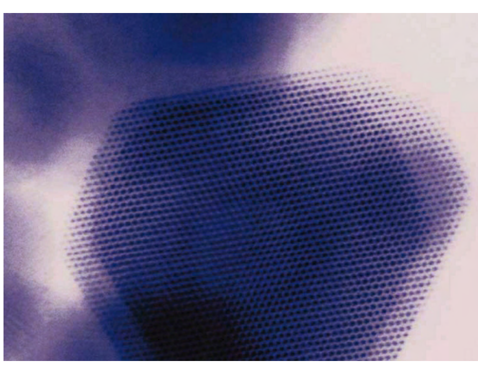
HTML research staff member Dr. Larry Allard and Dr. Paolo Ferreira of the University of Texas at Austin collaborated on a study of Pt_3Co nanoparticles and were the first to report PT segregation on the surface of Pt_3Co nanoparticles of ~5nm upon annealing at 1000K. Images from the the HTML's aberration-corrected electron microscope (ACEM) appear on the February 2008 cover of the *Materials Research Society Bulletin*, shown below with the accompanying description and link to the cover story on the MRS web site.

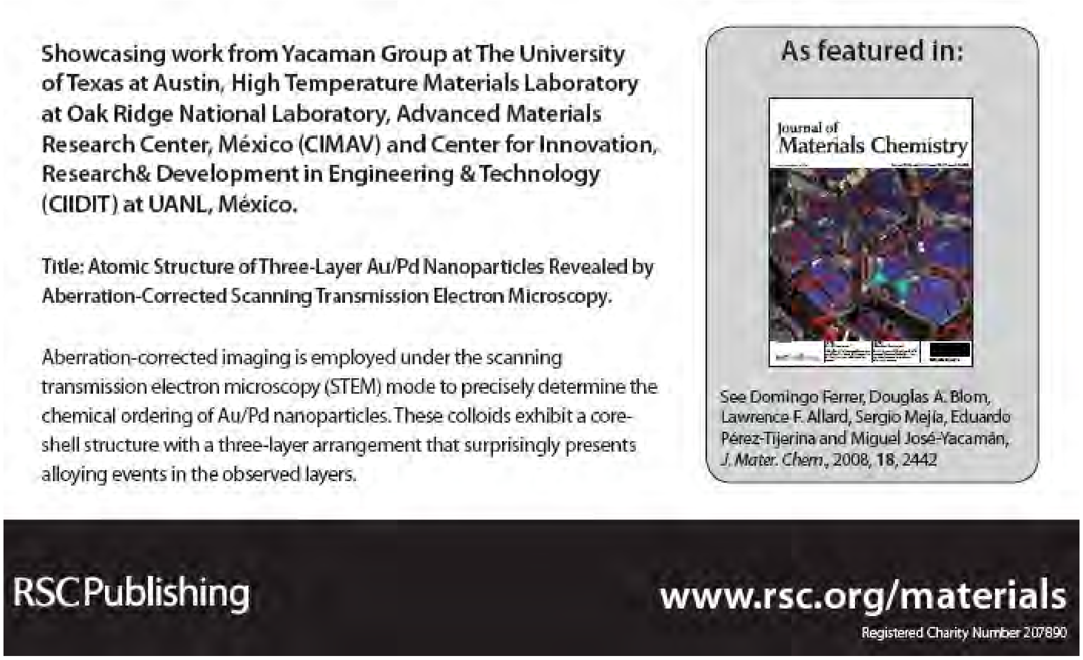
In Situ Transmission Electron Microscopy. A sequence of aberration-corrected high-angle annular darkfield scanning transmission electron micrographs shows the coalescence of platinum nano particles under the influence of the electron beam at ambient temperature. The images, left to right, were recorded in intervals of 20 seconds in the JEOL 2200FS located at Oak Ridge National Laboratory. In addition to the platinum nanoparticles, individual platinum atoms and platinum clusters can also be observed on the carbon support. (Image courtesy of P.J. Ferreira, **L.F. Allard**, and Y. Shao-Horn. Work supported by the Department of Energy Office of Vehicle Technologies, through the High Temperature Materials Laboratory User Program.) See the technical theme that begins on p. 83.

(see http://www.mrs.org/s_mrs/doc.asp?CID=13583&DID=207952&css=print)

ACEM image featured on back cover of *Journal of Materials Chemistry*

Dr. Miguel Jose Yacaman from the University of Texas at San Antonio continued work on his HTML user project with HTML researcher Dr. Larry Allard. They have studied the structure and morphology of Au/Pd bimetallic particles using primarily the high-angle annular dark-field (HAADF) imaging mode in an aberration-corrected STEM/TEM. Images from the aberration-corrected electronic microscope (ACEM) indicate that the surface layer of the nanoparticle contains kinks, terraces and steps at the nanoscale. In addition, the particles exhibit a coreshell structure with a three-layer arrangement, as shown in the following back cover for the March 2008 issue of the *Journal of Materials Chemistry*.





link to journal: http://www.rsc.org/Publishing/Journals/jm/ (previous issues, number 21)

SAE selects paper authored by MAUC's Harry Meyer as one of best SAE papers in 2007

The Society of Automotive Engineers (SAE) has selected Dr. Harry Meyer's paper, "Effects of silicon and boron additions on the susceptibility to quench embrittlement and the bending fatigue performance of vacuum carburized Modified 4320 steel," as one the best SAE technical papers of 2007. It will be published in the *SAE 2007 Transactions: Journal of Passenger Cars – Electronic and Electrical Systems*. The paper presents the results from an HTML user project with co-author Jason Spice from the Colorado School of Mines.

Mechanical Characterization and Analysis User Center (MCAUC)

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

Colorado School of Mines conducts *in situ* Raman analysis of beta-eucryptite pressure-induced transformation

Research problem: To develop greater understanding of the fundamentals of phase transformations in β-eucryptite

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

Description of Work: Several strategies to meet new diesel engines emission limits (no more than 0.2 grams per brake horsepower hour (g/bhp-hr) of nitrogen oxide (NOx) and 0.14 g/bhp-hr of non-methane hydrocarbons (NMHC) include the use of high-thermal-shock-resistant, low cost honeycomb soot filter compatible with advanced emissions control catalyst technologies. 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 subjected 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 for applications in catalyst support and filtration of diesel particulates.

Professor Reimanis' graduate student Timothy Jochum (Fig. 15) visited the HTML to continue working 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. The incident laser light was focused through a ~100 μm diameter flat diamond punch that was loaded between 1 and 6.5 kg. Since there was some residual Al_2O_3 present in the samples, the applied load could be calibrated using the well-known R-line luminescence peaks, which resulted in applied hydrostatic compressive stresses ranging from ~1.25 to 6.25 GPa. Figure 16 shows the shift in the Raman peak for β-eucryptite caused by loading the sample. The peak initially shifts to higher frequencies, which shows that the material is under compression; however, it then appears to decrease and reach a constant value corresponding to the phase transformation to ε-eucryptite. There also appears to be a hysteresis in the loading and unloading curves that may be caused by microcracking in the material. The investigation results suggest the potential of β-eucryptite for the development of tough, durable diesel particulate filters.

Fig. 15. Tim Jochum collecting Raman spectra on his β-eucryptite sample.

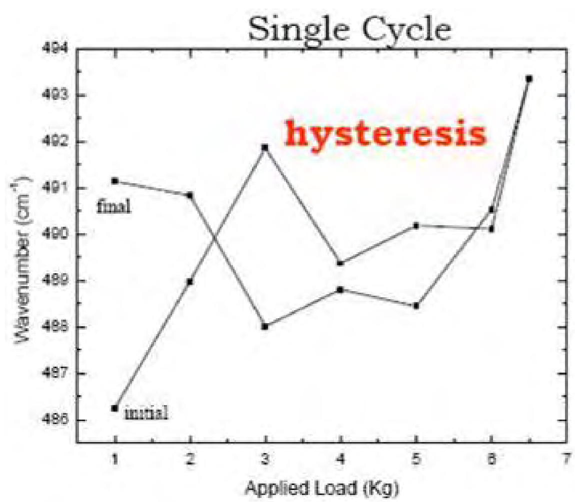


Fig. 16. The frequency shift with load of the β-eucryptite Raman peak. The stress increases until the phase change to ε-eucryptite.

SUNY-SB researchers evaluate elastic anisotropy in thermal barrier coatings

Research problem: Characterization and analysis of elastic properties including the anisotropy of thermally sprayed coatings

Implications: Understanding and predicting the long-term performance and lifetime of thermal barrier coatings (TBCs) for turbine engines and potentially for diesel engines

Description of Work: State University of New York – Stony Brook researchers Dr. Yang Tan and Prof. Sanjay Sampath have been working with HTML researchers to apply advanced characterization techniques to thermal barrier coatings (TBCs). In previous visits, the SUNY-SB researchers have collaborated with TTPUC researcher Dr. Hsin Wang to measure high temperature conductivity and the anisotropy in thermal conductivity of TBCs. During this visit, Dr. Tan (Fig. 17) worked with MCAUC researchers Amit Shyam and Edgar Lara-Curzio to characterize the elastic anisotropy of TBCs at room temperature and at elevated temperatures, using the non-destructive technique of Resonant Ultrasound Spectroscopy (RUS). Several different zirconia (YSZ) based coatings with different processing parameters were analyzed along with alumina, nickel, molybdenum and Ni-5Al coatings. It was possible to characterize the elastic modulus anisotropy of the coatings, as in the example shown in Fig. 18. The elastic anisotropy of the zirconia coatings was also determined up to 1200°C. It was found that most thermal barrier coatings possess hexagonal anisotropy due to the unique layered structure of splats shown in Fig. 18. Dr. Tan recently presented the results of the potential of application of RUS for TBCs to a consortium of industrial researchers, as elastic modulus anisotropy will provide industry with a critical tool for assessing and optimizing the operating performance of TBCs.


Fig. 17. Dr. Yang Tan with the resonant ultrasound spectroscopy (RUS) unit.

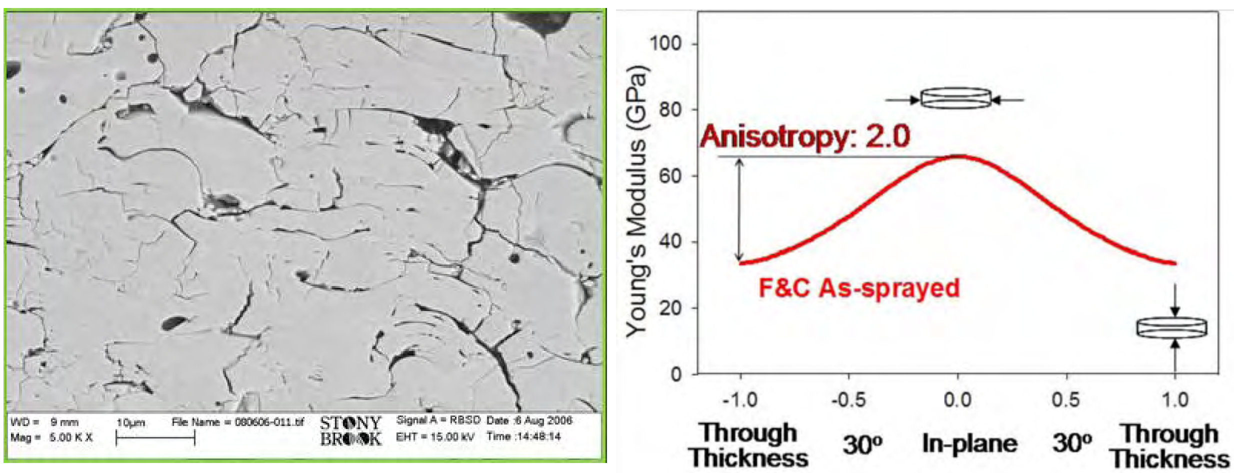


Fig. 18: The layered microstructure and the resulting elastic anisotropy in elastic properties for a YSZ thermal barrier coating. F & C ("fused and crushed") refers to the characteristics of the starting powder to make the thermal barrier coating.

Materials Innovation Technologies evaluates recycled carbon fibers

Research problem: Evaluate strength retention of carbon fibers recovered from scrapped or recycled composite pieces

Implications: Successful recovery of carbon fibers from rejected or recycled parts could help reduce the cost of carbon fibers

Description of Work: Significant efforts have been focused on developing structural materials for body and chassis applications that significantly reduce the weight of passenger vehicles without compromising vehicle lifecycle cost, performance, safety or recyclability. The greatest single barrier to the market

Materials Innovation TECHNOLOGIES

viability of lightweight materials for automotive and commercial vehicle applications, and fiber-reinforced polymer matrix composites in particular, is the high cost of finished materials.

Fig. 19. David Haack testing unused single carbon fibers under tension.

Materials Innovation Technologies LLC (MIT-LLC) is developing a process to recover carbon fibers from manufacturing scrap and end-of-life components. MIT-LLC estimates its process can recover and reprocess carbon fibers for less than 50% of the cost of virgin carbon fibers. Characterization of these recovered carbon fibers is being conducted at the HTML by Dr. David Haack from MIT-LLC (Fig. 19) and MCAUC staff members Rosa Trejo and Edgar Lara-Curzio. Tests on single carbon fibers in tension compared the retention of strength and modulus of the recovered fibers to unused fibers. Initial results are promising, and successful development of this technology could enable the cost-effective use of carbon fiber-reinforced composite materials on automobiles and transportation vehicles.

Ohio State characterizes interfacial properties of joints made by electromagnetic welding

Research Problem: Understand mechanical properties across electromagnetically-welded interfaces in aluminum and copper

Implications: Electromagnetic pulse welding could provide a low-cost joining technology for the automotive industry.

Description of Work: Ohio State University researcher Yuan Zhang (Fig. 20) and the HTML's Dr. Sanghoon Shim performed nanoindentation measurements across the interface of joints obtained by electromagnetic pulse welding techniques. Tests were conducted on both Al6061-Al6061 and Cu-Cu samples. Hardness measurements revealed that the interfacial region (30 to 50 μm on both sides of the interface) had 30% higher hardness than the base metal (Fig. 21, next page). It was also found that hardness and the size of the interfacial region depend on the type of material and the magnitude of the discharge energy used during the joining process. Microstructural analysis revealed that there were no brittle intermetallic phases present in the interfacial region, in contrast to traditional fusion welding. The results from this HTML user project suggest that electro-magnetic pulse welding could be an attractive technology for automotive applications.

Fig. 20. Ohio State University's Yuan Zhang verifies settings prior to placing sample in nanoindenter.

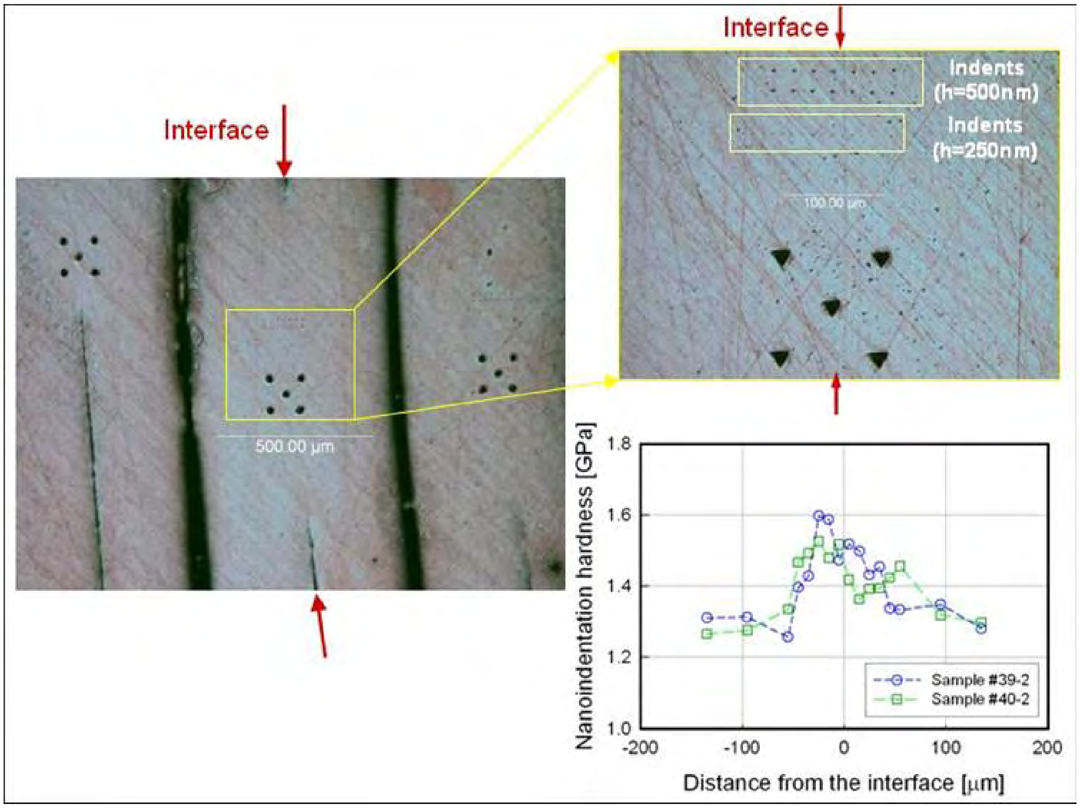


Fig. 21. (left) optical microscope image of welded interface for Al6061 plate, (top right) same image of Al6061 plate with higher magnification, and (bottom right) nanoindentation hardness profiles along the welded interface.

University of Tennessee Forest Products Center investigates mechanical properties of wood cells

Research problem: Characterization of bio-based materials using nanoindentation

Implications: Bio-composites decrease overall weight and make parts recyclable for the automotive industry.

Description of Work: Natural fiber-reinforced polymer composites (bio-composites) are of interest for applications in transportation (e.g., interior panels, headliners, and seat panels in automobiles) because they are lightweight, easy to process, and could cost less than traditional polymer composites. However, good strength is also a requirement for some of these parts, and it varies significantly for different types of wood. University of Tennessee graduate student Xinan Zhang and HTML researchers Rosa Trejo, Laura Riester, and Edgar Lara-Curzio are investigating the mechanical properties of wood cell walls via nanoindentation techniques. Test specimens consist of 4µm-diameter wood cell pillars prepared by focused ion beam milling techniques (Fig. 22a, next page) and are evaluated using the HTML's Nano II nanoindenter (Fig. 22b, next page). This on-going investigation will provide important information about the strength of wood cells to be used as natural fiber-reinforced composites.

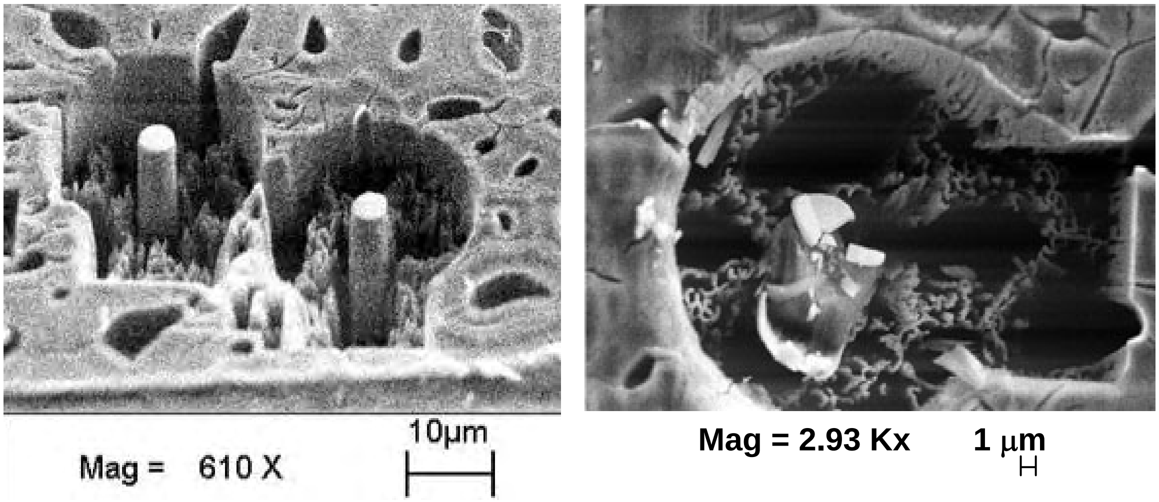


Fig. 22. a) wood cell columns prepared via FIB; b) wood cell column after compression with a nanoindenter eight micron punch.

Other Mechanical Characterization and Analysis User Center News

Michigan State User earns Ph.D. with research conducted at HTML

Fei Ren, a postdoctoral research associate at Michigan State University, successfully defended his dissertation, “Mechanical Characterization and Powder Processing of Lead Telluride Based Thermoelectric Materials,” and received his doctorate in Materials Science & Engineering in December 2007. A large part of his research was conducted under an HTML user proposal on characterization of thermoelectric materials. He was also nominated as Distinguished Graduate Student by his department (Chemical Engineering & Materials Science).

Residual Stress User Center (RSUC)

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

Mississippi State measures residual stresses in LENS-deposited SS410 thin wall plates

Research problem: Minimize residual stresses in LENS components in order to achieve high ductility and strength

Implications: Design of a process analysis tool to improve the LENS process and produce consistent LENS-manufactured components with known ductility and hardness, resulting in improved microstructure and minimized residual stress

Description of Work: Laser Engineered Net Shaping (LENS) is a technique used for the rapid fabrication of fully dense steel components. According to this process parts are constructed by focusing a laser beam onto the deposition region, where nozzles, using a computerized process, simultaneously inject metallic powders. The laser locally melts the powder to create a molten pool on the top surface of the growing part. After deposition of each layer, the powder delivery nozzle and the laser beam assembly are raised in the direction of growth, thereby building a three-dimensional component layer additively. The microstructural features and mechanical properties of the final part are significantly affected by the cooling rate and solidification velocity, and by the thermal cycles that may occur during the deposition process.

The Center for Advanced Vehicular Systems at Mississippi State University (MSU) is developing models for the LENS process under a DoD grant. Neutron residual stress mapping was selected to characterize the through thickness stresses in a series of SS410 thin walled plates in order to correlate residual stresses in the plates with laser power, laser speed, and powder flow rate. The ultimate goal is for the data to be used in both developing and validating models for the process.

Graduate student Phillip Pratt made two visits to the HTML to evaluate seven LENS-made samples under the guidance of HTML researcher Dr. Cam Hubbard. The high spatial resolution of the Neutron Residual Stress Mapping Facility at ORNL's High Flux Isotope Reactor was essential to performing valid measurements of the 1-2 mm thick wall samples. The relatively large grain size turned out to be a challenge that was overcome by oscillation methods. Typical data are shown in Figs. 23 and 24, which are plots of the stress perpendicular to the deposition lines mapped along two orthogonal lines. Figure 23 displays the increasingly compressive stress σ_z with change of height of the deposit, while Fig. 24 shows that σ_z along the length of the sample has random fluctuations. These fluctuations are likely due to localized changes in laser power absorption and deposition temperature. Modeling results show qualitatively similar trends in residual stresses. By establishing correlations between laser power, laser speed and powder flow rate and the resulting state of residual stresses it will be possible to optimize the mechanical properties of LENS-made components.

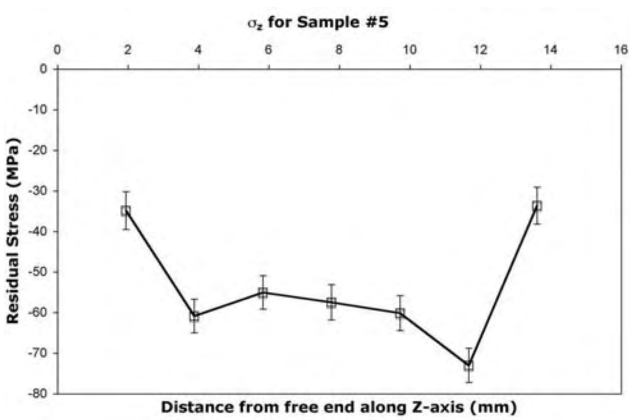


Fig. 23. σ_z mapped perpendicular to the deposition direction from top edge of sample toward the substrate.

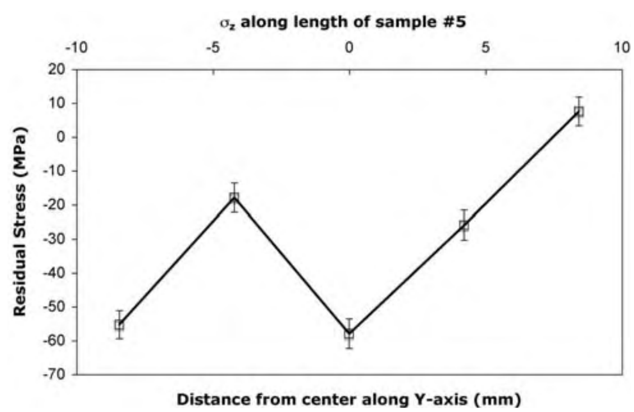


Fig. 24. σ_z mapped parallel to the deposition direction at mid height.

GM and UT study effects of hydrogen charging on properties of Al-Si castings

Research problem: Assessment of use of neutron based scattering and imaging techniques to develop fundamental understanding of structural characterization of commercially important Al-Si castings

Implications: Energy savings resulting from reduced scrap, ability to make sound thin-wall lightweight metal castings and to quantify casting quality non-destructively

Description of Work: Hydrogen is the only gas that is appreciably soluble in aluminum and its alloys. During the cooling and solidification of molten aluminum, dissolved hydrogen in excess of the solid solubility limit may precipitate in molecular form, resulting in the formation of voids. General Motors is collaborating with University of Tennessee-Knoxville Department of Civil Engineering Professor Dayakar Penumadu and his graduate students to assess the potential of using neutrons to characterize Al-Si castings. In particular they are interested in determining correlations between hydrogen gas pressure present over the molten Al-Si, on lattice parameters and residual strains in the alloy.

The RSUC's Dr. Cam Hubbard and graduate student Jeff Bunn of the University of Tennessee-Knoxville used the Neutron Residual Stress Mapping Facility at HFIR to examine several castings prepared by GM. Exploratory measurements of cast blocks and Ransley mold pins shows that the grain size in the blocks is larger than that in the smaller Ransley mold pins, likely due to differences in cooling rates. Only in the Ransley mold pins was a difference in d-spacing observed for the Al (311) planes between the specimens prepared from Al-Si with and without hydrogen gas (Fig. 25). The random variation of d-spacing for the Al (200) planes is due to large grain size. The strains in the silicon secondary phase in the castings were also measured, and when large grains of aluminum are present, the fine grained silicon can be used for macro strain measurement. In summary, there were no indications of significant changes in strain with hydrogen content in the molten aluminum alloy. These results will help GM optimize the casting of aluminum alloys for engine components.

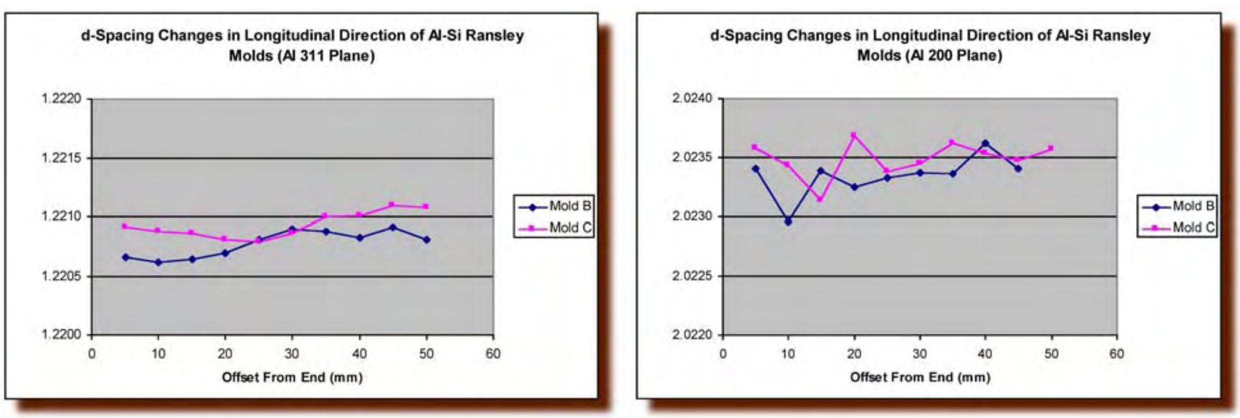


Fig. 25. Al (311) and Al (200) d-spacings for Ransley mold pins cast from binary Al-Si melt with and without hydrogen gas.

Metalsa, Inc. characterizes residual stresses in railsides for truck frames

Research problem: Correlate the hole cutting manufacturing process and choice of steel for the railside of heavy trucks with the level of residual stress and fatigue life

Implications: Knowledge of residual stresses and their correlation to fatigue life and crack propagation will enable Metalsa to improve life, safety, and potentially reduce weight of the truck railside.

Description of Work: Metalsa is a tier 1 supplier to over 50% of the North American heavy truck market. To improve manufacturing processes and the reliability of materials and to achieve reducing weight, Metalsa has initiated a study of railside fatigue life, comparing both processing and properties of high strength low alloy steel (HSLA) to the industry standard heat-treated rail side. Fatigue cracking around holes cut in the railsides is also a safety concern, and Metalsa needs to assure the railside will not fail in service due to cracking. Dr. Joaquin del Prado Villasana from Metalsa (Fig. 26) and Dr. Cam Hubbard from the HTML evaluated nearly 30 plates using both industry standard heat-treated and HSLA steels. Four methods of producing holes in the steel components were studied: plasma arc cutting, laser cutting, punching, and NC drilling. These processes will be correlated with the railside fatigue life and the level of residual stress. In addition, residual stresses in plates without holes will be mapped to determine the effect of metal processing prior to hole cutting.

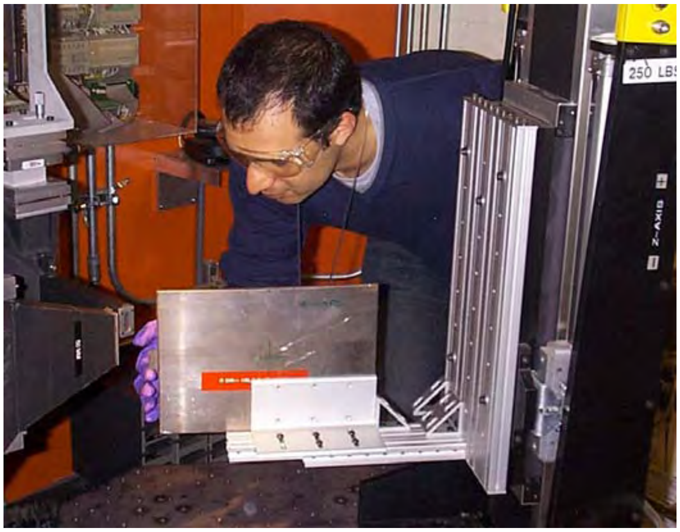


Fig. 26. Dr. Villlasana of Metalsa mounts a plate sample for through thickness strain mapping as a function of distance from the hole along a line transverse to the rolling direction of the plate.

Residual stresses were measured as a function of distance from the hole diameter and depth through the plate using the neutron residual stress mapping facility, NRSF2. Radial, transverse and normal strains were measured along a line perpendicular to the plate rolling direction. The strain mappings clearly showed that the hoop strains are largest and extend over a greater distance in the following order (from highest to lowest tensile stress levels): plasma, laser, punch and NC drilled. Figure 27 shows the residual stresses as a function of distance from the hole at the mid thickness of the plate (left image), while the right image plots the through thickness distribution at 4 mm from the hole edge. The two thermal cutting methods relax the residual stresses

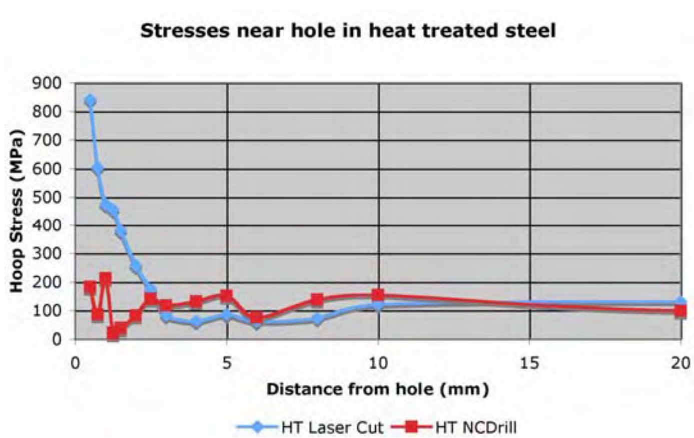


Fig. 27. Comparison of hoop residual stresses resulting from plasma arc cutting and NC drilling of holes in heat treated steel plates.

in the original plate, while introducing high levels of tensile residual stress that extend for several millimeters from the hole. The punch process also increased the plate stresses near the hole, while the NC drill cutting introduced the lowest change in residual stress. The knowledge gain will help Metalsa offer a high performing product capable of improving the durability, efficiency, and safety of heavy trucks.

NC State University investigates residual stresses on welded piping joints

Research Problem: To measure residual stresses using neutron diffraction on SS304 welded piping joints before and after fatigue loading

Implications: Potential to optimize weld procedure and avoid early fatigue failure on piping joints

Description of Work: In recent decades, some unexpected fatigue failures have occurred in welded joint of metal structures under cyclic loading, but often the cause of failure could not be determined. Researchers have proposed that fatigue failure of welded joints might be related to ratcheting, but the detail of the failure mechanism has not yet been discovered. One main factor anticipated to be the reason of ratcheting fatigue failure is weld residual stress at the weld toe or heat affected zone (HAZ). The goal of this research is to understand the role of residual stress on ratcheting failure.

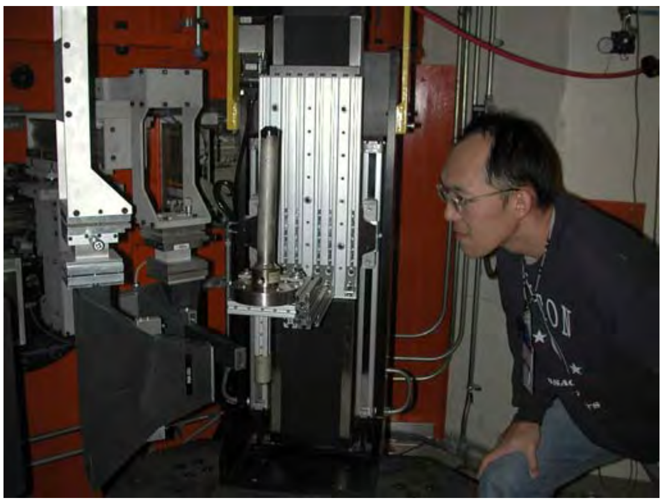


Fig. 28. Pei-Yuan Cheng from North Carolina State University inspects the sample mounting on the XYZ-stage of the NRSF2 at HFIR.

Pei-Yuan Cheng, an NCSU Ph.D. student (Fig. 28), visited the HTML to work with RSUC researchers Camden Hubbard, Thomas Watkins, Fei Tang, Barton Bailey, Amit Shyam, and Josh Schmidlin to examine three 304 stainless steel socket piping joint specimens. The three specimens were welded according to the welding parameters and procedures of the failing components. Initial residual stress measurements were performed in the HAZ of the each specimen. Subsequently, these three specimens were subjected to displacement-controlled fatigue tests for various cycles before the residual stresses were measured again at the HTML's Neutron Residual Stress mapping Facility (NRSF2) located at ORNL's High Flux Isotope Reactor (HFIR).

From the obtained residual stress data using neutron diffraction, the axial residual stresses were found to be compressive at the outer surface near the weld toe before fatigue loading. After fatigue loading, the axial residual stresses at these locations were found to become more tensile (Fig. 29, next page). Tensile residual stress can act as a positive mean stress, which causes ratcheting behavior in materials and becomes a source of early fatigue failure. This research also provides evidence that residual stresses are not totally relaxed after a long number of cycles. The residual stress results obtained in this project will also be used for the verification of an FEM analysis, which will be applied for the optimization of weld procedure to improve fatigue life of welded piping joints. This result contributes to DOE's missions that use piping, such as EERE's Hydrogen Delivery R&D activities.

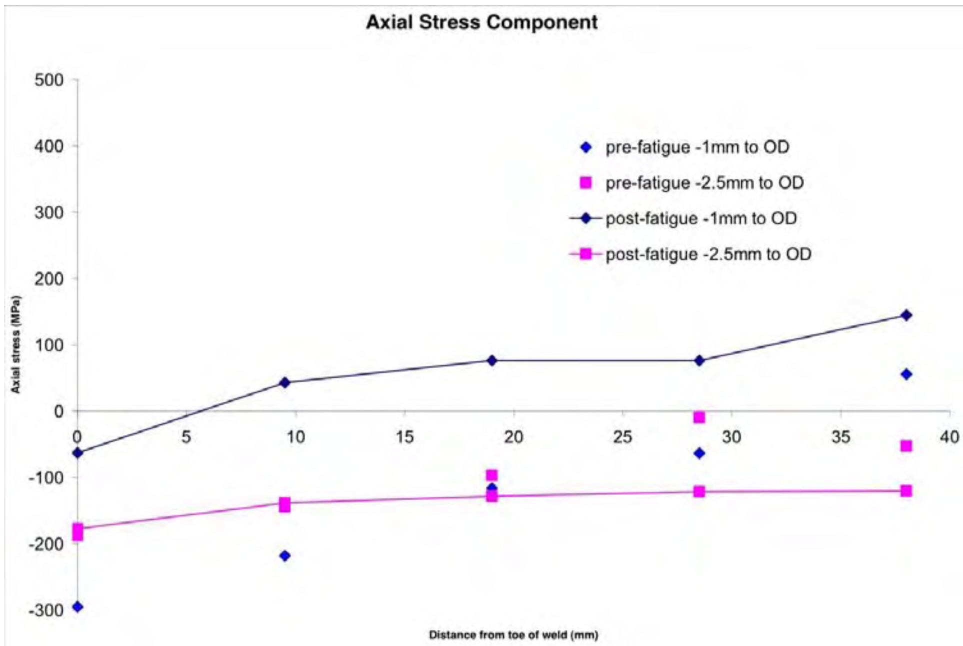


Fig. 29. Axial residual stresses near the outer surface (-1 mm from the outside diameter) relaxed or become tensile after fatigue loading. Smaller changes in residual stress were observed in deeper locations (-2.5 mm from the outside diameter).

John Deere studies the residual stress distribution in welded structures

Research problem: Determine the impact of residual stresses distribution on fatigue life of structural components

Implications: By understanding the effect of residual stresses on the fatigue life of structural components, it will be possible to develop improved models to predict the service life of off-highway vehicles and lightweight components with improved properties.

Description of Work: This research adds to the body of knowledge in weld life predictions for off-highway vehicles, in which prediction models currently only consider fatigue life initiation and neglect the propagation of the crack portion of the life prediction. It is estimated that by taking into account the propagation, the industry could reduce the thickness of material in designs by 10% or even greater in some cases. This will result in a substantial savings of materials, costs and energy for manufacturers.

In addition, residual stresses resulting from welding can cause significant problems both in manufacturing and in the service life of welded and heat treated assemblies. There are no methods of measuring post processing residual stresses that are simple, field deployable, non-destructive, through thickness, and cost effective. Quantification of such stresses would allow design and manufacturing engineers to more effectively define welded part geometries and the frequently used subsequent heat treatment processing to minimize detrimental effects of residual stresses.

Eric Johnson from John Deere visited HTML staff Thomas Watkins, Amit Shyam, Josh Schmidlin and Cam Hubbard to validate new welding simulation and prediction models by using neutron and x-ray diffraction for non-destructive mapping of the residual stresses throughout test samples. In particular, the research team investigated the residual stress distribution within

welded “T” plates (Fig. 30). In order to account for the fatigue life and the potential crack propagation, the residual strain/stress profile was measured from the weld toe through the thickness and along the length of the welded “T” plates. Data analysis and comparison with the process and fatigue models is still on-going, but once validated, the process and fatigue models will be widely used to improve the life and reduce the weight of many components in John Deere’s off-road vehicles (Fig. 31). Ultimately, this project will help John Deere produce better, long-lasting parts, resulting in energy conservation, since replacement parts do not have to be produced as frequently. Another benefit will be increased fuel efficiency because of lighter weight vehicles.

Fig. 30. Eric Johnson of John Deere positioning the welded “T” plates for neutron diffraction at the Engineering Diffractometer at High Flux Isotope Reactor (HFIR), Neutron Residual Stress Facility 2 (NRSF2).

Fig. 31. Similar welded structures are used in scraper tractors.

Radiation Monitoring Devices tests neutron imaging detector and aids a GM study of neutron imaging of hydrogen charging of Al-Si casting alloys

Research problem: To characterize the performance of neutron imaging detectors using high flux neutron beams

Implications: Improved techniques for *in situ* imaging of energy storage systems such as lithium ion batteries, hydrogen storage systems, hydrogen adsorption in piping and storage materials, and operating fuel cells

Fig. 32. Very high flux beam used to test imaging sensors.

Description of Work: In order to meet U.S. industry demand for improved neutron imaging facilities in order to expand non-destructive examination (NDE) options, Radiation Monitoring Devices, Inc. (RMD) is developing neutron imaging sensors designed for higher resolution and higher efficiency. Stuart Miller of RMD and Cam Hubbard of the HTML used the very high flux, small beams at the neutron residual stress mapping (NRSF2) facility at the High Flux Isotope Reactor to assess the performance of RMD’s developmental neutron imaging screens. The goal of the study at NRSF2 was to compare the performance of a series of sensor plates and then to subject one of the better performing plates to imaging tests on the NRSF2, so the sensors could be characterized for spatial resolution and linearity to neutron flux (Fig. 32). Jeff

Bunn, a graduate student working on a General Motors-funded project exploring hydrogen bubbles in Al-Si castings, is using neutron imaging to study defects in castings. Because of this interest, he participated in the RMD user project (Fig. 33).

Fig. 33. Stuart Miller (front) and Jeff Bunn examine a neutron image of steel tubes which had been subjected to deformation.

Neutron imaging has the advantage of deep penetration and different contrast mechanisms compared to alternatives such as X-ray imaging, particularly in its sensitivity for hydrogen. Advances in neutron imaging detectors and establishment of a world-leading neutron imaging facility have wide range of applications for transportation and energy-related technologies. Prior workshops at ORNL and overseas research have shown that cold neutrons and potentially pulsed neutrons hold promise for a breakthrough in neutron imaging. The studies with RMD, Inc. at NRSF2 and at the Spallation Neutron Source (SNS) are assuring that developments in neutron imaging detectors advance in support of such a future facility.

University of Tennessee studies deformation behavior of materials under tension and torsion

Research problem: Conduct fundamental studies of grain-to-grain deformation behavior and resulting residual stresses due to torsional deformation

Implications: Better tools and understanding to predict the deformation behavior of structural materials under complex states of stress

Description of Work: University of Tennessee's Professor Dayakar Penumadu and graduate student Jeff Bunn collaborated with the HTML's Dr. Cam Hubbard to characterize the intergranular residual strains as a function of radial location within the wall of tubular specimens of AISI 12L14 steel. The RSUC's neutron residual stress mapping facility was used for its high spatial resolution capability to characterize test specimens that were deformed by either pure torsion or uniaxial tension to two target values of equivalent octahedral shear stain. Three orthogonal strains (axial, hoop and radial) were measured as a function of location through the wall of the tube for both sets of specimens (Fig. 34). Diffraction from the (110), (200) and (211) planes was recorded for each sample. Differences in strains for different grain sets for a fixed location are due to intergranular strains that add algebraically to the macro residual stresses.

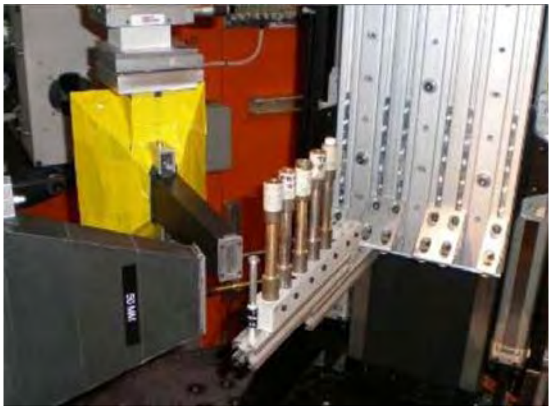


Fig. 34. Tension- and torsion-deformed steel tubes mounted on NRSF2 for measurement of the hoop strains.

The (110) data sets show large intergranular strain differences in axial strains for the tension versus torsion specimens. The (200) data sets show significant intergranular stain differences for the hoop and radial strains (Fig. 35). The (211) diffraction planes are generally free of intergranular effects, as is often observed in ferritic-based steels. Complete interpretation of the data will require advanced elastic-plastic modeling of the deformation processes, including probability of shear and grain reorientation as well as the crystallographic anisotropy of the grains. However, it is clear that the resultant strains for different sets of grains are quite different between the torsion and tension sets of specimens. The findings from this project will lead to substantial gains in accurately predicting differences in strength and probability of failure in real systems when subjected to complex states of stress.

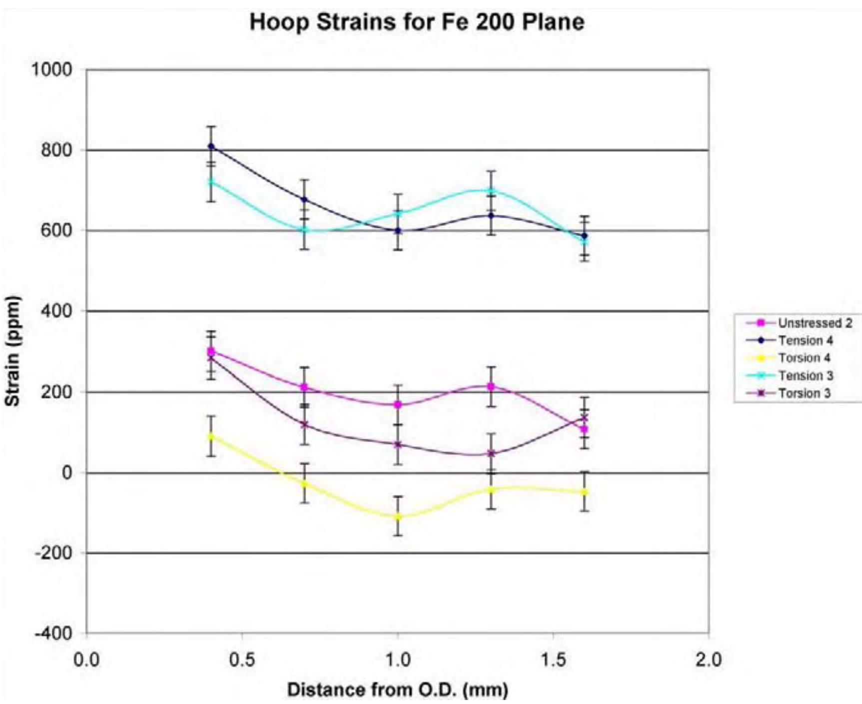


Fig. 35. Hoop strains for the (200) crystallographic planes as a function of depth in the tube wall.

Other Residual Stress User Center News

HTML User completes report for American Gear Manufacturing Association

The American Gear Manufacturing Association (http://www.agma.org/) has distributed the AGMA Foundation Grant final report "In-Situ Measurement of Stresses in Carburized Gears via Neutron Diffraction." This report was prepared by RSUC Users from The University of Tennessee-Martin led by Prof. Robert LeMaster and HTML staff members Camden Hubbard, Tom Watkins, and Barton Bailey.

The work was based on studies performed primarily on the neutron residual stress mapping facility (NRSF2) at the High Flux Isotope Reactor through the HTML User Program at ORNL.

Publications

"In-Situ Measurement of Stresses in Carburized Gears via Neutron Diffraction," Final Report to the AGMA Foundation, April, 2008
http://www.agmafoundation.org/AM/Template.cfm?Section=Research_Grant_Program

Thermography and Thermophysical Properties User Center (TTPUC)

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

Missouri University of Science and Technology characterizes SiC-ZrB_2 composites

Research problem: Characterize thermal conductivity and residual stresses of ZrB2/SiC composites

Implications: Development of high temperature, lightweight materials with optimized thermomechanical properties

Description of Work: Professor William Fahrenholtz and his research group at the Missouri University of Science and Technology (formerly the University of Missouri-Rolla) are developing ultra-high temperature ceramic matrix composites for various applications in propulsion and power generation. One of their goals is to design composite materials with optimum mechanical properties, so they are studying the effect of residual stresses on the strength and mechanical properties of these materials. They have developed finite-element stress analyses using commercially available software (ABAQUS) to estimate the internal or grain to grain stresses in ZrB_2 –SiC composites that develop during cooling after densification by hot pressing. Factors such as SiC particle size, shape and volume percent have been varied and modeled in an effort to understand their impact on internal stresses and material strength. HTML researchers Thomas Watkins (RSUC) and Claudia Rawn (DUC), as well as Ashfia Huq (Spallation Neutron Source), worked with Prof. Fahrenholtz and graduate student Michael Teague to analyze neutron diffraction data obtained at Argonne National Laboratory (Fig. 36). Rietveld refinements were conducted on data collected from room temperature to 1200°C using a hot-pressed sample (Fig. 37). In a future visit to the HTML they will acquire data from stress-free

Fig. 36. Michael Teague from MUS&T analyzes neutron time-of-flight data.

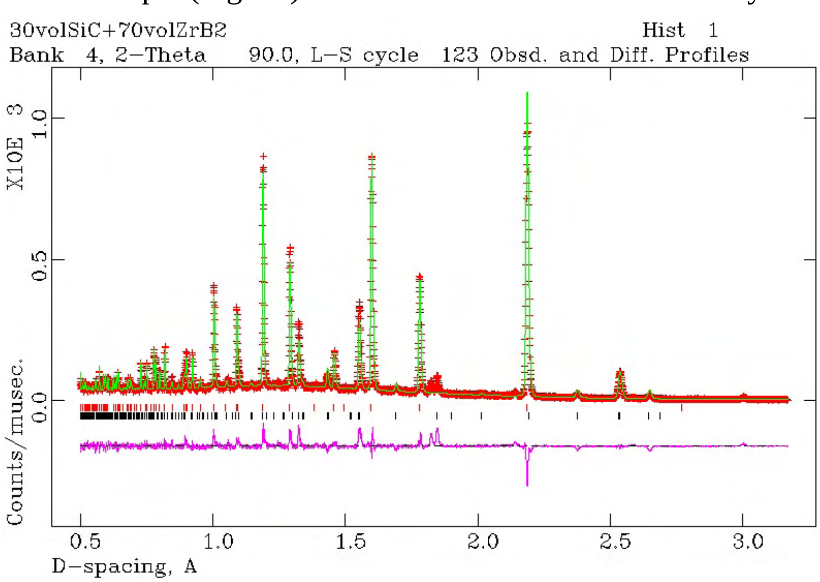


Fig. 37. A Rietveld refinement in progress for 30v%SiC-70v%ZrB_2 (B^{11}) hot-pressed composite. The data was taken *in situ* at 1200°C. The red "+" are the raw data, while the green curve is the fit of the model. The magenta curve is the difference between the raw data and the model. The gray (upper) and black (lower) hash marks are the reflection markers for the SiC and ZrB_2, respectively.

powders at elevated temperatures using neutron diffraction techniques. Mr. Teague also worked with the HTML's Dr. Hsin Wang to determine the effect on thermal transport properties of replacing the B^{10} isotope with B^{11} in the composite material (Fig. 38). Natural boron contains 80% B^{10} and 20% B^{11}, but B^{11} scatters neutrons better than B^{10} and is therefore preferred in neutron diffraction studies of materials containing boron. ZrB_2/SiC composites containing natural boron and B^{11} were prepared at MUS&T using identical processing techniques. Upon initial inspection the two materials were identical in terms of density and microstructure. However, significant differences were found between the thermal conductivities of samples containing natural boron and those containing B^{11}. The thermal conductivity of composites containing B^{10} was always significantly greater than composites with B^{11}.


Fig. 38. Michael Teague of MUS&T working at the TTPUC's laser flash thermal diffusivity

The results obtained in this project will enable the design of ultra-high temperature ceramic composites with optimized thermo-mechanical properties. These results also suggest strategies to design materials such as thermoelectrics with specific thermal properties by changing their isotopic composition, with vehicle applications in energy waste recovery from internal combustion engines.

Tribology Research User Center (TRUC)

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

UCF researcher examines wear resistance and creep behavior of Al-Al_2O_3 nanocomposites

Research problem: To investigate the tribological characteristics and creep behavior of powder metallurgy processed Al-Al_2O_3 nanocomposite materials

Implications: Al- Al_2O_3 nanocomposites could enable the fabrication of components that are both lightweight and wear resistant.


Fig. 39. Prof. Linan An conducting a ball-on-flat friction and wear test.

Description of Work: Prof. Linan An, from the University of Central Florida (Fig. 39), worked with TRUC staff member Dr. Jun Qu to evaluate the tribological properties and creep resistance of Al-Al_2O_3 composites with different particle sizes (50 nm, 1 μm, and 3 μm) and concentrations (5, 10, and 15 vol%). Test results from one size/concentration combination are shown in Fig. 40 (next page). Friction and wear tests were conducted on those composites against AISI 52100 bearing steel using a ball-on-flat reciprocating sliding arrangement. Tests were conducted at room temperature and no lubricant was applied. It was found that composites reinforced with smaller particle size and higher concentration yield wear resistance by as much as 3 orders of magnitude compared to pure aluminum. A hot hardness test was used to study the creep behavior of the composite materials. Microindentation tests were conducted at ~250°C under a 1.86 N load for a variety of dwell times (0.1, 2, 5, 10, 25, 50, and 100 min). There was a strong correlation between the indent size and the dwelling time and the results clearly showed enhanced creep-resistance of the composites compared with pure aluminum. $Al\text{-}Al_2O_3$ composite materials could enable the manufacture of automotive components that are both lightweight and wear resistant.

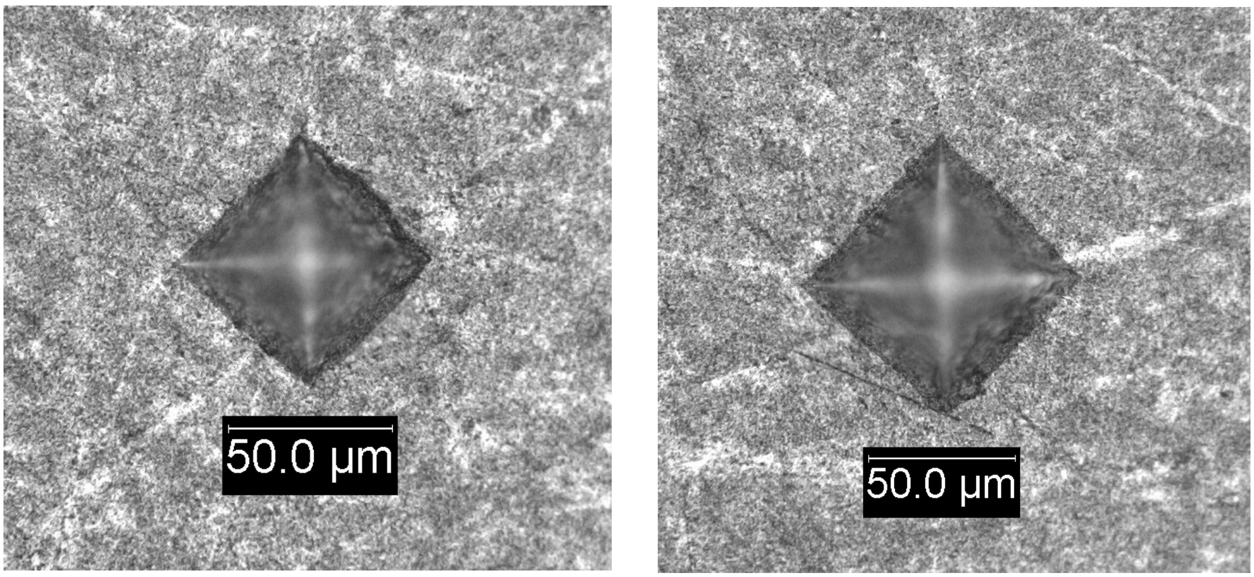


Fig. 40. Comparison of indents on an $Al\text{-}Al_2O_3$ composite (1 µm particle size and 15 vol% concentration) surface after different dwelling times; (a) = 4 sec and (b) = 100 min.

MSU Continues Research on the Abrasive Wear of Forging Die Materials

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: The TRUC assisted Mississippi State University researchers from the Center for Advanced Vehicle Systems (CAVS) in obtaining data that would support their efforts in modeling the wear characteristics of powder metal forging dies. During MSU's second visit, Ph.D. student Wei Li continued work that was begun and reported on last quarter. Working with Peter Blau, he evaluated an alternative method to assess the abrasive wear of hardened steel forging die materials against lightweight alloy powders. The earlier method used a steel-pin test specimen oscillating under pressure against a workpiece consisting of cold-compressed aluminum alloy powder. This method proved unsatisfactory because the compressed powder specimens failed to provide a sufficiently rigid counterface to generate measurable wear on the steel. Blau suggested an alternative method of using a steel plate and inserting a layer of powder between it and the oscillating pin. This might better simulate powder that has been squeezed between mating die surfaces. This seemed to work much better and enabled the abrasive characteristics of the aluminum/hard particle mixtures to be evaluated.

Abrasive mixtures were prepared by adding either 75 wt% silicon carbide (SiC) or 75 wt% alumina (Al_2O_3) powder to aluminum powder. Pins and plates were comprised of two different tool steel grades (CPM 10M, CPM T15) and tungsten carbide. Interval wear tests were run at under a normal force of 25 N, with an oscillating speed of 5 cycles per second, and with a 10 mm stroke length. Wear was determined as a function of time, as illustrated for the tool steels in Figure 3. There was a greater difference between the wear of the two steels for tests with interposed alumina-rich powder than for tests run with silicon carbide-rich powder (Fig. 41). The causes for these differences in observed wear behavior are still being analyzed, and the data will be used by MSU to develop improved models for die wear.

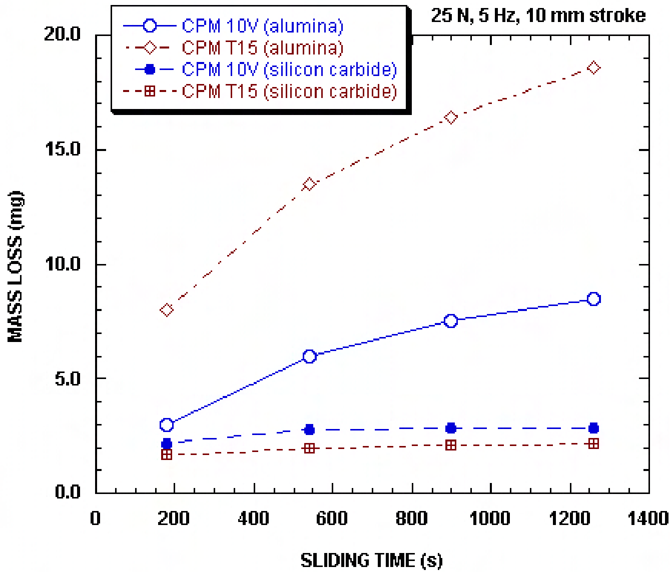


Fig. 41. Cumulative mass loss data for two steels sliding in the presence of two types of loose powders.